CONTROLLING THE DAMPING BEHAVIOR OF PITCH-BASED CARBON FIBERS

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The damping capacity of intercalated graphite fibers has been found to be significantly greater than that of pristine fibers. An effort is discussed to control and optimize the damping behavior via intercalation. A resonant flexural free decay test method was used to measure the damping of single pristine and intercalated pitch-based carbon fibers (Thornel P100). The fibers were tested in high vacuum, at temperatures from 77 K to 675 K, and at frequencies from 50 to 2000 hz. The fibers were intercalated by two methods. The resulting damping capacities are compared and contrasted. The effects of changes in the intercalation processes are discussed as a means of controlling the fiber damping capacity. In addition, the retention of increased damping capacity following thermal cycling was measured and is discussed.

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BACKGROUND

$$\zeta_{composite} = \zeta_{fiber} \frac{E_{fiber}}{E_{composite}} + \zeta_{matrix} \frac{E_{matrix}}{E_{composite}}$$

- **UNIQUE FACILITY FOR MEASUREMENT OF DAMPING CHARACTERISTICS OF SINGLE FILAMENT FIBERS.**
- **COOPERATIVE RESEARCH WITH SPARTA, INC. SHOWED DAMPING CAN BE INCREASED SIGNIFICANTLY USING INTERCALATED FIBERS.**
- **TECHNOLOGICALLY SIGNIFICANT DAMPING PEAK OBSERVED IN TEMPERATURE RANGE OF INTEREST FOR VAPOR BROMINATED P100 FIBERS.**

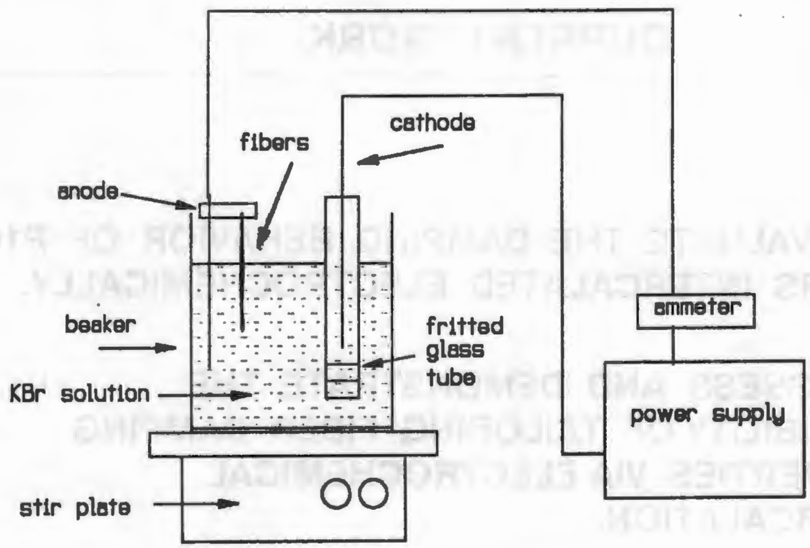
The majority of intrinsic material damping in polymer and metal matrix composites is contributed by the fibers rather than the matrix damping properties. Increased damping can reduce or eliminate vibration loads and reduce acoustic noise. Additionally, passive damping, via fibers in composites, is an important attribute for many space structures and could alleviate the need for more complex active damping mechanisms. A flexural free decay fiber test facility enables the measurement of the damping characteristics of single filament fibers [1]. This is beneficial for both providing constituent data for modelling composite behavior and allowing direct and simple measurement of changes in fiber damping behavior resulting from chemical or physical treatments.

Using the facility, Lesieutre et al [2-4] measured the damping characteristics of various graphite fibers and demonstrated that the damping capacity of pitch-based graphite fibers can be significantly increased by intercalation treatments. The authors reported a damping capacity peak on the order of 3 percent, in a narrow temperature range, for P100 fibers intercalated via a bromine vapor treatment.

