

Experimental investigation and modeling of effective thermal conductivity and its temperature dependence in a carbon-based foam

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The effects of test temperature and a graphitization heat treatment on thermal and thermo-mechanical properties of a carbon-based foam material called CFOAM[®] are investigated experimentally. Thermal diffusivity is determined using a laser flash method, heat capacity via the use of differential scanning calorimetry, while (linear) thermal expansion is measured using a dilatometric technique. Experimental results are next used to compute the effective thermal conductivity and the coefficient of thermal expansion as a function of test temperature. The computed thermal conductivity results are then compared with their counterparts obtained using our recent model. The agreement between the experiment-based and the model-based results is found to be fairly good only in the case when the graphitization temperature is high relative to the maximum test temperature and, hence, CFOAM[®] does not undergo a significant additional graphitization during testing. A potential use of CFOAM[®] as an insulation material in thermal protection systems for the space vehicles is discussed. © 2006 Springer Science + Business Media, Inc.

Nomenclature

α	– Thermal diffusivity
C_p	– Heat capacity
CTE	– Coefficient of (linear) thermal expansion
$d_{(002)}$	– The (002) inter-planar spacing in nanometers
ΔL	– A change in the linear dimension relative to the reference linear dimension L_0
ΔT	– Temperature change relative to the reference temperature
k	– Thermal conductivity
L_0	– Reference-temperature value of the linear dimension
p	– Degree of graphitization
ρ	– Foam density
T	– Temperature

1. Introduction

Due to their light weight and potential tailorability of their physical and mechanical properties over a wide range, foam-like materials based on metals, ceramics, polymers and their mixtures have received over the last several years a great deal of attention [e.g. 1–7]. Recently, a new carbon-based foam called CFOAM[®] was

developed which possesses an excellent combination of tailorable thermal, mechanical and physical properties [8]. For example, electrical conductivity of CFOAM[®] can be varied over seven orders of magnitude using different heat treatments which produce various levels of graphitization in this material. This material does not produce off-gases at elevated temperatures and does not support ignition and, hence, possesses a high fire resistance. In addition, CFOAM[®] has excellent sound-absorbing capabilities [8].

CFOAM[®] is produced by Touchstone Research Laboratory, Ltd. from high sulfur bituminous coal using a proprietary technology and it is available in two principal microstructures: (a) an open-cell reticulated microstructure consisting of a ligament network and (b) a cellular microstructure consisting of thin-wall spherical cells. CFOAM[®] with the open-cell reticulated microstructure has the lowest densities and, since it easily graphitizes, it possesses the highest levels of thermal and electrical conductivities among the CFOAM[®] family of materials. Since the open cell structure in reticulated CFOAM[®] is composed of carbon ligaments, this type of CFOAM[®] offers polymer reinforcement capabilities [8]. This type of CFOAM[®]

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is commonly referred to as CFOAM-17[®] where the number 17 pertains to its density in lb_m/ft³. The cellular CFOAM[®], commonly referred to a CFOAM-25[®], has a microstructure consisting of sintered thin-wall, hollow spheres with only sporadic pinholes in the cell walls. Consequently, this type of CFOAM[®] has a very low permeability for fluids (e.g. flammable liquids and gases) and acquires a high fire-resistance and good fire-retardation properties. In addition, since CFOAM-25[®] is less graphitizable, it possesses a lower thermal conductivity and a higher strength, relative to those encountered in lighter-weight CFOAM-17[®] materials. Currently, both types of CFOAM[®] are being considered for use in thermal-insulation and structural applications. Specifically, the CFOAM[®] materials are presently being studied as candidate insulation filling materials in metallic thermal protection system (TPS) panels and for backside insulation of the carbon-carbon TPS sections of the nose cap and the leading edges on the future space vehicles.

As mentioned above CFOAM[®] is being considered as an insulation material in high-temperature thermal protection systems. At the present time, the key thermal properties of CFOAM[®] at elevated temperature are not known. Therefore, the objective of the present paper is to determine and analyze the main thermal conduction properties (thermal diffusivity and thermal conductivity) of CFOAM-17[®] at temperatures ranging from the room temperature up to 2300K. A critical step during processing of CFOAM[®] and controlling the properties of this material is the graphitization heat treatment. In particular the properties of CFOAM[®] are found to be greatly affected by the graphitization temperature. In general, lower graphitization temperatures yield the preferred lower values of thermal conductivity for CFOAM[®]. However, when CFOAM[®] is graphitized at a lower temperature and then it is exposed to elevated service temperatures, the graphitization process may resume causing thermal conductivity to increase (due to an increase in the degree of graphitization of the material).

