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SUMMARY

Composites using vapor-grown carbon fibers (VGCF), the most conductive of the carbon fiber types, are attractive for applications where low density, high strength, and at least moderate conductivity are required, such as electromagnetic interference shielding covers for spacecraft. The conductivity can be enhanced another order of magnitude by intercalation of the VGCF. If a high Z intercalate is used, the protection of components from ionizing radiation can be enhanced also. Thus, the intercalation of VGCF with IBr is reported. Since composite testing is required to verify properties, the intercalation reaction optimization, stability of the intercalation compound, scale-up of the intercalation reaction, composite fabrication, and resistivity of the resulting composites is also reported. The optimum conditions for low resistivity and uniformity for the scaled up reaction (20-30 g of product) were 114 °C for at least 72 hr, yielding a fiber with a resistivity of 8.7 ± 2 μΩ-cm. The thermal stability of these fibers was poor, with degradation occurring at temperatures as low as 40 °C in air, though they were insensitive to water vapor. Composite resistivity was 200 ± 30 μΩ-cm, as measured by contactless conductivity measurements, about a factor of five higher than would be expected from a simple rule of mixtures. The addition of 1.0 percent Br_2 intercalated microfibers increased the resistivity of the composites by more than 20 percent.

INTRODUCTION

There has been increasing concern with lowering the mass of spacecraft as a means to save launch costs. Much of this has involved replacing aluminum parts with graphite fiber composites. This has proved to be difficult, however, when the electrical conductivity of the aluminum is central to its use, as is the case with electromagnetic interference (EMI) shielding covers.

The most effective way to increase the conductivity of a composite is to increase the conductivity of the fibers themselves (ref. 1), and the most conductive of the graphite fibers are those grown from gaseous carbon feedstocks, commonly referred to as vapor-grown carbon fibers (VGCF). The resistivity of VGCF is typically 70-90 μΩ-cm, lower by a factor of 30 than conventional PAN fibers (ref. 2). The resistivity of VGCF can be lowered by another order of magnitude by the process of intercalation, the insertion of guest atoms or molecules, such as Br_2, between the graphene layers (ref. 3).

Besides blocking EMI, these shielding covers must also protect sensitive electronics from high energy photons and particles. This type of shielding can be enhanced by the use of intercalates containing atoms of high atomic number. Tests have shown that IBr intercalated pitch-based fiber composites make

better shields of high energy photons than do those using Br_2 as the intercalate (ref. 4). In this study we have combined the use of the highly conductive VGCF with the resistivity and shielding enhancement of IBr to make high performance composites coupons for evaluation.

METHODS AND MATERIALS

Three types of VGCF were kindly provided by Applied Sciences, Inc. (Cedarville, OH). The first type, Pyrograf I short staple fibers, had diameters ranging from 10-30 μm, and lengths of a few cm. These fibers were used for all of the preliminary studies. The second type, Pyrograf I mat fibers were similar in structure to the Pyrograf I short staple, except that they were much longer (~1 m) and somewhat interwoven into a mat about 5 cm wide and 1 m long. The third type, Pyrograf III are smaller, having diameters near 0.1 μm, and lengths near 1 mm. All three of these fiber types were grown using a proprietary process which utilized natural gas as a feed material. The fibers were heat-treated to 3000 °C before intercalation to improve their graphitization.

Pyrograf III fibers were intercalated with Ibr using the conditions determined for the Pyrograf I fibers, and with Br_2 using a room temperature vapor phase intercalation. Because of their small size, the two-point resistivity of compacts of the Pyrograf III fibers were measured by placing the fibers in an insulated chamber with a conductive floor and piston. The resistance is dependent on the applied pressure and the contact resistance, so the absolute numbers are difficult to interpret, but relative measurements can be made. Additional details of the method have been described elsewhere (ref. 5). Previous studies suggest that the resistivity of the pristine fibers should be about 100 μΩ-cm.

