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### Thermomechanical behavior of a graphite foam

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#### Abstract

The thermomechanical behavior of a graphite foam derived from pitch for use in thermal management was studied in air up to 150 °C. The damping capacity or loss tangent under flexure was 0.17 at 30 °C for the graphite foam, compared to 0.02 for conventional graphite (not a foam), 0.15 for flexible graphite and 0.22 for PTFE. The loss tangent of the graphite foam decreased with increasing temperature, whereas that of conventional graphite did not. The compressive strain of the graphite foam strongly depended on time, compressive stress and temperature. Due to creep at 30 °C, it reached 3% at 200 kPa, in contrast to 0.7% for conventional graphite. Thermal softening increased the compressive strain in the graphite foam upon heating and subsequent cooling, such that the thermal expansion phenomenon was overshadowed. In contrast, thermal softening was less in conventional graphite. The storage modulus of the graphite foam under flexure was lower than that of conventional graphite. Its fractional decrease with increasing temperature was more than that of conventional graphite.

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#### 1. Introduction

Monolithic forms of porous graphite are useful for thermal management, due to their high thermal conductivity, low thermal expansion coefficient, low density, open porosity and chemical resistance. Applications relate to vehicle radiators, satellite panels, heat exchangers for refrigerators and air conditioners, engine cooling, power electronics cooling, brake and clutch cooling, oven wall lining, etc. In particular, the open-cell structure is attractive for heat transfer from a solid to a fluid. Porous graphite is to be distinguished from porous carbon, which is not graphitic in crystal structure and in consequently lower in thermal conductivity.

A monolithic form of porous graphite is graphite foam [1-11] with an open microcellular structure, such that the foam ligaments are mainly graphitic and have thermal conductivity estimated to be between 700 and 1200 W/m

K [3]. The material is derived from pitch [1–11]. The vertical direction during foaming is referred to as the out-of-plane direction. The bulk thermal conductivity of the graphite foam of Ref. [3], as manufactured by Poco Graphite, Inc. (Decatur, TX), is shown in Table 1, along with other properties (http://www.poco.com). Because of the low density of 0.5 g/cm³, the specific thermal conductivity is more than four times greater than that of copper [3].

Although the oxidation resistance of graphite foam, as shown by weight loss measurement, is good up to  $400\,^{\circ}\mathrm{C}$  in oxygen (http://www.poco.com), the thermomechanical behavior of this material, even at temperatures much below  $400\,^{\circ}\mathrm{C}$ , is of concern. This concern is due to the high porosity and sponge-like structure, which are expected to result in viscoelastic behavior. Thus, this paper is focused on the thermomechanical behavior of graphite foam from 25 to  $150\,^{\circ}\mathrm{C}$ . This temperature range is relevant to the majority of heat transfer applications.

The thermomechanical behavior of graphite foam is investigated in this work by studying: (i) the effects of compressive stress (in the out-of-plane direction) and temperature on the out-of-plane dimension; and (ii) the effect of temperature on the dynamic flexural behavior

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Table 1
Properties of graphite foam and conventional graphite

	Graphite foam	Conventional graphite
Thermal conductivity (W/m K) <sup>a</sup>	135 (out-of-plane)	95
	45 (in-plane)	
Electrical resistivity $(\Omega \text{ cm})^a$	$2\times10^{-3}$ $\Omega$ .cm (in-plane)	$1.47 \times 10^{-3}$
Oxidation threshold (°C) <sup>a</sup>	400	450
Heat capacity <sup>a</sup>	0.7 J/g K	/
Grain size (µm) <sup>a</sup>	/	5
Average pore diameter (µm) <sup>a</sup>	350	0.8
Total porosity (% volume) <sup>a</sup>	75	20
Open porosity (% of total) <sup>a</sup>	96	80
Density (g/cm <sup>3</sup> )	0.55°	1.79 <sup>b</sup>
Coefficient of thermal expansion (°C <sup>-1</sup> )	$-0.7\times10^{-6}$ (out-of-plane)	$7.9 \times 10^{-6a}$
	$0.6\times10^{-6}$ (in-plane)	
Compressive strength (MPa)	1.8±0.1 (out-of-plane) <sup>b</sup>	138°
Graphitization temperature (°C)	2800	2400

<sup>&</sup>lt;sup>a</sup> From web site (http://www.poco.com).

(both the storage modulus and the loss tangent, as tested in three-point bending). The loss tangent is the same as the damping capacity. Damping is desirable in most applications.