To address these issues, CFOAM-17[®], graphitized at four different temperatures, is studied in the present work. To better understand the experimentally measured thermal properties and their variation with temperature, the experimental results are compared with those obtained using the model proposed in our recent work [9]. The model treats the transfer of heat through a foam-like material, such as CFOAM[®], as a combined effect of conduction through the solid phase of the foam, conduction through the gas residing within the pores of the foam, and radiation of the foam ligament/cell surfaces. The model accounts for the fact that relative contributions of these three modes of heat transfer vary as a function of the local conditions of temperature and pressure and they are also affected by the microstructure of the foam itself.

The organization of the paper is as follows. A brief discussion of the experimental procedures used in the present paper is given in Section 2.1. The effective thermal conductivity model proposed by Grujicic et al. [9] has been briefly summarized in Section 2.2. The

experimental and computational results obtained in the present work are presented and discussed in Section 3. The main conclusions resulting from the present work are summarized in Section 4.

2. Procedure

2.1. Experimental procedure

The objective of the experimental procedures used in the present work is to determine the effect of test temperature on thermal conductivity in CFOAM-17[®] graphitized at four different temperatures (1473K, 1923K, 2273K and 2773K). Heat treatment designations used and the tests carried out for each heat-treatment of CFOAM-17[®] are given in Table I. Since the experimental facilities for direct measurement of thermal conductivity at various temperatures were not available in the present work, thermal conductivity, k , was determined from the measured values for thermal diffusivity, α , heat capacity, C_p , and density, ρ , using the following equation:

$$k = \frac{\alpha}{\rho C_p} \quad (1)$$

In the following three subsections, brief descriptions are given of the experimental procedures used to determine the effect of temperature on thermal diffusivity, heat capacity and density of CFOAM-17[®].

2.1.1. Thermal diffusivity measurements

The use of thermal diffusivity measurements for determination of the thermal conductivity is often preferred over the direct determination of thermal conductivity via heat flux measurements because of the ease of sample preparation, fewer material requirements, relatively fast measurements and a typical high level of $\pm 5\%$ accuracy.

Within the laser flash method used in the present work, which is one of the most popular thermal diffusivity measurement methods, an unfocused laser beam is absorbed in a thin surface layer at the front face of a test sample, while the temperature of the back face of the sample is monitored as a function of time. To determine thermal diffusivity from the temperature vs. time data at the rear face of the sample, a corresponding one-dimensional transient Fourier-type heat conduction problem has to be solved. In this problem, the initial condition is typically assumed to involve two constant-temperature layers: (a) a thin layer (whose thickness is small relative to the test-sample thickness) at the front face of the sample within which a pulse of radiation energy has been instantaneously and uniformly absorbed resulting in a constant high – temperature profile and (b) the remainder of the sample subjected to a constant lower test temperature. Since the sample is typically in the shape of a thin disk, the thermal conduction problem can be considered as nearly one-dimensional and can be solved in a closed form. The only unknown parameter in this solution is thermal diffusivity. Thus, thermal diffusivity is determined by varying its value, in a systematic manner, until the overall difference between

TABLE I Designation Used for Various CFOAM-17[®] Heat Treatments and a List of Experimental Measurements Carried Out in the Present Work.

Heat Treatment Designation	Graphitization Temperature K	Property Measured		
		Thermal Diffusivity	Specific Heat	Coefficient of Thermal Expansion
A	1473	Yes	Yes	Yes
B	1923	Yes	Yes	Yes
C	2273	Yes	Yes	Yes
D	2773	No	No	Yes

the measured and the model-predicted temperature vs. time curves at the back face of the sample is minimal.

Thermal diffusivity of CFOAM-17[®] studied in the present work has been determined using a Netzsch laser flash apparatus, Model LFA-427. This equipment enables thermal diffusivity measurements at temperatures up to 2273K. The active medium, a neodymium: doped gallium-gadolinium garnet (Nd:GGG) of the laser in this equipment emits a 1064nm wavelength radiation in the infrared range. Two krypton discharge lamps are used to supply excitation power to the Nd:GGG crystal, which provides the higher pulse energy required for the laser flash method. A liquid-nitrogen cooled infrared sensor, which is housed in a separate casing directly above the tube furnace of the laser flash apparatus, has a “visual contact” with the back face of the test sample through a calcium fluoride window, and enables measurements of the temperature at the back face of the sample.