IBr intercalation has not been widely studied, and all of the studies to date have been carried out on mg quantities of pitch-based fibers (ref. 6). Since gram quantities of IBr intercalated VGCF would be required to fabricate the composites, it seemed prudent to first optimize the intercalation reaction conditions. A small quantity (20 - 70 mg) of Pyrograf I short staple fibers was placed in an 8 mL (1 dram) glass vial which was in turn placed in a weighing bottle with a volume of about 100 mL and ground glass lid. About 1-2 grams of solid IBr (well in molar excess over carbon in VGCF) was placed in a second vial and also placed in the weighing bottle. The lid was sealed with Halocarbon grease (VWR Scientific). The weighing bottle was sealed in a paint can and placed in an oven preset to the appropriate temperature. The paint can was a precaution against IBr leaking from the weighing bottle.

The initial conditions chosen were 60 °C for 24 hours. At this temperature the vapor pressure of IBr was expected to be about 100 torr from consideration of the vapor pressure curves of Br_2 and ICl (ref. 7). From experience with Br_2 and ICl 24 hours at 100 torr was expected to yield complete intercalation in VGCF.

IBr readily dissociates into Br_2 and I_2 at the reaction temperatures used for these experiments (K_{eq} = 0.22 at 298 K, and 9.9 at 387 K) so it is important to verify whether the intercalating specie is actually IBr, Br_2 or I_2. It has been found that Br_2 does not intercalate graphite at temperatures of 60 °C or higher (ref. 8), and I_2 does not intercalate graphite under any known conditions. The intercalation experiment was attempted for both Br_2 and for I_2 under the same conditions as IBr, and the result was no mass uptake, and no decrease in the resistivity. Thus, it was concluded the IBr is indeed the intercalating specie.

Mass uptake, resistivity, and temperature coefficient of the resistivity (TCR), and x-ray diffraction analysis were used to judge the success in intercalation. The resistance was determined by mounting the fibers using silver paint (Structure Probe Inc., West Chester, PA) on specially designed four-point mounting chips. Current was supplied to the outer leads using a Keithley Model 225 constant current

source operated at 1.000 mA, and voltage was measured using a Keithley Model 181 nanovoltmeter. Fiber length was determined using a reticule in a 10× dissecting microscope, and diameter from photographs taken at 2000× and compared to a 10 μm calibration target. These chips were mounted to an Air Products Displex liquid helium cold finger for the TCR measurements with a capability to cool to 10 K. X-ray diffraction patterns were taken using a Huber precession camera mounted on a Rigaku Rotaflex rotating anode generator using filtered Mo K_α radiation.

After intercalation under those conditions was confirmed, reaction time was varied from 0.1 hr to three days. Next, the reaction temperature was varied from 50 - 114 °C for the optimized time. Finally, reaction time was again varied for the optimized temperature. In each case, mass uptake, four-point resistivity, and temperature coefficient of the resistivity, and x-ray diffraction analysis were used to judge the success in intercalation.

Studies on pitch-based fibers indicated that IBr intercalation compounds were environmentally stable (ref. 6), but since stability has been shown to be a function of graphitization of the carbon (ref. 9), IBr in VGCF stability, as judged by fiber resistivity, was evaluated under ambient, high temperature, and high humidity conditions.

Initial intercalation reactions were carried out using about 20 mg of VGCF, but for even modest composite samples (7.6 cm × 7.6 cm) the reaction must be scaled up by three orders of magnitude. This process involved additional time-temperature-time studies using the small-scale optimized condition as a starting point. All three types of VGCF were used at this point.

Composite fabrication was carried out by hand laying up four-ply laminates within a stainless steel mold. Two types of Pyrograf I were used; the mat filaments were much longer than the mold and had an alignment of better than 5°, and the short staple filaments with an average length of 2-4 cm, which were often gently curled, had an alignment better than about 15°. First a Teflon (PTFE) face sheet was placed in the bottom of the mold so that the finished composite could be removed easily. Then, 0.25 g of fibers was trimmed and placed in the bottom of a 3 in. by 3 in. mold so that the fibers were parallel to the edge of the mold. A second layer of 0.25 g of fibers was laid into the mold perpendicular to the first. This was repeated for two more layers.