CURRENT WORK

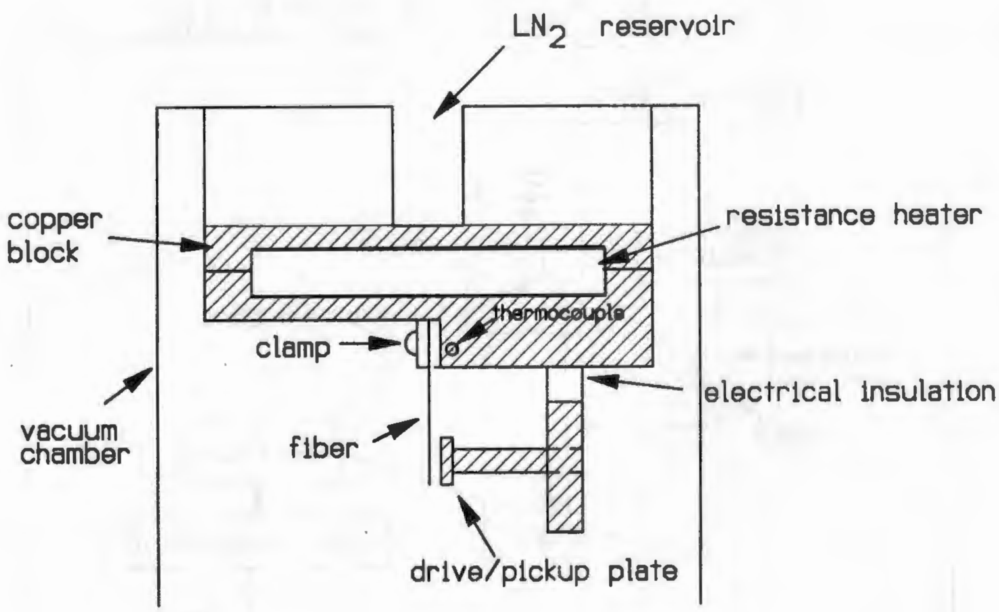
- **TO EVALUATE THE DAMPING BEHAVIOR OF P100 FIBERS INTERCALATED ELECTROCHEMICALLY.**
- **TO ASSESS AND DEMONSTRATE THE FEASIBILITY OF TAILORING FIBER DAMPING PROPERTIES VIA ELECTROCHEMICAL INTERCALATION.**

Ho and Chung [5] demonstrated that P100 fibers can be intercalated by both vapor and electrochemical methods. Since electrochemical methods allow greater control of intercalation parameters, this study is being undertaken to evaluate the damping differences between vapor brominated and electrochemically brominated P100 fibers. Comparison of damping from the two methods and the greater control of the electrochemical technique may allow determination of the mechanisms at work that result in various damping behavior. This work-in-progress paper summarizes the research to date and demonstrates the feasibility of tailoring fiber damping properties via electrochemical intercalation.