Thermal diffusivity measurements are typically carried out using an unfocused laser beam of a sufficient intensity which is evenly absorbed, in an infinitely short time, over the entire front face of the test sample. However, it is important to note that while thermal diffusivity is an inherent property of the material under investigation, the accuracy of thermal diffusivity measurements is affected by a combination of the applied laser power and the sample thickness. It should be noted that measurements will be valid only if the entire laser energy is absorbed in a thin layer adjacent to the front face of the sample. This is accomplished by adjusting the laser power and the pulse width. In general, the temperature increase in the test sample due to laser energy impulse should not be large when one studies the effect of test temperature on thermal diffusivity. However, the laser intensity resulting from low power settings may produce a signal at the back face of the sample, which is below the detection limit of the infrared detector. Consequently, the laser parameters have to be established to obtain the minimum amount of power, while exceeding the minimal required signal at the infrared detector. It is also important to note that materials exhibiting relatively high thermal diffusivity generally require thicker samples in order to produce a long enough rise time for temperature at the sample back face. However, a disproportionate length-to-diameter ratio promotes heat losses through the side surfaces of the sample and may invalidate the one-dimensional nature of the heat transfer problem. While some of these effects can be corrected with the use of a variable laser power and pulse width, the establishment of an optimum sample

length-to-diameter ratio is critical for determination of an accurate value of the thermal diffusivity.

CFOAM-17[®] test samples investigated in the present work were disk-like in shape and had a 12.5mm diameter and a 4 mm thickness. This geometry of the test samples is consistent with the one-dimensional heat transfer analysis used during determination of thermal diffusivity.

2.1.2. Heat capacity measurements

The rate at which the heat (enthalpy) is absorbed by a material as its temperature is increased under constant pressure is defined as the constant-pressure specific heat or heat capacity. Since the computation of thermal conductivity from the measured values of thermal diffusivity according to Eq. (1) entails the knowledge of heat capacity (and its variation with temperature), heat capacity as a function of temperature is measured in the present work. Within graphite based materials, like CFOAM[®], heat capacity is typically found not to be a sensitive function of material’s heat treatment/microstructure. However, the heat capacity is generally found to be a very sensitive function of temperature.

The heat capacity for CFOAM-17[®] studied in the present work was determined using a Netzsch STA-429 Heat Flux DSC/TGA/DTA Analyzer. This equipment, in its Differential Scanning Calorimetry (DSC) mode, enables heat capacity measurements in a temperature range between the room temperature to 2673K, and also, in its Differential Gravimetric Analysis (DGA) mode, allows for the simultaneous measurements of a weight loss in the test sample. The Differential Scanning Calorimetry technique used to measure the heat capacity of CFOAM-17[®] is based on monitoring the difference in heat flow to or from a test sample and to or from a reference sample as a function of temperature while the samples are subjected to a controlled temperature program.

Instrument calibration, i.e. validation of the heat capacity values at different temperatures for a standard material, was accomplished using a Sapphire standard sample and the test temperatures between 323K and 1298K. All measurements were carried out at a constant 10K/min heating rate and under an argon atmosphere.

Initial measurements involving solid disk-shaped CFOAM-17[®] samples were not successful since a very low density of this material yielded insufficient mass of the test samples. To overcome this problem, CFOAM-17[®] was milled to a fine powder and 1g of the powder was placed into a platinum crucible with a diameter of

1.25 mm. Next, a forming tool was used to compress the powder inside the platinum crucible to the thickness of approximately 4mm. Compaction of the powder gave rise to an improved heat transfer and improved experimental accuracy so that a typical error range was about $\pm 3\%$.

2.1.3. Thermal expansion measurements

According to the Eq. (1), the computation of thermal conductivity from thermal diffusivity entails the knowledge of both heat capacity and density, both as a function of temperature. Variation of the density of CFOAM-17[®] with temperature is determined via the experimental measurements of linear thermal expansion in a temperature range between the room temperature and 2273K. Linear expansion, which is defined as a percentage change in a linear dimension of the test sample caused by a temperature change relative to the room temperature, is found to be nearly isotropic and hence only its values averaged in the three orthogonal directions are represented here. The averaged thermal expansion values are next used to compute the test sample volume at a given temperature. The current apparent density is then obtained by dividing the current sample mass by its current volume.

Determination of thermal expansion for CFOAM-17[®] studied in the present work has been done as a function of temperature using a Netzsch Single-sample Dilatometer Model DIL 402E. The furnace in this instrument is rated to 3073K, and an optical pyrometer is used to determine the sample temperature at temperatures above 800K. At lower temperatures, a type S thermocouple is used in lieu of an optical pyrometer since the pyrometer does not accurately determine the temperature at temperatures below 800K. The instrument was calibrated by validating the values of thermal expansion for a standard material. A standard graphite sample was used in this calibration.

A typical test sample used for determination of thermal expansion was rod-like in shape with a 1.25 cm diameter and 4 cm length.