Two types of resins were used, RS-3 (YLA, Inc., Benicia, CA) and room-temperature cure epoxy. A mass of 0.8 grams of RS-3 resin was dissolved in about 40 mL of propanone (acetone). This was poured over the fibers as evenly as possible. A second teflon face sheet was placed over the fibers, the mold plunger was placed in the mold on top of the fibers, and the assembly was placed in a Wabash four-posted hydraulic press which had been previously heated to 120 °C (250 °F). The top platen was brought down until it just made thermal contact with the mold, but with a minimum of pressure. It sat in this configuration until most of the propanone evaporated (1.0 hr). At that point 9 MPa (1300 psi) of pressure was applied and the temperature was raised to (350 °F). It took about 20 minutes for the press to come to the new temperature, where it was held for an additional 2.0 hr.

The procedure to fabricate composites using the room temperature cure epoxy was slightly different. An excess of epoxy resin-hardener mixture was mixed and 0.80 g of it was evenly applied to the top teflon face sheet. This was placed in the mold, followed by the plunger. The assembly was placed in a Carver press and held under pressure for more than 24 hr. A variation of the room temperature procedure involved adding 1.3 g of Br_2 intercalated Pyrograf III fibers by stirring them into 98 g of resin and adding 3.7 g of hardener before it was applied to the face sheet. This resulted in 1 percent Pyrograf III fibers to provide additional current paths between the Pyrograf I fibers, and so to increase the composite conductivity.

The conductivity of the composites was determined using a Leheighton 1010A contactless conductivity probe which has been factory modified to operate at 55.55 kHz. Each composite was marked off in 6.4 mm (0.25 in) grid lines. The thickness was measured at each of the grid points using a digital micrometer. The thickness measurements are then dialed into the 1010A and the conductivity is read directly. Contour plots of both thickness and conductivity were then analyzed.

RESULTS AND DISCUSSION

Optimization

The first stage of the optimization of the IBr reaction was carried out at 60 °C, the temperature utilized by the pitch-based fiber work (ref. 6). Determining the mass uptake for the fibers was not as simple as might be supposed because as soon as the fibers are removed from the IBr vapor, they begin to lose mass. In Br_2 intercalated fibers this has been shown to be due to out-gassing of Br_2 from the surface of the fiber. This continues until a stable "residue compound" is formed, which has a stage 2 intercalated (two carbon layers for each Br_2 layer) core and a concentration gradient to a pure carbon surface (ref. 10). It is thought that the IBr intercalation mechanism is probably analogous. Figure 1 shows this behavior for a typical sample of about 40 mg of fiber. The stoichiometry changes from less than $C_{15}IBr$ to nearly $C_{50}IBr$ (after ultimate stabilization).

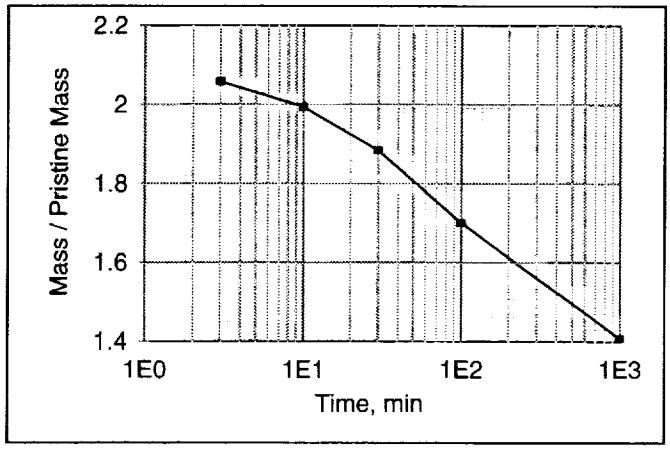

Figure 1.—Mass relative to pristine mass of a 40 mg VGCF sample as a function of time removed from Ibr illustrating typical deintercalation behavior.

The initial mass uptake is the most useful mass measurement in determining the extent of reaction, but it is also the most difficult to accurately measure. The reaction vial must be removed from an oven and rapidly cooled so that buoyant forces do not distort the mass measurement. But the IBr, which is very corrosive, must be allowed to escape while the vial is warm or it will solidify, exaggerating the mass uptake. It can be inferred from the semilog plot in Figure 1 that the degassing exhibits first order kinetics. Thus a linear regression of the semilog data was performed without the 1.0 minute data, and extrapolated to 1.0 minute.