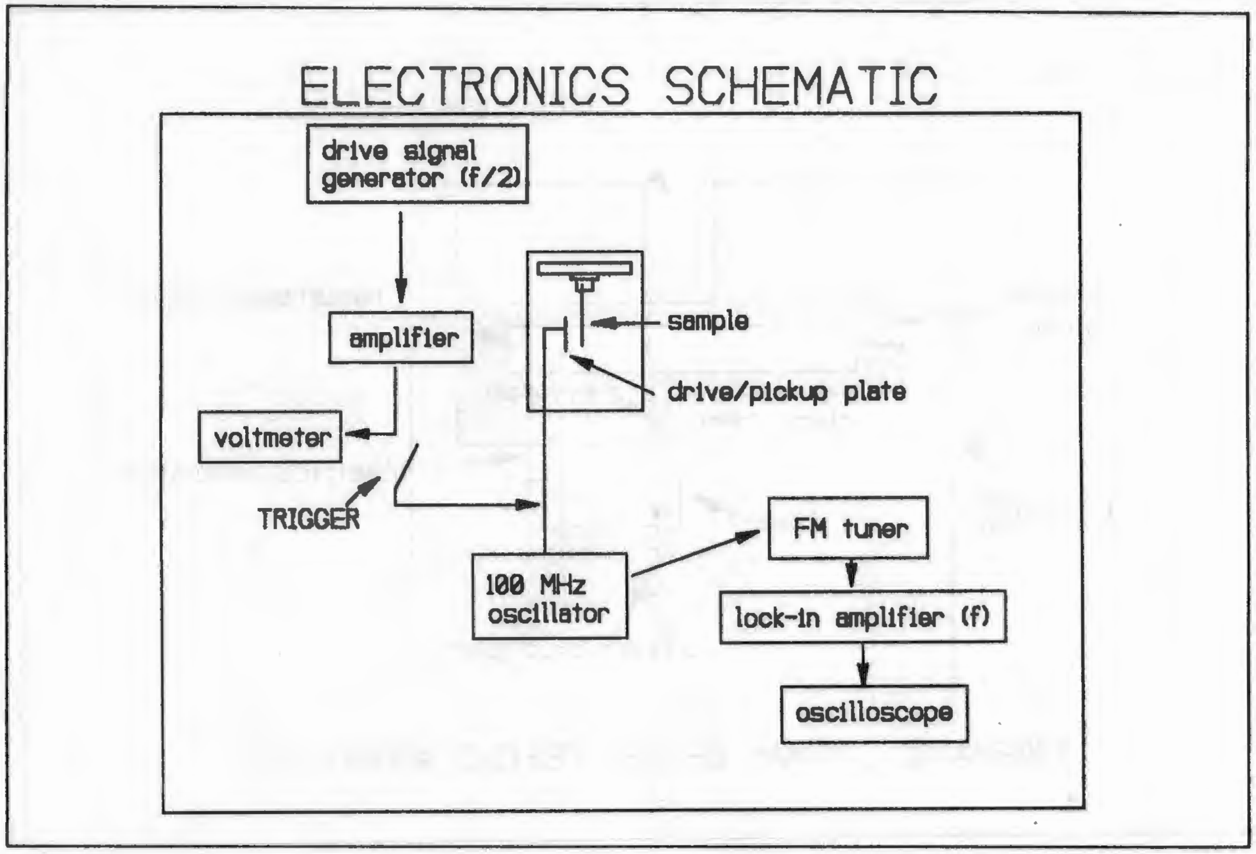
SCHEMATIC DIAGRAM OF INTERCALATION PROCESS

The intercalation process used was similar to the one used by Ho and Chung [5], with some minor modifications. Pristine P100 graphite fibers, unsized and continuous filaments, were used in this study. Fiber tows of approximately 2000, 10 μm diameter filaments were suspended in a saturated aqueous potassium bromide solution. A constant current was then passed through the tow and platinum fixture. The fixture, with tow, was removed from the solution at specific time increments (25, 49, 80, and 100 h.) After each removal, the tow was thoroughly rinsed with deionized water, and allowed to dry overnight in air at room temperature.