#### 2. Experimental methods

The graphite foam was kindly provided by Poco Graphite (Decatur, TX, USA). It was in the shape of a rectangular slab. The material properties are shown in Table 1. Specimens for testing were obtained by cutting from the same slab. For the sake of comparison, testing was also conducted on conventional graphite (AXF-5Q) supplied by Poco Graphite. The basic properties of both graphite foam and conventional graphite are shown in Table 1.

The Perkin-Elmer Corporation (Norwalk, CT, USA) Series 7 Thermal Analysis System was used for testing the graphite foam and conventional graphite. The thermomechanical analyzer (TMA7) in the system was used to measure the out-of-plane dimension (thickness) vs. time of application of various compressive stresses ranging from 40 to 200 kPa in the thickness direction at 30 °C. After this test at 30 °C, each specimen (7.5×7.0×2.5 mm) was heated to 150 °C at a heating rate of 5 °C/min and then cooled, while a constant compressive stress was applied in the thickness direction using a cylindrical quartz probe of diameter 4 mm and the specimen thickness was measured by using the probe in conjunction with TMA7.

For investigating the effect of temperature on the dynamic flexural behavior, the dynamic mechanical analyzer (DMA7, ASTM D4065-94) in the system was used during heating and subsequent cooling, both at a rate of 5 °C/min, under three-point bending, with a span of 20 mm and a controlled frequency of 1.00 Hz. Measurements

of tan  $\delta$  and storage modulus were made simultaneously as a function of temperature. The specimens were in the form of beams (24×7.5×2.5 mm). The heating rate was selected to prevent any artificial damping peaks which may be caused by higher heating rates. The loads used were all large enough so that the amplitude of the specimen deflection was always over the minimum value of 5  $\mu$ m required by the equipment for accurate results. The deflection was typically under 10  $\mu$ m. The loads were set so that each different type of specimen was always tested at its appropriate stress level.

The effect of heating on the compressive strength (out-of-plane) and the volume electrical resistivity (in-plane) was studied by measuring these quantities before and after heating, which was conducted by heating in air at a rate of 5 °C/min from room temperature to 150 °C, holding at 150 °C for 1 min and then cooling to room temperature at a rate of 5 °C/min. This heating—cooling schedule was also used in tests using TGA7, TMA7 and DMA7. Compressive testing was conducted on specimens of size  $7.5\times7.0\times2.5$  mm, using a screw-action mechanical testing system (Sintech 2/D, MTS Systems Corp., Stoughton, MA, USA).

Resistivity testing was conducted on specimens of size  $50\times7.0\times2.0$  mm, using the four-probe method. Electrical contacts, in the form of silver paint in conjunction with copper wires, were applied perimetrically at four planes perpendicular to the longest dimension of the specimen (e.g. perpendicular to the current direction). The outer two probes (for passing current) were 40 mm apart and the inner two probes (for voltage measurement) were 30 mm apart. The four probes were symmetrically positioned relative to the mid-point of the longest dimension of the specimen. A Keithley 2002 multimeter was used. The resistivity obtained for the graphite foam was an apparent resistivity, as the high porosity caused the current penetration from the surface electrical contacts to be limited.

<sup>&</sup>lt;sup>b</sup> From this work.

At least three specimens were tested for each type of test, except that only two specimens were tested for the resistivity before heating and two other specimens were tested for the resistivity after heating.

#### 3. Results and discussion

#### 3.1. Apparent resistivity and compressive strength

The apparent resistivity of graphite foam before and after heating (up to  $150\,^{\circ}\text{C})$  was  $0.099\pm0.004$  and  $0.115\pm0.004$   $\Omega$  cm, respectively. The compressive strength of graphite foam before and after heating was  $1.77\pm0.10$  and  $1.76\pm0.10$  MPa, respectively. Hence, the heating had no effect on the compressive strength and essentially no effect on the apparent resistivity. Note that the apparent resistivity was much higher than the true resistivity in Table 1, due to the limited current penetration from the surface electrical contacts into the cross-section of the specimen.

## 3.2. Effect of compressive stress and its time of application on the dimension

Fig. 1 shows the effect of the time of compressive stress application on the dimension (i.e. the thickness) of graphite foam in the stress direction for fixed compressive stresses ranging from 40 to 200 kPa at 30 °C. The fractional drop in thickness at any time increased with increasing stress. At a fixed stress, the thickness decreased abruptly at the start of stress application and leveled off as the time of

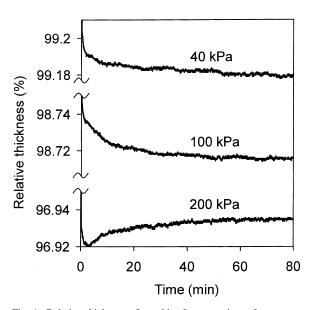


Fig. 1. Relative thickness of graphite foam vs. time of compressive stress application at  $30\,^{\circ}\text{C}$  for three levels of stress.

application increased. This indicates the occurrence of creep at 30 °C. At a fixed stress of 200 kPa, the thickness bounced back partially after the initial abrupt thickness drop. At a lower fixed stress of 40 or 100 kPa, the thickness dropped monotonically without any rebound. The rebound observed at 200 kPa is attributed to the viscoelasticity in the graphite foam.