Using this mass value, the empirical formula of the resulting intercalation compound can be calculated as a function of intercalation time. A plot of the ratio of C to IBr is shown in Figure 2 as a function of intercalation time. It can be seen that, according to this analysis, the intercalation reaction under these conditions requires about 24 hr, and there is no significant improvement or degradation by extending that time to as long as 120 hours.

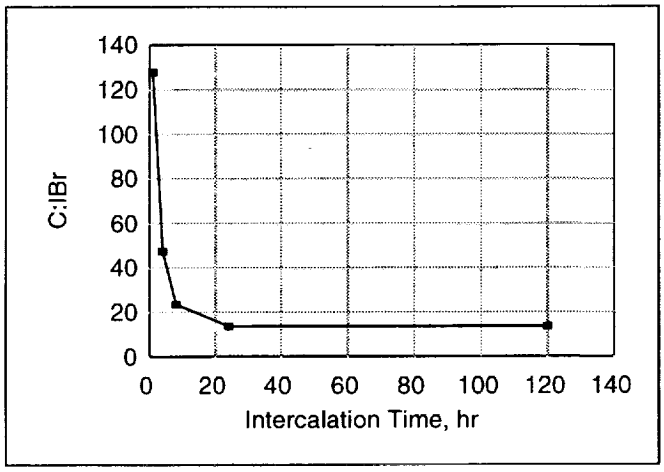

Figure 2.—Ratio of C to IBr atoms as a function of time fibers exposed to IBr vapor at 60 °C.

The overall stoichiometry of the compound 1.0 min after removal from the reactor is about $C_{14}IBr$, perhaps a nominal stage 2 intercalation compound. But in pitch based fibers Br_2 has been shown to be effectively absent from the surface, while making up nearly 40 percent of the mass in the fiber center (ref. 10). Thus, the distribution of IBr is probably not uniform within the fiber, so characterization of the stage has little meaning.

Attempts to determine the stage of the residue compound by X-ray diffraction were also inconclusive. The 2θ values along the 00ℓ axis appeared to be shifted slightly giving a stage 3 intercalation compound with a c axis of about 13.5 Å. But without additional diffraction peaks the stage identification must be regarded as tentative.

For a single fiber, however, it is much easier to accurately measure the resistivity than the mass, which for a 1 cm long fiber is typically about 1 μg. Resistivity has been shown to be a very sensitive indicator of the amount of intercalate within the fiber (ref. 11). Resistivity is also the property most important to the application of the fiber. So for each reaction, the resistivity of a sample of 6 - 18 individual fibers was measured. This also enabled the determination of the uniformity of reaction throughout the sample. Figure 3 is a plot similar to Figure 2, but using resistivity instead of C to IBr ratio.

It can be seen that the trend in Figure 3 is the same as that in Figure 2. Both lead to the conclusion that at 60 °C an intercalation time near 24 hr is optimal. Although the mean resistivity at 120 hr was slightly higher than that at 24 hr, the difference was not statistically significant.

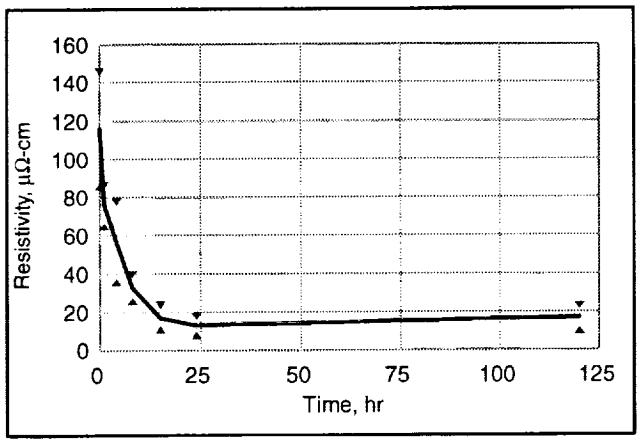

Figure 3.—Average resistivity of fibers as a function of time exposed to IBr vapor at 60 °C.