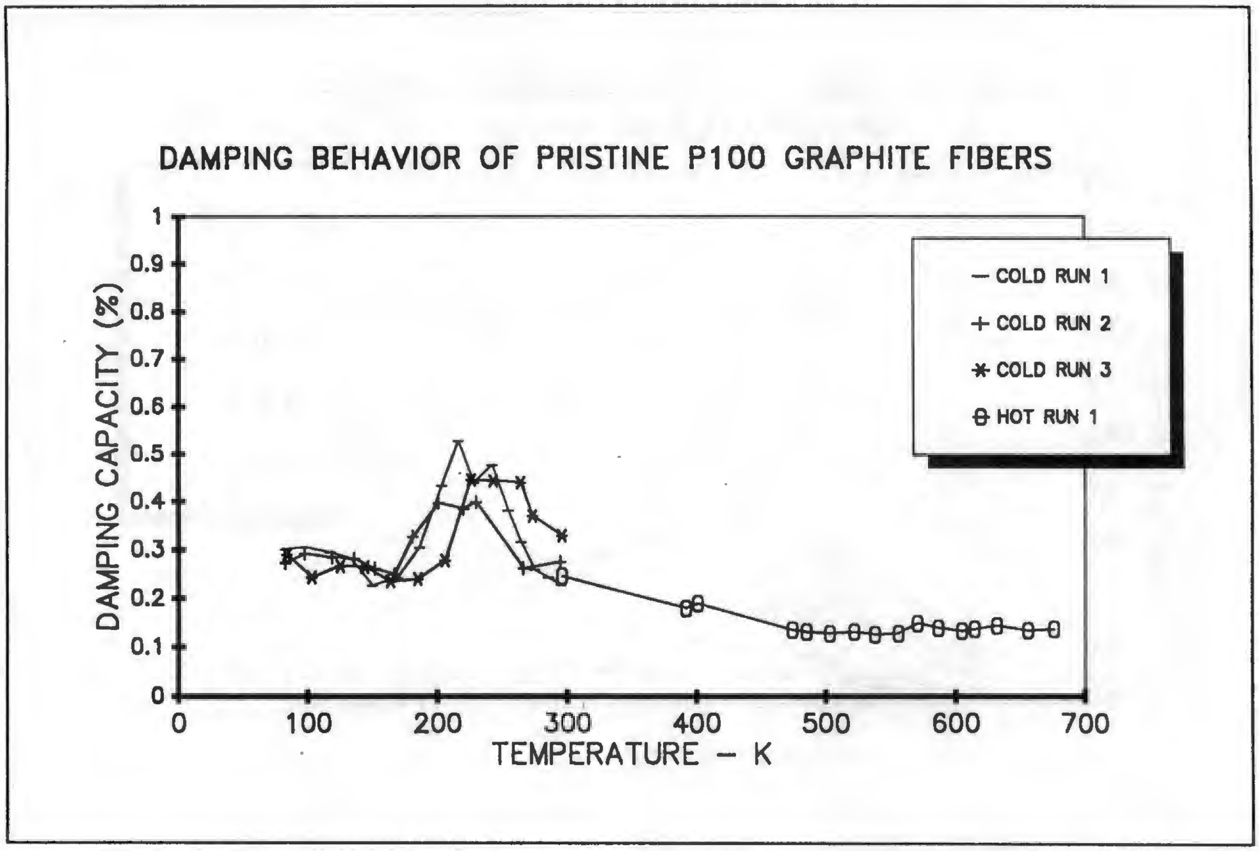
SCHEMATIC DIAGRAM OF THE TESTING APPARATUS

Individual fibers were removed from fiber tows and mounted in tantalum tabs following the procedure described by Lesieutre et al [2,4]. The mounted fiber specimen was clamped to a copper block which served as both a seismic and a thermal mass. The temperature of the block was controlled by pouring liquid nitrogen into a reservoir on the top, initially cooling to 77 K (-196 C) and then slowly heating to 673 K (400 C) by adjusting the current through an embedded resistive heater. A thermocouple attached to the block near the fiber root measured the temperature. The combination of high fiber longitudinal thermal conductivity and slow heating rates (2 K/min) ensured that the fiber temperature was effectively that measured by the thermocouple. Data points were taken usually every 15-20 K, depending on the situation. The drive/pickup plate was mechanically attached to the specimen mounting block, but was electrically insulated from it. The entire block was placed inside a vacuum chamber (10^{-4} to 10^{-6} torr) to eliminate the effects of air damping, which was significant on these fibers.