2.2. Computational method

In this section a brief overview is given of the effective thermal conductivity model for foam-like materials recently proposed by Grujicic et al. [9]. The model neglects the contribution of convection to the heat transfer through the foam since the internal structure of the foam partitions the gas residing within the foam into isolated packets and prevents a large-scale motion within the gas. Hence, the heat transfer through the foam is assumed to take place by: (a) conduction through the solid phase of the foam; (b) conduction within the gas-phase packets; and (c) radiation between ligament/cell walls of the foam. The mathematical formulation of the model then involves two coupled partial differential equations: one pertaining to (one-dimensional) transient heat conduction and the other pertaining to the radiation heat transfer between foam ligament/cell surfaces. The first equation includes a combined thermal conductivity of the solid and the gas phase and

their temperature and pressure dependences. The two equations are coupled since the first one includes a term pertaining to the gradient in the radiation heat flux while the second involves temperature which is the dependent variable defined by the first differential equation.

When the two equations are subjected to the appropriate initial and boundary conditions, and solved using a numerical method, one can calculate the steady state heat flux through a foam sample. From the knowledge of the temperature difference across the sample thickness and the steady-state heat flux, the effective thermal conductivity of the foam material is calculated as a function of pressure and (mean) temperature. This quantity represents the value of thermal conductivity that the foam material would have to possess in order to yield the same value of the steady-state heat flux under a condition when the only mechanism for heat transfer through the foam is conduction. Since, the laser flash method described in Section 2.2.1 presumes that heat conduction is the sole mechanism for heat transfer through the test sample, the effective thermal conductivity values based on the model of Grujicic et al. [9] are calculated and compared with their experimental counterparts obtained using the laser flash method described in Section 2.1.1.

3. Results and discussion

3.1. Experimental measurements

In this section, a brief overview and discussion are given of the experimental results obtained for thermal diffusivity, heat capacity and thermal expansion in four heat treatments of CFOAM-17[®]. Refer to Table I for the graphitization temperatures for the heat treatments designated A through D corresponding to ranging from 1473K through 2773K, respectively.

3.1.1. Thermal diffusivity

The effect of test temperature on thermal diffusivity in three heat treatments of CFOAM-17[®] is shown in Fig. 1. CFOAM-17[®] graphitized at the highest temperature of 2773K (heat treatment D) was not tested since our preliminary experiments showed that the temperature dependence of thermal diffusivity in this material was quite similar to that observed in the case of heat treatment C.

The results displayed in Fig. 1 show that, in general, thermal diffusivity increases with an increase in temperature at temperatures above ~ 1300 K. At lower temperatures, thermal diffusivity is either fairly insensitive to the test temperature (heat treatment A) or decreases with an increase with temperature (heat treatments B and C). These results will be discussed later in conjunction with the effect of test temperature on the apparent density of CFOAM-17[®] in different heat-treatment conditions.

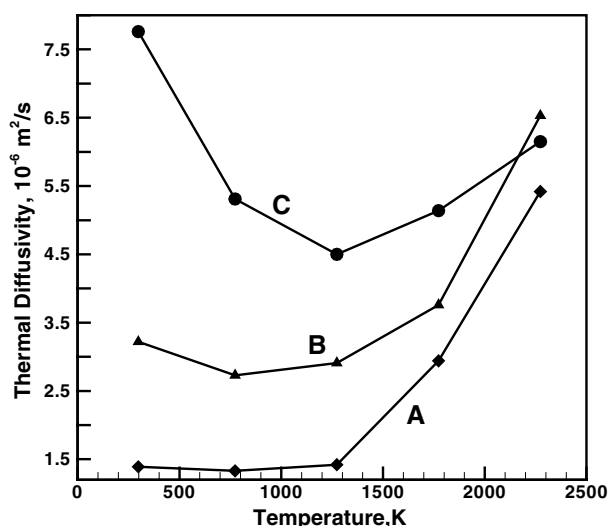


Figure 1 The effect of test temperature on the thermal diffusivity in three different heat treatments of CFOAM-17[®]. Please see Table I for explanation of heat-treatment designations (A–C).

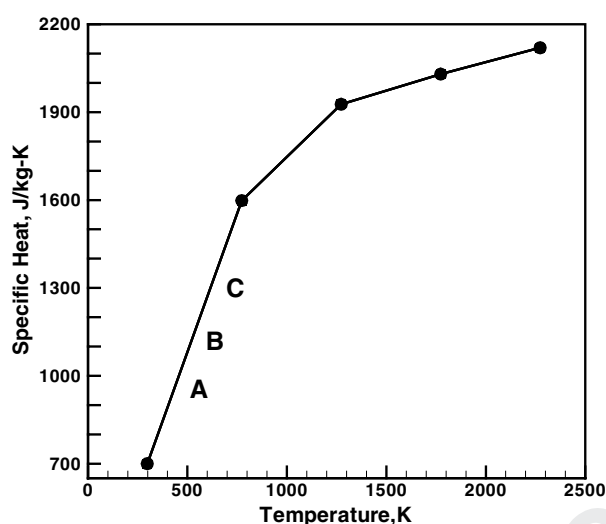


Figure 2 The effect of test temperature on the heat capacity in three different heat treatments of CFOAM-17[®]. Please see Table I for explanation of heat-treatment designations (A–C).