Intercalation with strong oxidizers such as IBr results in the addition of hole carriers in the graphite lattice. The mobility of these carriers is very high, so their addition results in a significant decrease in the resistivity. As a result the fiber resistivity changes from semiconductor-like behavior (negative TCR) to metal-like behavior (positive TCR). Figure 4 shows the resistivity/300 K resistivity as a function of temperature for fiber samples from various times in 60 °C IBr vapor. Note that only the pristine fiber exhibits semiconductor-like behavior. Note also that the slope tends to become more positive as the time increases. Although the trend is not as clear as the average room temperature resistivity of the fibers, it is clear that the fibers are intercalated.

A similar strategy was used to optimize the temperature, though only the resistivity was monitored over the rest of the tests. As can be seen in Figure 5, VGCF resistivity was measured as reaction time was held at 24 hr, and the temperature was increased to 114 °C. The average resistivity dropped slightly as the temperature increased, but more importantly, the standard deviation in the resistivity values decreased. That is, the more graphitic of the fibers could be intercalated at any of the temperatures, but the less graphitic required higher temperatures. Thereafter, reaction time was varied at the 114 °C

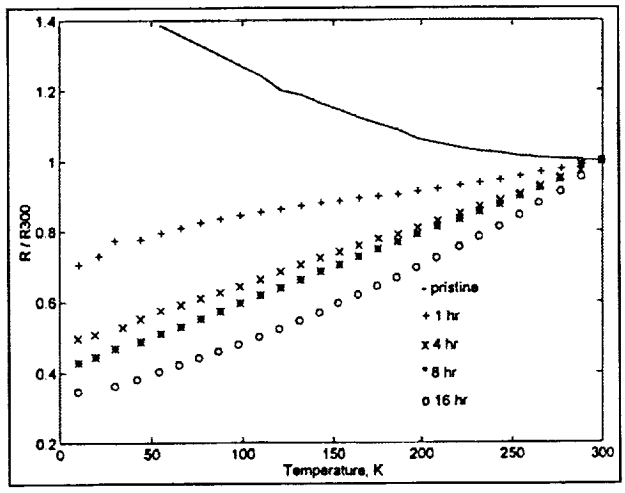

Figure 4.—The temperature coefficient of resistivity for pristine VGCF and those intercalated by IBr for various times at 60 °C.

conditions, and though the fibers intercalated more quickly, the standard deviation decreased at longer times. The optimal conditions of 114 °C for 24 hr, resulted in VGCF resistivity of 8.7 ± 2 μΩ-cm, comparable to nickel.

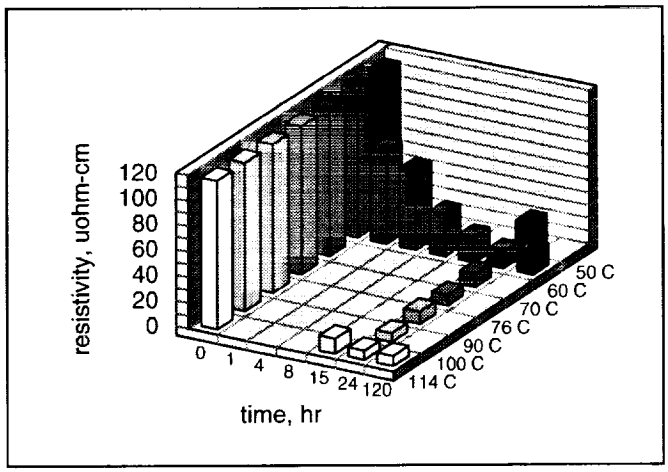

Figure 5.—Average fiber resistivity as a function of reaction temperature and time exposed to IBr vapor.

Stability

The thermal stability of optimally intercalated VGCF proved to be much lower than anticipated. Figure 5 shows the average resistances as a function of time for temperatures up to 300 °C in air divided by their initial resistances. They indicate that the resistivity degrades at temperatures as low as 40 °C. This was much less stable than anticipated from previous results of the stability of IBr intercalated pitch fibers (ref. 6), and the stability of Br_2 intercalated VGCF (ref. 12). The threshold stability is so low that it could hinder their use in polymer matrix composites, which typically have resin cure temperatures near 175 °C.

