INCREASED GRAPHITE FIBER DAMPING VIA INTERCALATION

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ABSTRACT

The damping of several commercial graphite fibers was experimentally characterized over a temperature range from -200°C to +400°C, for frequencies from about 50 to 2000 hz. A unique flexural test apparatus was used with *single* 10-micron diameter fibers. The baseline fibers exhibited peak damping ratios in the vicinity of 2×10^{-4}. Damping in some copper chloride- and bromine-intercalated P100 fibers was also measured over similar ranges of temperature and frequency—peak damping values were observed to increase by more than an order of magnitude. Results from the literature indicate that intercalation does not deleteriously affect other fiber engineering properties.

INTRODUCTION

In order to better observe and influence our world, the U.S. commercial and military presence in space will continue to expand. The future capabilities of the U.S. in strategic defense will rely on space assets with very demanding performance requirements. Many of these spacecraft will require precise payload pointing, rapid slewing, and extreme dimensional stability in severe mechanical and thermal environments, including enemy-induced hostile environments. Damping will be essential to the attainment of performance goals in these precision spacecraft. Arising, however, as the result of a multitude of complex unrelated physical mechanisms acting in concert, passive damping remains the least understood aspect of structural dynamic behavior.

In common built-up structures which operate in the atmosphere, air damping and joint damping typically dominate system damping. However, material damping will be an important contributor to damping in "monolithic" structures and to on-orbit damping in precision spacecraft. Air damping is clearly eliminated in space, and the effects of joint damping will be reduced because of requirements for precision ("tight" joints) and low vibration levels (friction "lockup").

Some effort has gone into the integration of high-damping *non-structural* materials with a control/structure design, but associated weight penalties, potential outgassing problems, and temperature sensitivity may limit their use. The development of high damping *structural* materials therefore represents a high-payoff research direction.

Graphite-reinforced composite materials are ideal for use in these structures because of their superior mechanical and thermal properties—high modulus, high thermal conductivity, low density, and low coefficient of thermal expansion are especially important. Constituent contributions to damping in a mechanically-perfect composite material are weighted by their contribution to strain energy.

When such composite materials are deformed, the high modulus of the carbon fibers relative to that of typical reinforcing matrix materials causes most of the strain energy (typically 80–90%) to be found in the fiber. Efforts to increase composite material damping are therefore best focused on the highly-leveraged carbon fibers [1].

A large number of graphite fibers are currently commercially available, and little is known in general about their damping properties. Much of what is known has been inferred from tests of built-up composite materials, although a few researchers have directly investigated the damping of single fibers. The authors have previously reported the results of an investigation of the damping of some commercial graphite fibers [2], as well as known related work [*c.f.*, 3, 4]. Some of the findings are repeated herein to provide context for new data on the damping of intercalated fibers.

EXPERIMENTAL PROCEDURE

The damping of baseline commercial and experimental intercalated graphite fibers was measured over a temperature range from -200°C to +400°C, for frequencies from about 50 to 2000 hz. High modulus fibers were emphasized because of their promise for used in advanced spacecraft. The baseline fibers included three mesophase pitch-based fibers of different modulus (P55, P100, P120), and one polyacrylonitrile (PAN)-based fiber having a modulus close to that of one of the pitch fibers (T50). Damping in some copper chloride- and bromine-intercalated P100 fibers was also measured over similar ranges of temperature and frequency.