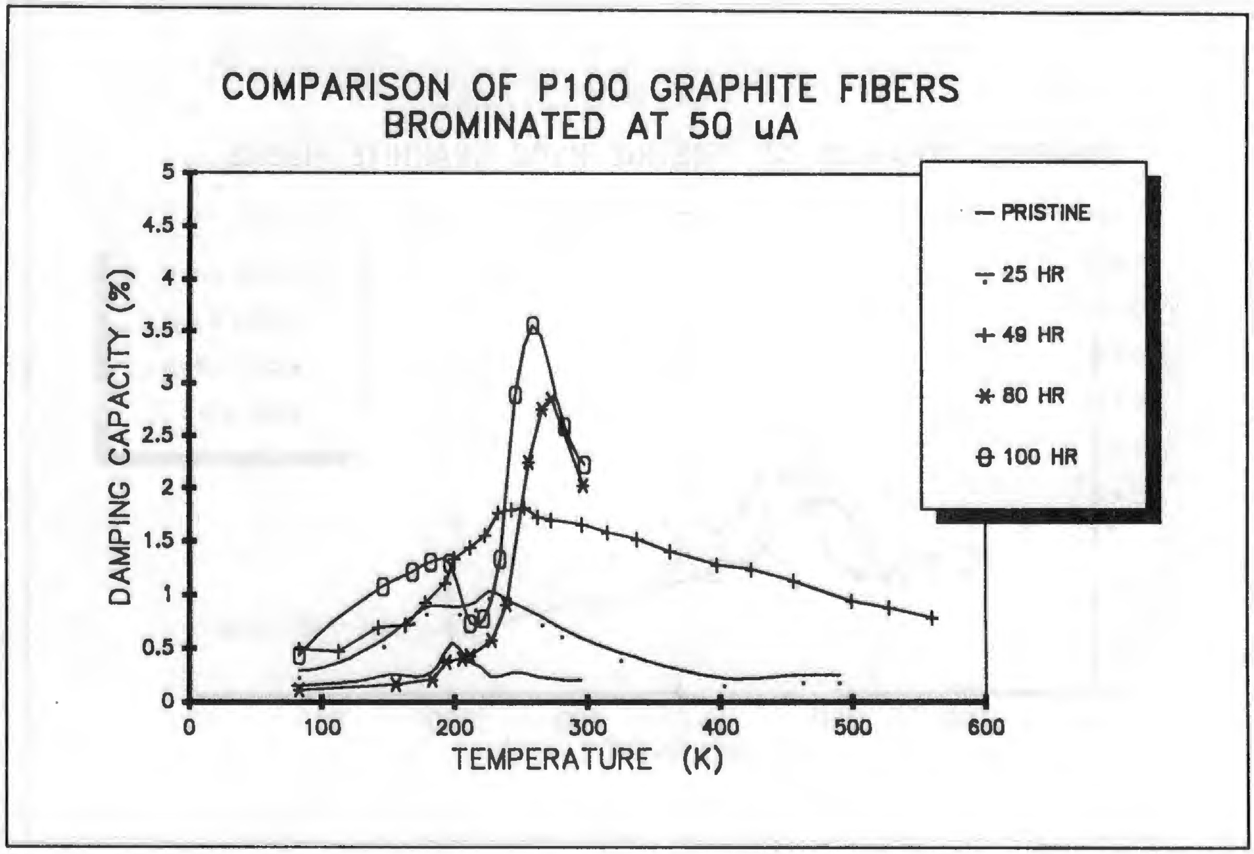
The fiber was driven electrostatically at one of its resonant flexural frequencies, f_n, by applying an alternating voltage at $f_n/2$ between the fiber and an adjacent drive plate [1]. The fiber-plate separation was adjusted by a screw type manipulator attached to the drive plate. Specimen vibration amplitude was controlled by the output voltage from the drive amplifier. 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.

Specimen motion was detected by placing the fiber-plate capacitor into the tank circuit of a 100 MHz RF oscillator. Fiber vibrations produced an oscillating capacitance which directly modulated the RF oscillator via a half-wavelength coaxial cable. A commercial FM tuner detected these modulations and converted them back to an audio signal with frequency f_n and amplitude directly proportional to that of specimen vibration. Damping was determined by disconnecting the drive signal (triggering the signal) and allowing the fiber resonant vibrations to decay freely. The decaying signal was displayed on an oscilloscope and recorded photographically. Damping values are calculated from free decay data and are reported as damping capacity ($\psi = \Delta w/w$ = relative vibration energy lost per cycle).