Similar tests were run to judge the susceptibility of the intercalated fibers to high humidity. This test has typically been carried out at 100 percent humidity at 60 °C. Since these fibers degrade at 60 °C, the degradation rates of the high humidity samples were compared with the heated air samples. Those results indicate no significant susceptibility to humidity (Figure 7).

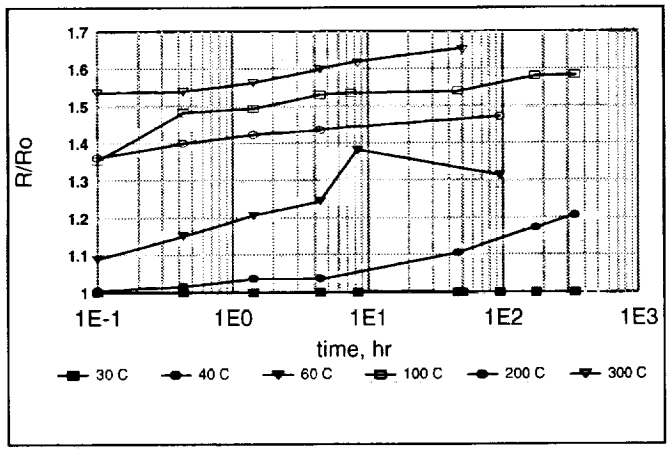

Figure 6.—The average resistivity of at least 5 IBr intercalated VGCF's as a function of time in air heated to the indicated temperatures divided by their initial resistivities.

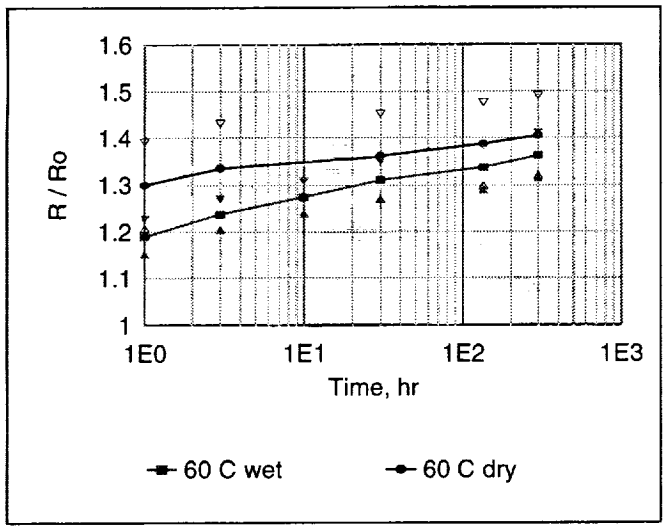

Figure 7.—Comparison of the change in resistance ratio with time for VGCF+IBr at 60 °C in air and high humidity environments.

Scale-Up

A single 7.62×7.62×0.10 cm composite which is 60 percent fiber by volume requires 3.48 cm^3 or, assuming a density of 2.1 g/cm^3, about 7.3 grams of fibers. Since our initial optimization experiments utilized only 20 - 70 mg of fibers, they needed to be scaled up by three to four orders of magnitude. Our initial reaction chamber has a volume of about 100 mL, and the scale-up of the reactor utilized a 5 cm (2 in) diameter, 53 cm (21 in) long tube furnace with a volume near 1.0 L. Thus, though our mass was to increase four orders of magnitude, our reactor volume only increased one order of magnitude. Since our supply of VGCF was severely limited, initial studies were carried out in the 1.0 L reactor with 4 - 6 g of IBr to provide sufficient vapor pressure, but with only about 20 mg of fibers. Particular attention must be paid to the termination of the intercalation reaction. If the IBr vapor pressure drops before the tube cools, then the fibers will deintercalate because their thermal stability is poor. Thus, the tube must be thoroughly cooled before any IBr is released. One end of the reactor tube was cooled with ice so that the IBr would not condense out on the fibers as the temperature dropped.