All testing was performed at the NASA Lewis Research Center in Cleveland, Ohio. The test technique consisted basically of the forced flexural vibration and free decay of cantilevered fibers in a high-vacuum cryostat furnace. Figure 1 shows a schematic of the equipment configuration, and Figure 2 illustrates in more detail the means used to control the specimen

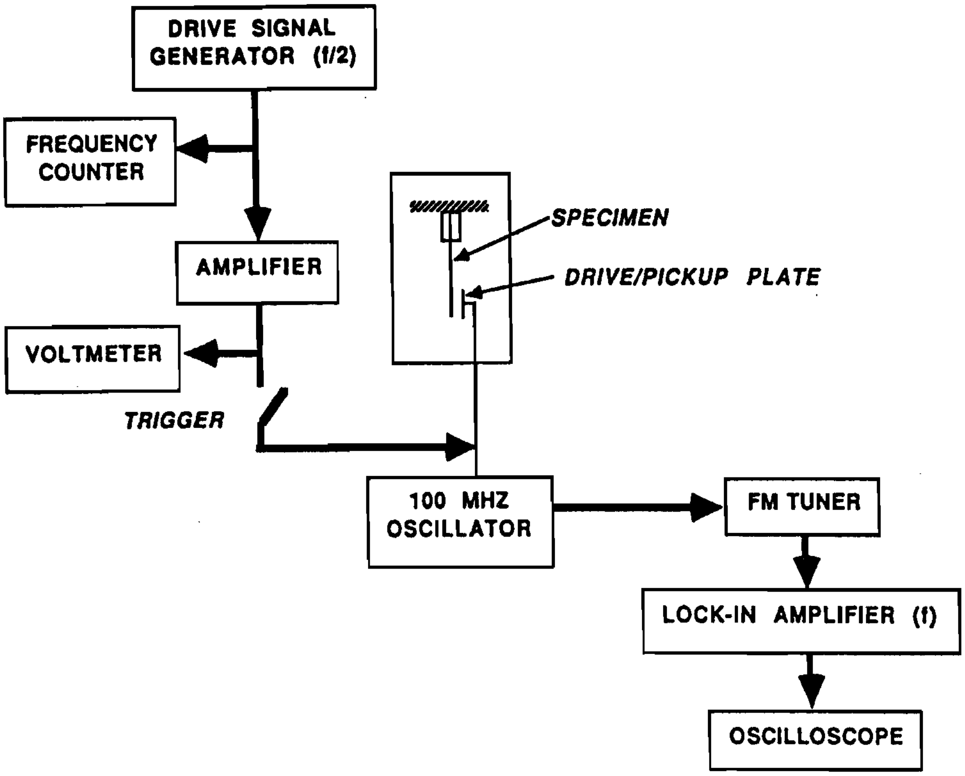

Figure 1: Schematic of the Test Apparatus

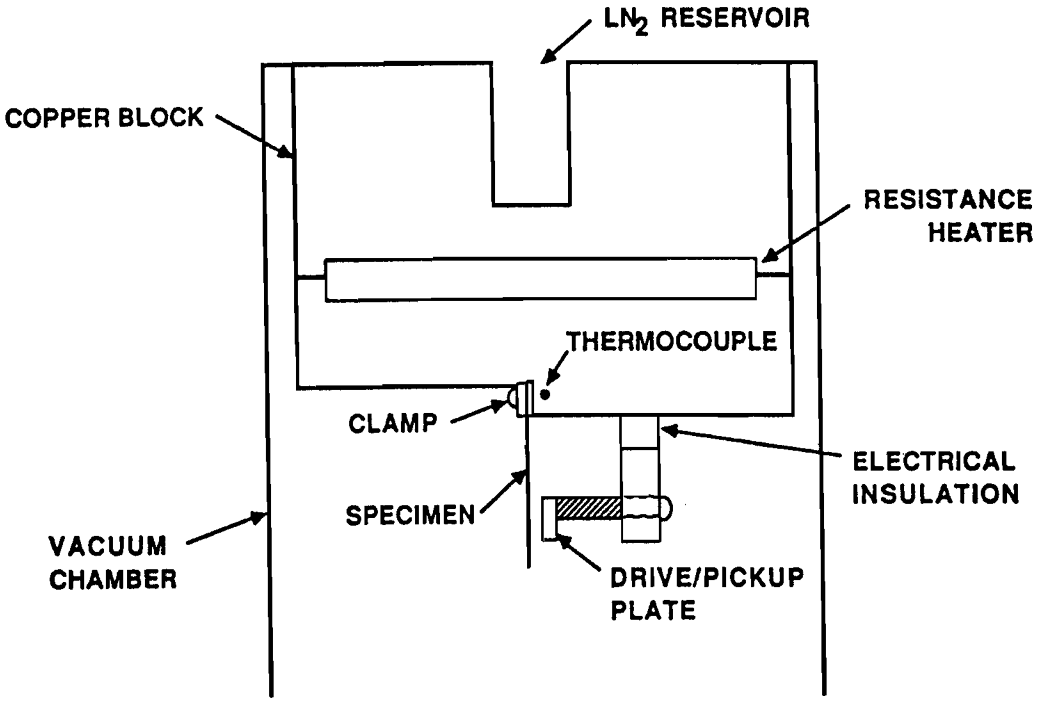

Figure 2: Detail of the Specimen Mount, Drive/Pickup, Vaccum, and Thermal Control

environment. The test procedure and some of the significant challenges posed by testing single graphite fibers are discussed in [2].

Observed material damping depends on the type of deformation experienced under load. An extensional or a flexural test method was required to generate data relevant to the deformation experienced by single fibers as part of a composite spacecraft structure. The flexural vibration test method was selected, because it had been successfully used at the NASA Lewis Research Center to characterize other small-diameter fibers and therefore represented less development risk [5]. The authors are unaware of any previous measurements of the damping of single graphite fibers in such an appropriate mode of deformation.

The tests were performed in a diffusion-pump vacuum (10^{-6} torr), as vibration response amplitudes were typically 200 times smaller in vacuum than at one atmosphere. Strain amplitudes at the fiber root surface were on the order of 10^{-6}, and no significant amplitude-dependence of damping was observed for amplitudes near this level.

DATA REDUCTION

MEASURES OF DAMPING

As a consequence of the second law of thermodynamics, no real structure can sustain unforced vibratory motion indefinitely. Many different approaches to quantifying this damping ability have been devised, *e.g.*, the time required for the displacement response amplitude to settle to some fixed fraction of its initial amplitude. The most useful of these damping measures, however, are dimensionless, and generally quantify the fractional displacement or energy loss per cycle or radian of vibration. A few of these dimensionless measures are shown below:

Ψ	Damping capacity
δ	Log decrement
Q	Quality factor
η	Loss factor
ζ	Damping ratio

Damping ratio is the most common measure of damping used by satellite designers and structural dynamicists. Because one of the purposes of this work is to make damping more accessible as a design property of advanced materials and structures, damping ratio is the unit in which all damping results are presented. For small damping, these results can be converted to other units with the use of the following scale factors.

$$\zeta = \eta/2 = 1/2Q = \delta/2\pi = \Psi/4\pi$$

LOG DECREMENT METHOD

As discussed in [2], a variation of the conventional log decrement method was used to determine damping ratios from fiber free decay data. In practice, the amplitude of vibration was read off photographs of decay traces at a number of equally-spaced time increments, instead of just two as in the conventional log decrement method, and a line was fit to the data. A computer program was written to automate this calculation, to determine the quality of the fit to the data, and to plot the data. In all, some 650 damping ratios were calculated for various fibers under various conditions, from 6000 measured data points. The great majority of data did fit a line well.

RESULTS

As reported in [2], the three baseline pitch fibers exhibited similar damping, with a peak damping ratio of just over 2×10^{-4} observed at a temperature of about -40°C, dropping off slowly at lower temperatures and more rapidly at higher temperatures. At 400°C, a typical damping ratio was found to be about 0.35×10^{-4}. The PAN fiber exhibited damping similar to the pitch fibers at high temperatures, but had only about a third the damping at lower temperatures. Figure 3 summarizes the damping and frequency results for the baseline fibers. Reference [6] contains complete data for each fiber and each test run.