3.1.2. Heat capacity

The effect of test temperature on heat capacity in three heat treatments A, B and C of CFOAM-17[®] is shown in Fig. 2. It should be noted that since only one set of measurements was carried out no direct estimation of the standard deviation could be made. However, the technique used is generally assessed to be accurate within $\pm 5\%$. CFOAM-17[®] with the heat-treatment designation D was not tested for the same reasons explained in Section 3.1.1.

The results displayed in Fig. 2 show that the graphitization temperature has a negligible effect on heat capacity of CFOAM-17[®] and its temperature dependence. This finding is consistent with the previous observations [8] that heat capacity is essentially a microstructure insensitive property in carbon-base foam-like materials. In the temperature range examined, heat capacity of the CFOAM-17[®] is dominated by its lattice (phonon) contribution which is evidenced by the shape of the C_p vs. T curves in Fig. 2. The fact that heat ca-

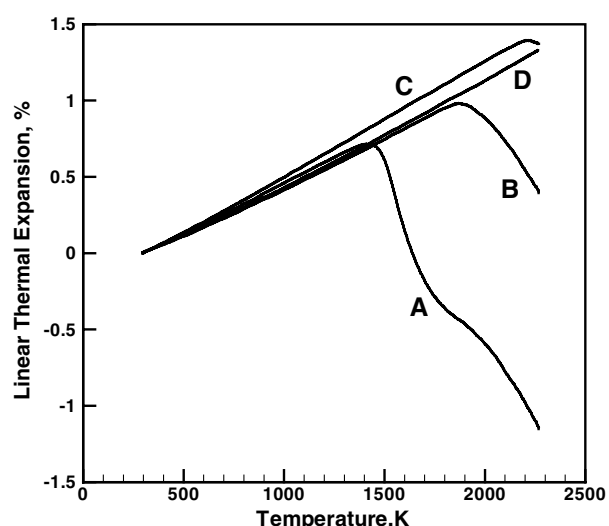


Figure 3 The effect of test temperature on the linear thermal expansion in three different heat treatments of CFOAM-17[®]. Please see Table I for explanation of heat-treatment designations (A–D).

capacity does not level off at the highest temperature but rather continues to increase with an increase in temperature (although at a progressively decreasing rate), in an indication of the contribution of lattice anharmonicity to the heat capacity at very high temperatures.

3.1.3. Thermal expansion

The effect of test temperature on linear expansion in all four heat treatments of CFOAM-17[®] is shown in Fig. 3. As discussed in Section 2.1, thermal expansion measurements were carried out in order to determine the effect of temperature on the apparent density of the material, since this information is needed, according to Eq. (1), to compute thermal conductivity and its dependence on test temperature. To determine the apparent density of CFOAM-17[®] at a given test temperature, the current test specimen volume is first computed using specimen dimensions corrected for the effect of thermal expansion. Next, the current specimen mass is divided by its current volume. The resulting apparent density data for four heat treatments of CFOAM-17[®] at temperatures between the room temperature and 2773 K are shown in Fig. 4.

The results displayed in Figs 3 and 4 show that only for the CFOAM-17[®] with a heat-treatment designation D, the thermal expansion increases and the apparent volume decreases monotonically with an increase in temperature. In the case of other heat treatments, the variation of these quantities with test temperature is more complicated. It should be recalled that only in the case of heat treatment D, the graphitization temperature (2773 K) is higher than the maximum test temperature (2273 K).

For the heat treatment designation C (graphitization temperature = 2273K), the thermal expansion and the apparent density vary monotonically up to a temperature which is only 50 K below the calcinations temperature. The fact that only in the last 50K the apparent density decreases with an increase in test temperature suggests that, at these temperatures, graphitization of