Fig. 2 shows corresponding data for conventional graphite. The thickness dropped as in Fig. 1, but the effects were much smaller, as shown by the difference in vertical scale between Figs. 1 and 2. Thus, graphite foam was much more prone to compressive deformation and much more viscoelastic than conventional graphite. This behavior of graphite foam is believed to be due to the foam macrostructure, together with the shear between the crystallites in the ligaments.

## 3.3. Effect of heating and compressive stress on the dimension

Fig. 3 shows the effect of heating and subsequent cooling in air on the dimension (i.e. the thickness) of graphite foam in the stress direction for a fixed compressive stress of 40 kPa. The measurement in Fig. 3 was conducted on the same specimen immediately after completion of the measurement at 40 kPa in Fig. 1. The thickness had stabilized at 30 °C at the end of the measurement at 40 kPa in Fig. 1, but subsequent heating in Fig. 3 caused the thickness to further drop, due to softening upon heating (Section 3.4). However, the frac-

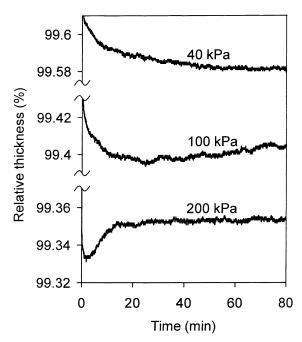


Fig. 2. Relative thickness of conventional graphite vs. time of compressive stress application at 30 °C for three levels of stress.

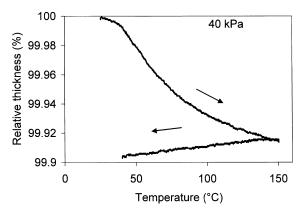


Fig. 3. Relative thickness of graphite foam vs. temperature during heating and subsequent cooling at a fixed compressive stress of 40 kPa.

tional drop in thickness in Fig. 3 is much less than that in Fig. 1. The thickness dropped monotonically with increasing temperature in Fig. 3. During subsequent cooling, the thickness continued to drop at the same rate, suggesting viscoelastic behavior. If the thickness drop upon cooling were due to thermal contraction, the coefficient of thermal expansion (CTE) would have been  $1.3 \times 10^{-6}$ , which is inconsistent with the out-of-plane CTE of  $-0.7 \times 10^{-6}$  (Table 1).

Fig. 4 shows corresponding result obtained for graphite foam at a compressive stress of 100 kPa. The measurement in Fig. 4 was conducted on the same specimen immediately after the measurement as a function of time at 100 kPa in Fig. 1. The fractional thickness drop in Fig. 4 is less than that in Fig. 3, in spite of the higher stress in Fig. 4. This is because the fractional thickness drop attained during the prior measurement at 30 °C and 100 kPa is larger than that attained during the prior measurement at 30 °C and 40 kPa (Fig. 1). The relatively large extent of

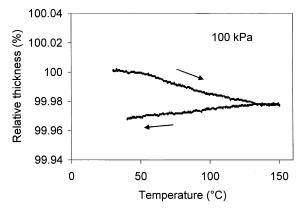


Fig. 4. Relative thickness of graphite foam vs. temperature during heating and subsequent cooling at a fixed compressive stress of 100 kPa.

prior thickness drop at 30 °C (Fig. 1) resulted in a relatively small thickness drop during the subsequent heating of the same specimen (Fig. 4).

Fig. 5 shows corresponding result obtained for conventional graphite at a compressive stress of 100 kPa. The measurement in Fig. 5 was conducted on the same specimen immediately after the measurement as a function of time at 100 kPa in Fig. 2. The thickness did not decrease upon heating, in contrast to the decrease in Fig. 4 for the case of the graphite foam. In Fig. 5, the thickness increased upon heating above about 100 °C and decreased upon subsequent cooling. The thickness increase is attributed to thermal expansion, which occurred in spite of the compressive stress of 100 kPa. In the case of the graphite foam, the thermal expansion was overshadowed by the compressive strain, thereby causing the thickness to decrease upon heating (Fig. 4). As shown in Section 3.4, the modulus decreased upon heating for graphite foam, such that the fractional decrease was more than for the conventional graphite.