Damping in the intercalated P100 fibers was measured over a similar range of temperatures and frequencies. Peak damping values increased by more than an order of magnitude, as shown in Figure 4. The P100/$CuCl_2$ fiber exhibited a peak damping ratio of nearly 3×10^{-3}, at a temperature of about 200°C. Irreversible processes at high temperatures resulted in a permanent loss of peak damping in this fiber (note the arrow on the data trace); this finding is consistent with environmental stability results published in the literature [7]. Brominated P100 fibers exhibited a peak damping ratio of about 2.3×10^{-3}, in the vicinity of -50°C. This behavior was relatively stable over the temperatures considered.

A second brominated fiber was tested to verify the results, and similar damping was observed. Because the results appeared to be characteristic of a point defect damping mechanism, the damping was measured in detail at two separate frequencies, over a range of temperatures. Figure 5 shows this damping data. A curve fit of the data yielded estimates of the temperatures at which peak damping is observed. Some key values are summarized below:

Mode	Frequency	Temperature(ζ_{peak})
A	76.7 hz	210.46 °K
B	465.4 hz	222.16 °K

By noting the temperature shift between the data taken at two frequencies, some characteristics of the physical mechanism underlying the observed damping were established. Based on the assumption of a point defect mechanism, an activation energy of 0.47 eV was estimated. The following equation was used to determine this [8]:

$$Q = k\,((T_1T_2)/(T_2-T_1))\,\ln(f_2/f_1)$$

Where:
Q is the activation energy
k is Boltzmann's constant
T_i is the temperature at which peak damping is observed at frequency f_i

In addition, a characteristic jump frequency ($\nu_0 = 1/\tau_0$) of about 1.1×10^{14} s^{-1} was determined. The assumption of a constant activation energy and jump frequency was found to be good, but not perfect. This information can be used to estimate fiber damping at any frequency and temperature via the following equations:

$$\zeta = \zeta_0\,(2\omega\tau) / (1 + (\omega\tau)^2)$$

with $$\tau = \tau_0\, e^{Q/kT}$$

Where:
ζ_0 is the peak damping ratio for the defect
ω is the radian frequency of vibration
τ_0 is the jump time constant for zero activation energy