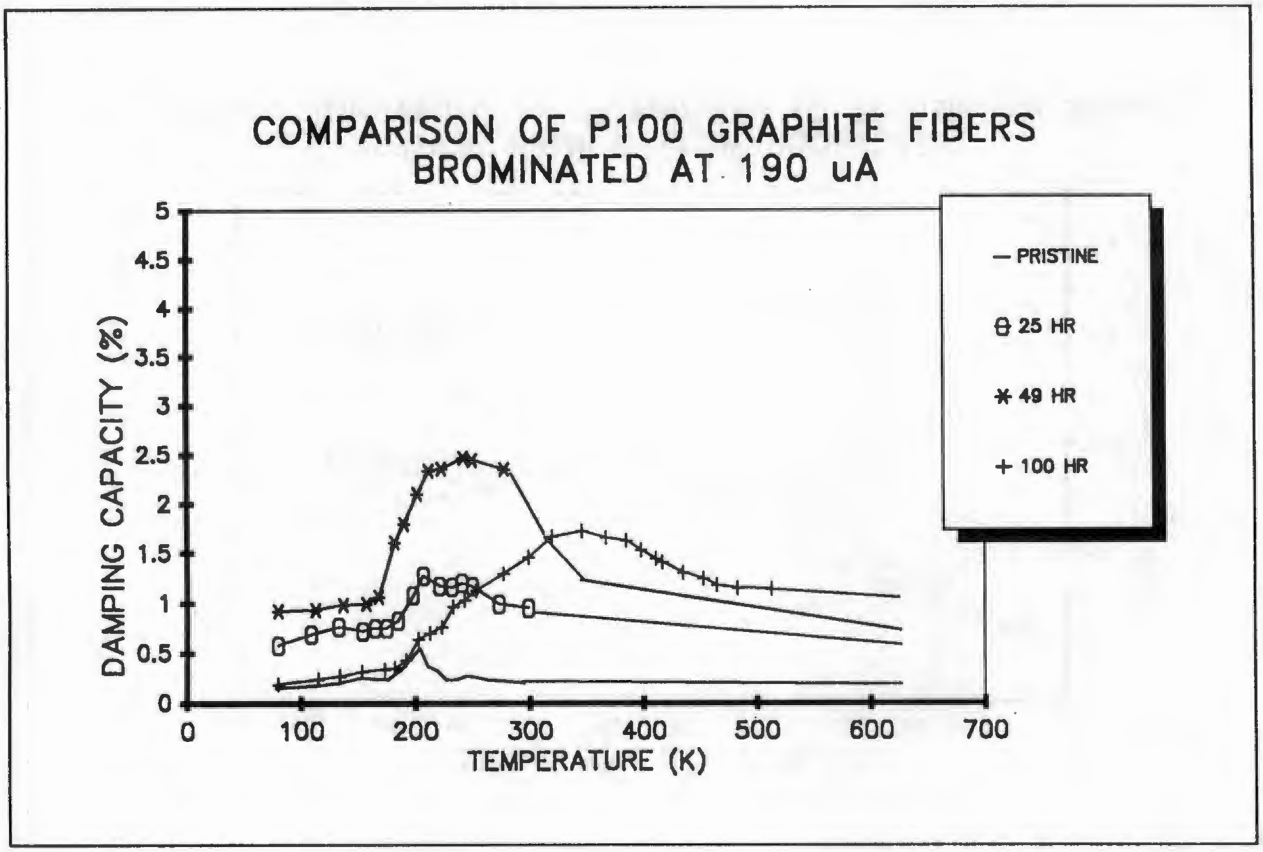


Various treatments were performed on pristine P100 graphite fibers to determine their effects on damping behavior. These included vacuum cycling, thermal cycling, and exposure to air and nitrogen gas. These were performed since they are typical of treatments the fibers may see during composite processing.