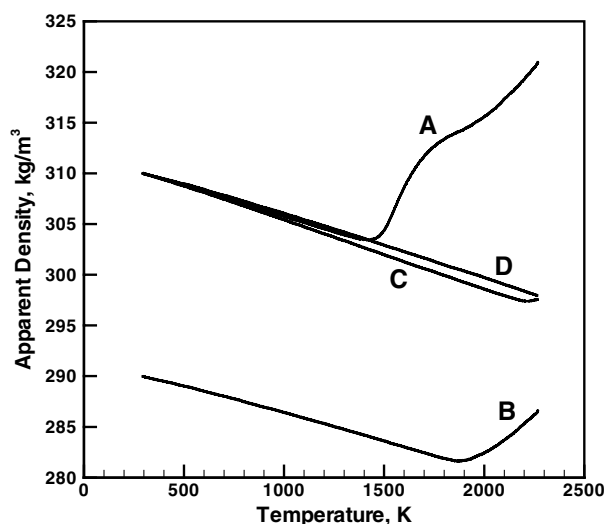


Figure 4 The effect of test temperature on the apparent density in three different heat treatments of CFOAM-17[®]. Please see Table I for explanation of heat-treatment designations (A–D).

the material resumes leading to a more regular arrangement and denser packing of the graphene platelets in the CFOAM-17[®]. This conjecture is further supported by the results for heat treatments A and B displayed in Figs 3 and 4. In general it is seen in Figs 3 and 4 that if CFOAM-17[®] is heat treated at a lower temperature, that the reorganization and packing of the graphene platelets start at a lower temperatures giving rise to a progressively lower (and ultimately a negative) thermal expansion and to an increase in the material density.

These findings are generally consistent with the results of a previous experimental investigation carried out by Morgan [14]. Morgan studied graphitization behavior of carbon foams produced from three types of precursors: (a) a urethane foam, (b) petroleum pitch and (c) coal. In the case of all three precursors, the foam is produced through calcination, a heat treatment in an inert atmosphere at a temperature around 1473K. The resulting carbonized solid material of the foam consists of a completely random distribution of the graphene platelets. This type of material is generally referred to as “turbostratic carbon”, to be distinguished from graphite in which graphene layers are stacked regularly in an ABAB... sequence.

The tendency of the turbostratic carbon to undergo a graphitization phase transformation during the subsequent heat treatment is greatly affected by the nature of the carbon precursor. In the case of urethane precursors, the carbonized material is quite “hard” and does not readily graphitize. Conversely, in the case of petroleum pitch precursors, the turbostratic carbon is quite “soft” and can readily graphitize. Coal-based foams, on the other hand, are found to have an intermediate graphitization tendency.

Franklin [11] proposed a simple and convenient X-ray diffraction based method for assessing the degree of graphitization, p , in the carbonized material as:

$$d_{(002)} = 0.344 - 0.0086(1 - p^2) \quad (2)$$

where $d_{(002)}$ is the (002) inter-planar spacing in nanometers.

According to Eq. (2), the higher is the degree of graphitization, the smaller is the (002) inter-planar spacing, i.e. the larger is the material density.

A detailed X-ray characterization of the effect of test temperature and the graphitization temperature on the (002) inter-planar spacing is being currently carried out by the authors and the results of this study will be reported in a future correspondence. The preliminary X-ray results obtained in this ongoing work support our previous contention that an increase in the degree of graphitization and the resulting reduction in the (002) inter-planar spacing during heating, are indeed responsible for the observed increase in the apparent material density in the case of CFOAM-17[®] in heat treatments A, B and C, Fig. 4. The reduction in the (002) inter-planar spacing with an increase in the extent of graphitization is not unique to CFOAM-17[®] but has been rather observed in a number of carbon-based materials (e.g. [8]).

The results displayed in Fig. 4 show that the density of CFOAM-17[®] in the heat treatment condition B is lower by about 7% than its counterparts in the other three heat treatments. This is simply a reflection of the natural variation in the material density from one batch to another.

The use of CFOAM[®] in the advanced TPS applications entails a consideration of not only thermal but also of mechanical and thermo-mechanical properties of this material. The thermal expansion data shown in Fig. 3 are hence used to compute the average coefficient of (linear) thermal expansion (CTE) as:

$$CTE = \frac{\Delta L/L_o}{\Delta T} \quad (3)$$

where ΔL is a change in the linear dimension of the test sample relative to a reference temperature, L_o is the reference-temperature value of the linear dimension, and ΔT is the temperature change relative to the reference temperature. The room temperature is selected as the reference temperature in the present work.

The computed values of CTE for the four heat treatments of CFOAM-17[®] are shown in Fig. 5. The results displayed in Fig. 5 show that in all four heat treatments of CFOAM-17[®], before an additional graphitization takes place, the mean coefficient of thermal expansion is about $6 \times 10^{-6} \text{K}^{-1}$ and increases very little with an increase in test temperature. In heat treatments A, B and C, however, when graphitization resumes and graphene platelets begin to pack more closely and regularly, the mean coefficient of thermal expansion begins to decrease (and ultimately becomes negative in the case of heat treatments A and B).