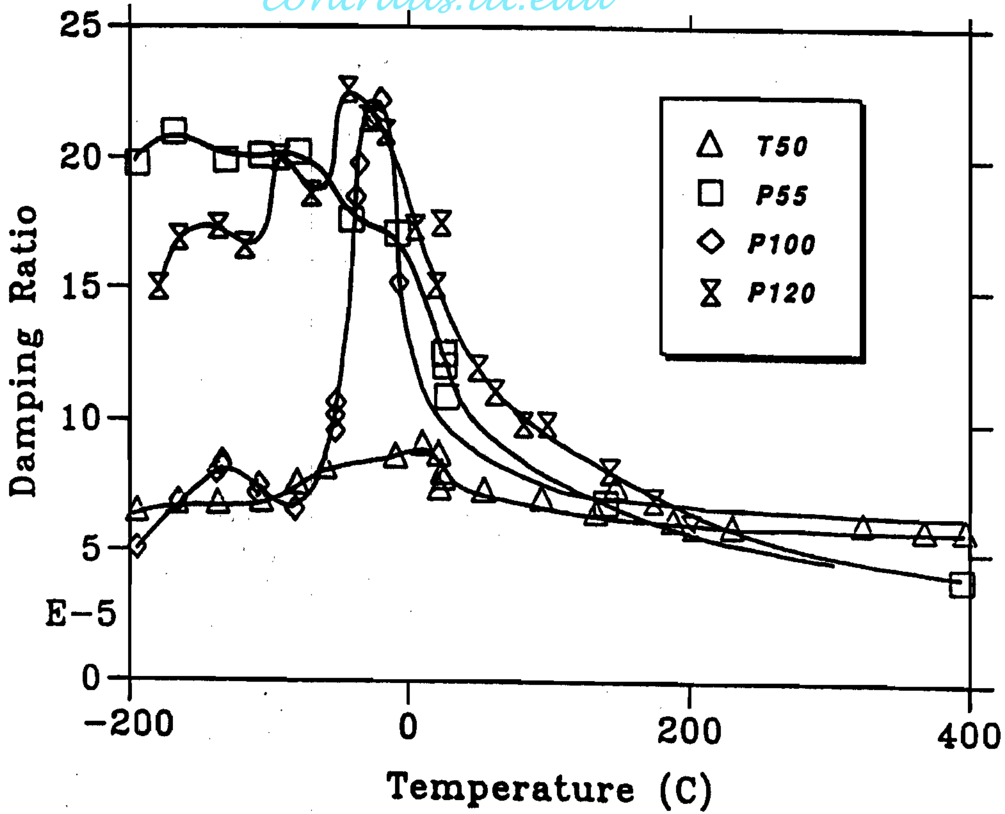

Figure 3(a): Baseline Fiber Damping versus Temperature

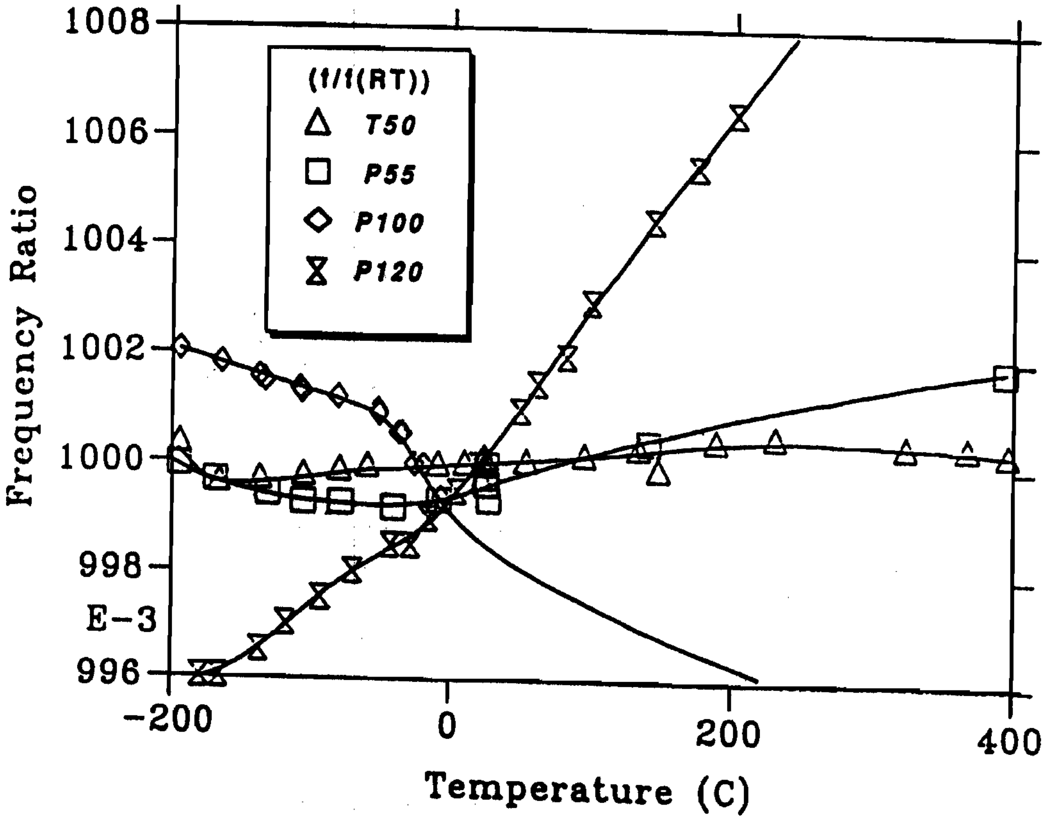