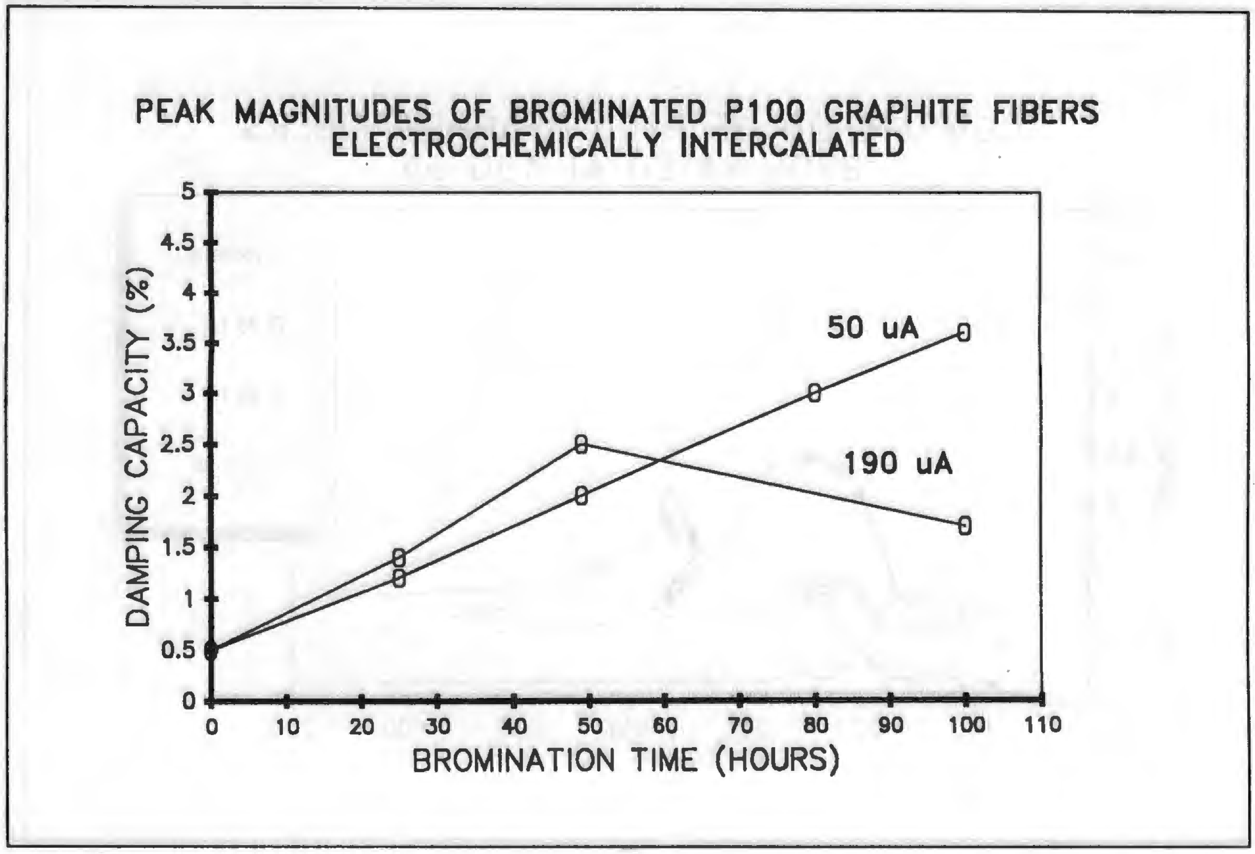
Upon heating from liquid nitrogen to room temperature (cold run 1) the pristine P100 fiber exhibited a damping peak at 213 K (-60 C) which is small in magnitude, with ψ about 0.5%. The thermal cycling included runs to elevated temperatures (673 K [400 C], hot run 1) and back to cold temperatures (77 K [-196 C], cold run 2). During this thermal cycling, the damping peak location remained the same at 213 K (-60 C), and the peak height remained virtually unchanged at 0.5%. Also, the third cold temperature run (cold run 3) was conducted after the test chamber was brought up to atmospheric pressure in air for 24 h and then re-evacuated. There was no change with any of these treatments to the damping of the pristine fibers. The next treatment was exposure to gaseous nitrogen (not shown). After evacuation, the vacuum chamber was back filled with gaseous nitrogen for 18 hours. Damping data was then taken on the fiber. The damping peak again occurred at a temperature in the vicinity of 213 K (-60 C) and had a peak height of 0.45%. This value is almost identical to the pristine fibers. Also, the baseline remained around 0.18%, similar to the pristine fiber. It can then be concluded that these basic treatments to the pristine fibers do not introduce significant changes in damping behavior.