3.2. Effective thermal conductivity

The thermal diffusivity data displayed in Fig. 1, the heat capacity data displayed in Fig. 1 and the apparent density data displayed in Fig. 4 are next used in conjunction with Eq. (1) to compute the effect of test

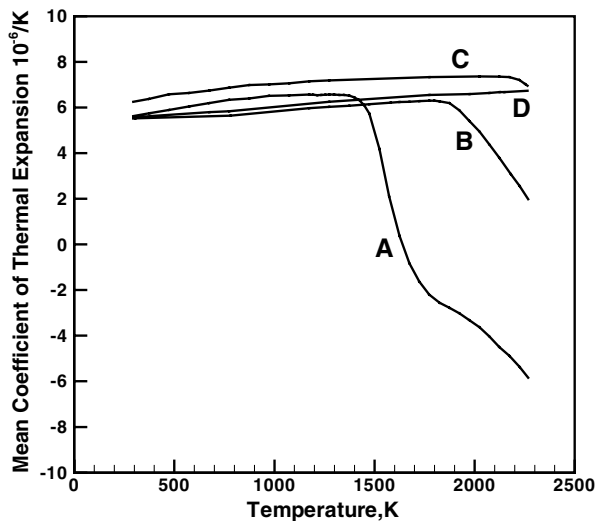


Figure 5 The effect of test temperature on the mean coefficient of thermal expansion in three different heat treatments of CFOAM-17[®]. Please see Table I for explanation of heat-treatment designations (A–D).

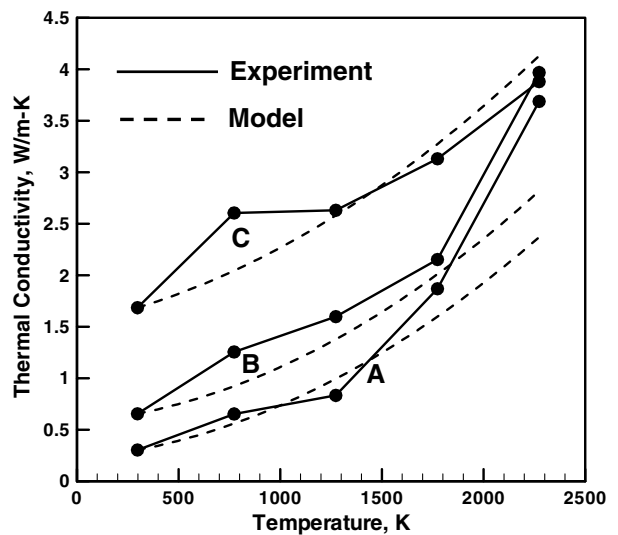


Figure 6 The effect of test temperature on the thermal conductivity in three different heat treatments of CFOAM-17[®]. Please see Table I for explanation of heat-treatment designations (A–C).

temperature on thermal conductivity of CFOAM-17[®] in heat-treatment conditions A, B and C. The results of this calculation are shown in Fig. 6. It should be noted that since only one set of measurements was carried out no direct estimation of the standard deviation could be made. However, the technique used is generally assessed to be accurate within $\pm 5\%$. Along with these experiment-based results, also shown in Fig. 6 are the thermal conductivity vs. test temperature results predicted by the model of Grujicic et al. [9]. As explained in Ref. [9], the necessary input parameters to the model are: thermal conductivity of the carbonized solid phase of the foam, the average diameter of the foam ligaments, and the relative porosity of the foam. A careful examination of scanning electron micrographs of the CFOAM-17[®] microstructure in the three heat treatment conditions revealed that the mean ligament diameter and the foam porosity are not visibly affected by the choice of the heat treatment. Hence these two parameters were assigned the values 0.3mm and 88% [8]. As far as thermal conductivity of the solid phase of the foam is concerned, this quantity is affected by the degree of graphitization. However, since the degree of graphitization was not measured in the present work, the values of thermal conductivity of the solid phase of the foam are selected for each of the three heat treatment conditions in such a way that the room temperature thermal conductivity predicted by the model is equal to its experimental counterpart. Then the model is applied to predict thermal conductivity of the CFOAM-17[®] as a function of test temperature in the three different heat treatment conditions. In these calculations, thermal conductivity of the solid phase of the foam is taken to be temperature invariant and the predicted effect of test temperature on thermal conductivity arises from the effect of temperature on thermal conductivity of air residing within the pores of the foam and from an increased radiation at elevated temperature. It should be recalled that due to the fact that heat is transferred through CFOAM-17[®] by conduction and radiation, thermal conductivity considered here is, in

fact, the effective thermal conductivity as defined in Section 2.2.