Figure 3(b): Baseline Fiber Frequency versus Temperature

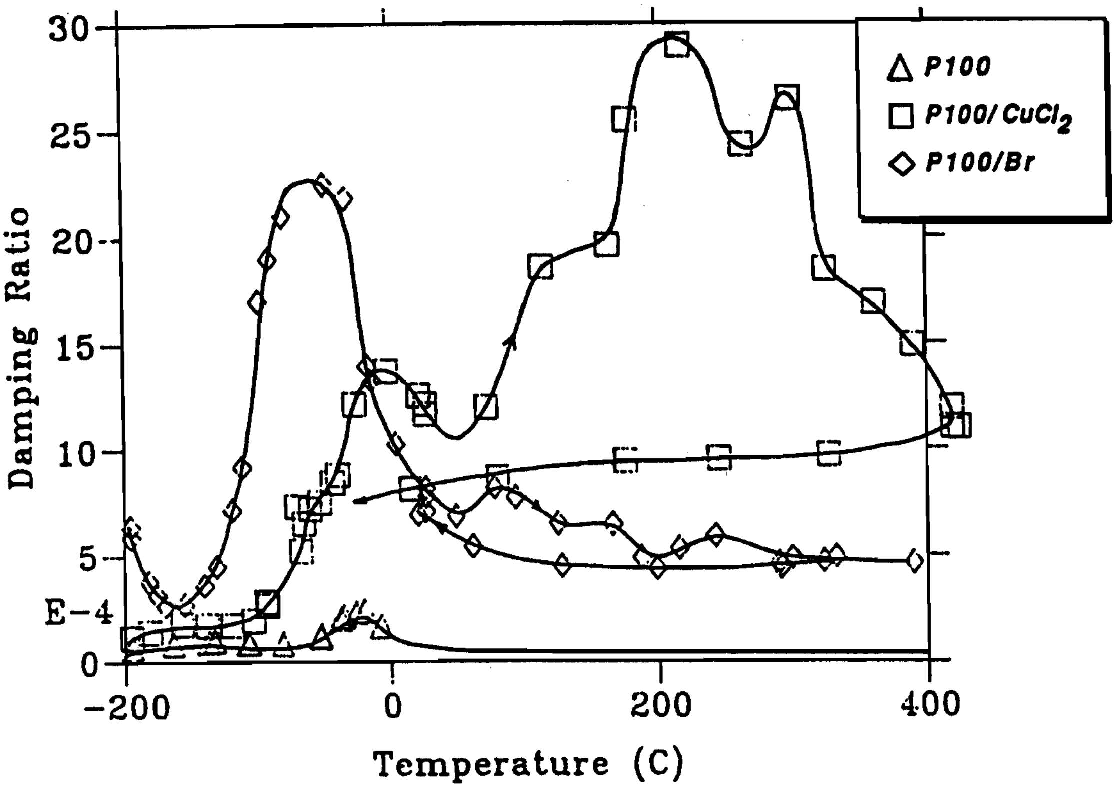

Figure 4: Damping of Intercalated Fibers and Baseline P100 Fiber versus Temperature