#### 3.4. Effect of heating on the dynamic flexural properties

Fig. 6 shows that both the loss tangent ( $\tan \delta$ , i.e. the damping capacity) and the storage modulus of graphite foam decreased upon heating. The decrease in modulus is due to thermal softening, as expected due to the increase in ease of shear between the crystallites in the ligaments as the temperature increases. The decrease in loss tangent may be due to subtle microstructural changes which have not been identified.

Upon cooling, the measured modulus of graphite foam was higher than that during prior heating at the same temperature. This was an artifact, presumably due to the slight compressive deformation of the graphite foam at the three points involved in three-point bending. Therefore, the modulus and loss tangent during cooling are not shown in

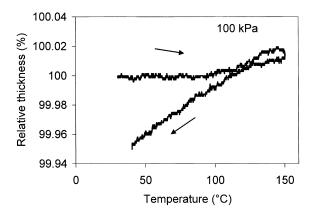


Fig. 5. Relative thickness of conventional graphite vs. temperature during heating and subsequent cooling at a fixed compressive stress of 100 kPa.

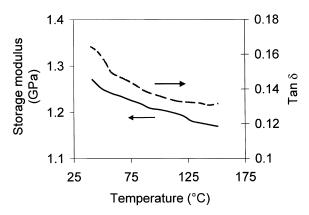


Fig. 6. Storage modulus vs. temperature and loss tangent ( $\tan \delta$ ) vs. temperature during heating of graphite foam.

Fig. 6. Since the artifact caused the measured modulus to increase, the observed decrease in modulus during first heating had to be real. On the other hand, the loss tangent, which recovered upon cooling, was much less affected by the presumed compressive deformation.

Fig. 7 shows corresponding results for conventional graphite. The modulus of conventional graphite (4.1 GPa) was higher than that of graphite foam (1.3 GPa) at any of the temperatures investigated, as expected. The modulus of conventional graphite decreased upon heating, as for graphite foam, but the fractional decrease  $((4.7\pm0.5)\%)$  was smaller than that of graphite foam  $((8.5\pm0.8)\%)$ .

The loss tangent (Fig. 7) of conventional graphite was lower than that of graphite foam at any of the temperatures investigated, as expected. The loss tangent of conventional graphite did not change with temperature, in contrast to the decrease with increasing temperature for graphite foam (Fig. 6).

The high value of the loss tangent (0.17 at 30 °C, compared to 0.15 for flexible graphite [12] and 0.22 for polytetrafluoroethylene or PTFE [13]) of graphite foam is

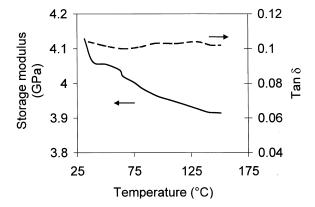


Fig. 7. Storage modulus vs. temperature and loss tangent ( $\tan \delta$ ) vs. temperature during heating of conventional graphite.

attractive for vibration damping and is consistent with the viscoelastic behavior observed (Fig. 1). In contrast, the loss tangent of conventional graphite is only 0.02 at 30 °C. It is significant that the loss tangent of graphite foam approached that of PTFE and exceeded that of flexible graphite. Both PTFE and flexible graphite were tested using the same method, frequency (1.0 Hz) and equipment as this work. Vibration damping is a new application for graphite foam, which is attractive for its combination of high damping capacity, low density, chemical resistance and low thermal expansion, in contrast to the high thermal expansion and limited chemical resistance of polymeric vibration damping materials.

#### 4. Conclusion

Graphite foam was found to be more prone to compressive strain, creep and thermal softening (in air up to 150 °C) than conventional graphite, in spite of its negligible change in compressive strength or electrical resistivity after heating at 150 °C. The compressive strain depended on time, compressive stress and temperature. At 30 °C, it reached 3% at 200 kPa, in contrast to 0.7% for conventional graphite. Subsequent heating under compression caused additional strain, which continued during still subsequent cooling, due to the viscoelastic behavior and the thermal softening. In contrast, thermal softening was less in conventional graphite, and consequently thermal expansion overshadowed the compressive strain. The thermal softening is supported by the observed decrease in the flexural storage modulus upon heating.

The loss tangent of graphite foam decreased upon heating, whereas that of conventional graphite did not change upon heating. Compared to conventional graphite, graphite foam exhibited low storage modulus (1.3 vs. 4.1 GPa) and high loss tangent (0.17 vs. 0.02). The loss tangent of graphite foam exceeded that of flexible graphite and approached that of PTFE.

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