To determine the effects of electrochemical bromination parameters, a current of 50 microamperes (μA) was selected and the fibers were brominated for 25, 49, 80, and 100 h. The damping peaks increased in magnitude, as well as shifting to higher temperatures, as bromination times increased. The 100 hour test showed the highest peak magnitude, ψ_{max} = 3.6%, at 253 K (-20 C). The peaks shift to higher temperatures as a result of increased concentrations of bromine added to the fiber. In addition, all of the 50 μA samples showed a second, smaller peak between 133 and 183 K (-140 C and -90 C.) This peak is quite small in magnitude, except for the 100 hour test, in which the peak has a magnitude of approximately 1.4% and occurs at 183 K (-90 C.)



The next step was to determine if increased currents and corresponding increases in bromine concentration would further improve the damping behavior of the fibers (actual determination of bromine mass and mass distribution in single fibers is difficult and has not yet been done.) The current was increased to 190 μA and fibers were brominated through a similar cycle as the 50 μA samples. The damping continually increased until the 49 h point where it had a peak of 2.5%. Temperatures at which damping peaks occurred increased as the bromination time increased. At 100 h, the peak magnitude decreased to 1.7% and the temperature of the peak was 60 C. No explanation for this behavior is provided, although work is still proceeding. The baseline itself also had a value 5 times that exhibited by pristine graphite fibers, measuring approximately 1.0%.



The peak magnitudes of damping for the electrochemically intercalated fibers generally increased with time of bromination. Work is continuing to evaluate a broader range of currents and investigate the decrease in damping for the 100 hour bromination treatment at 190 μA.

CONCLUSIONS

- **P100 GRAPHITE FIBERS INTERCALATED ELECTRO-CHEMICALLY DEMONSTRATE SIGNIFICANT INCREASES IN DAMPING VALUES OVER PRISTINE FIBERS.**
- **THE DAMPING CHARACTERISTICS OF P100 GRAPHITE FIBER CAN BE TAILORED BY ELECTROCHEMICAL INTERCALATION.**
- **THE HIGHEST DAMPING OBSERVED TO DATE (~ 3.5%) IS OBTAINED AT LOWER INTERCALATION CURRENTS AND LONGER TIMES.**
- **ADDITIONAL STUDY IS NEEDED TO RESOLVE MECHANISMS FOR DAMPING BY BROMINE INTERCALATION.**

[1] J.A. DiCarlo and J.E. Maisel, "Measurement of the time Dependent Dynamic Mechanical Properties of Boron/Aluminum Composites." p 201-27 in Fifth Conference on Composite Materials: Testing and Design. ASTM-STP-674, Amer. Soc. for Test Materials, Philadelphia, Pa., 1979

[2] G.A. Lesieutre, A.J. Eckel, and J.A. DiCarlo "Temperature Dependent Damping of Some Commercial Graphite Fibers" Metal Matrix, Carbon and Ceramic Matrix Composites, NASA CP-3018, 125-133, 1989.

[3] G.A. Lesieutre, A.J. Eckel, and J.A. DiCarlo "Increased Graphite Fiber Damping via Intercalation" pp. GDA1-GDA9 in Proceedings of Damping '89, WRDC-TR-89-3116, Vo. II, 1989.

[4] G.A. Lesieutre, A.J. Eckel, and J.A. DiCarlo "Damping of Bromine-Intercalated P100 Graphite Fibers" Accepted for publication in Carbon February, 1991.

[5] C.T. Ho and D.D.L. Chung "Carbon Fibers Brominated by Electrochemical Intercalation" Carbon, Vol 28,4 521-528 (1990).