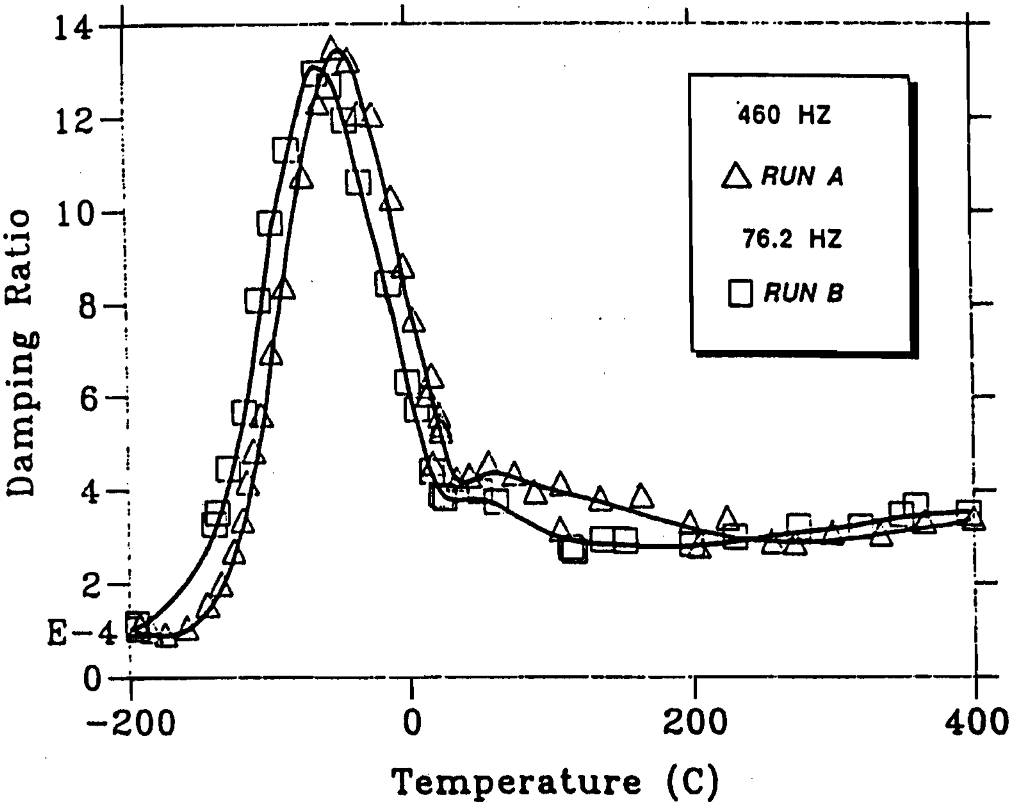

Figure 5: Damping of Bromine-Intercalated Fibers versus Temperature at Two Frequencies

In order for spacecraft designers to select and define specific materials for specific applications, they must have data on all relevant design properties. Advantages and drawbacks of various materials designs can then be weighed in making a final decision. Of interest for demonstrated increased electrical conductivity, intercalated graphite fibers have been studied by other researchers in the past. Data from those studies was collected to supplement that measured in this work. Although damping changes significantly from its baseline values in brominated fibers, other design properties such as modulus, strength, and thermal conductivity change only slightly [9], as shown in Table 1.

ACKNOWLEDGMENTS

This work reported herein was the result of an informal collaborative effort between SPARTA, Inc. and the NASA Lewis Research Center. SPARTA's efforts were supported by the Strategic Defense Initiative Organization, the Office of Naval Research, and the Naval Surface Warfare Center under a Small Business Innovation Research (SBIR) program (Contract N60921-87-C-0257). Mr. A.P. (Dave) Divecha of NSWC and Dr. Steven Fishman of ONR monitored the work.

SPARTA has a patent pending in this new technology area.

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Table 1: Property Tradeoffs for Bromine-Intercalated Graphite Fibers

PROPERTY	P100	P100/BR	COMMENTS
ζ (10^{-4})	2	20	ORDER OF MAGNITUDE INCREASE
R ($\mu\Omega$ - cm)	250	50	DECREASE BY 5
E (GPa)	690	735	SLIGHT INCREASE
σ_U(GPa)	2.2	?	LITTLE CHANGE
ρ (g/cm^3)	2.18	2.30	SLIGHT INCREASE
k_L(W/mC)	350	315	SLIGHT DECREASE
α_L(ppm/C)	-1.6	?	LITTLE CHANGE

- *FIBER DIAMETER INCREASES BY ABOUT 5%*
- *HIGHER CONCENTRATION OF BR IN FIBER CENTER*