A comparison of the experiment-based and the model-based results in Fig. 6 shows that the model successfully predicts the effect of test temperature on thermal conductivity only in the case of CFOAM-17[®] heat treated at the highest temperature (2273K), the heat treatment designation C. In the case of heat treatments A and B, the model underpredicts the effect of test temperature on thermal conductivity. These findings are consistent with those reported in discussed in Section 3.1 and can be rationalized as follows: In the case of heat treatment C, the graphitization temperature is equal to the highest test temperature and, hence, additional graphitization does not take place to a significant extent during testing at elevated temperatures. Since the model of Grujicic et al. [9] assumes that the degree of graphitization remains constant, the agreement between the model and the experiment for the heat treatment C is reasonably good. In sharp contrast, in the case of heat treatments A and B, significant additional graphitization takes place during testing. Since such graphitization increases thermal conductivity of the solid phase of the foam, and the model does not account for this change, the model predicts a significantly smaller increase in thermal conductivity with an increase in test temperature relative to such increase observed experimentally.

The results displayed in Fig. 6 show that the thermal conductivity of CFOAM-17[®] heat treated at lower temperatures is undergoing a substantial change during experimental investigation due to the attendant increase in the degree of graphitization of this material. Therefore, thermal properties like thermal conductivity of CFOAM-17[®] heat treated at lower temperatures and measured during the first testing thermal cycle represent tentative properties of this material. To distinguish between these tentative properties and their counterparts in the CFOAM-17[®] heat treated at high temperatures, the former should be labeled as the “apparent” thermal properties of CFOAM-17[®]. To demonstrate

that the thermal properties of CFOAM-17[®] have indeed changed during the first testing cycle, these properties are redetermined at the room temperature upon the completion of the first testing cycle. The results obtained indicated that the room-temperature thermal conductivity has been increased to the levels essentially identical to those found in the CFOAM-17[®] heat treated at high temperatures. This finding suggests that the effect of thermal-property testing in CFOAM-17[®] heat treated at lower temperatures was nearly identical to the effect of the initial heat treatment in CFOAM-17[®] heat treated at high temperatures. In other words, the attractive low thermal conductivity properties of the CFOAM-17[®] heat treated at lower temperatures are essentially lost during the first thermal cycle.

The results displayed in Fig. 6 show that the values of thermal conductivity in CFOAM-17[®] fall into a range between 0.25 and 4W/m-K. Considering the fact that thermal conductivity of CFOAM-17[®] is found to increase as a result of a high temperature exposure (which is generally viewed as quite undesirable) and that thermal conductivity of (high-purity polycrystalline-alumina fibers-based) Saffil[®] insulation material currently used in metallic TPS panels [12] is about 0.06W/m-K, CFOAM-17[®] does not appear, at the first glance, to be a viable candidate for thermal protection applications in the space vehicles. However, in addition to offering a relatively low level of thermal conductivity, CFOAM-17[®] also offers a good combination of high temperature mechanical properties. Therefore, some of the structural components in the current design of the metallic TPS panels such as the structural brackets [13] which are responsible for carrying the major portion of the aerodynamic and acoustic load can be downsized or even eliminated. Since such structural components are generally made of metal-based super-alloys such as Inconel 617, and act as heat shorts in a typical TPS panel, their downsizing or elimination could potentially compensate for the observed higher values of thermal conductivity in CFOAM-17[®]. In other words, the final decision regarding the suitability of CFOAM-17[®] for use in the advanced thermal protection systems will be based not solely on the level of thermal conductivity but rather on a consideration of the entire spectrum of thermal, thermo-mechanical, mechanical and physical properties of this material.

4. Conclusions

Based on the results obtained in the present work, the following main conclusions can be drawn:

1. Both the test temperature and the temperature of a graphitization heat treatment have a significant effect on thermal properties (thermal conductivity and thermal diffusivity) of the coal-derived carbon-based foam, CFOAM-17[®].

2. When the graphitization temperature is relatively low, the degree of graphitization is smaller and

CFOAM-17[®] has lower thermal conductivity and thermal diffusivity values. However, when these properties are experimentally measured at temperatures comparable to or higher than the graphitization temperature, the graphitization process tends to take place during testing giving rise to an increase in the material's ability to conduct heat.

3. The range of thermal conductivity values ($k = 0.25 - 4.0\text{W/m-K}$) obtained in CFOAM-17[®] graphitized at different temperatures and the tendency of the material to undergo an additional graphitization phase transformation during high temperature exposure does not suggest, at a first glance, that this material is a strong candidate for insulation applications in the advanced thermal protection systems. However, the fact that CFOAM-17[®] has a relatively high strength and is able to retain it at high temperatures could provide opportunities for new designs of the metallic TPS panels. Therefore, the use of CFOAM-17[®] in the advanced thermal protection systems is still being considered.

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