Table I shows the results of the scale-up trials. It was noted that 114 °C reaction temperature was near optimal, but the time must be increased three-fold. Up to 30 grams of fiber was packed into the part of the tube furnace that was within the center and right heating zones, and 30 grams of IBr in the left zone. The zones were heated up to 114 °C for the intercalation, and at the end of 72 hours, the IBr end was cooled first as the furnace returned to room temperature.

Table I.—Pyrograf I Scale-Up Parameters

Fiber Type	Mass gram	Time hr	$\rho_{IBr\ end}$ μΩ-cm	ρ_{middle} μΩ-cm	$\rho_{far\ end}$ μΩ-cm
short staple	30	64		9.3	35.
short staple	30	70.5		10.2	51.
mat	20	74.5	6.0	19.1	13.0
mat	20	71.5	8.4	10.0	31.
mat*	20	73	44.	81.	89.

*seal leaked

It was found that the fibers located near the IBr source intercalated more fully than those far from it, probably due to mass transfer problems. This could be corrected by incorporating more than one IBr source within the tube.

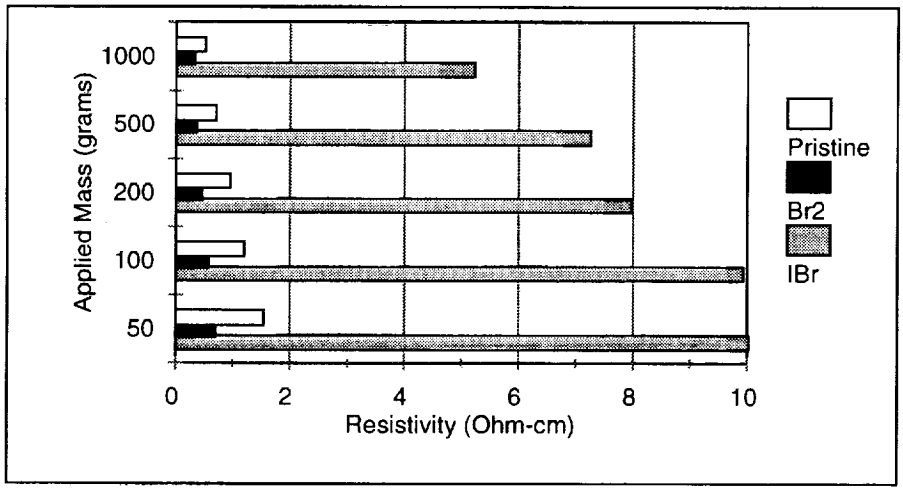

Figure 8.—Resistivity of compacts of Pyrograf III fibers.

Pyrograf III fibers were reacted with IBr at 114 °C for 24 hr in an attempt to intercalate them also. Resistivity measurements on compacts of these fibers indicated that the resistivity of the fibers increased upon reaction with IBr (Figure 10). In addition, the mechanical properties were noticeably different, with the IBr reacted fibers being much more brittle than the pristine. When the Pyrograf III fibers were reacted with Br_2 vapor at room temperature, the resistivity dropped by about a factor of two, and had mechanical properties similar to pristine. It was concluded that IBr did not intercalate Pyrograf III under the same conditions as Pyrograf I, but no further studies were undertaken to find those conditions. Since Br_2 lowered the resistivity and increased the Z of the fibers, the Br_2 intercalated fibers were used in the composite studies.

Composite Resistivity

The expected four-point composite resistivities ($\rho_{composite}$) can be calculated from fiber resistivities (ρ_{fiber}). The fibers comprised about 60 percent of the fiber volume (f_{fiber}), and the resin is effectively non-conducting. Due to the 0-90° geometry of the composite, only half of the fibers will be running parallel to the current. Thus:

$$\rho_{composite} = \frac{\rho_{fiber}}{0.5\ f_{fiber}} = \frac{8.7\ \mu\Omega\text{-}cm}{0.5\ (0.60)} = 29\ \mu\Omega\text{-}cm$$

The eddy-current composite resistivity for the composite types is shown in Table II. For composites made with IBr intercalated VGCF the resistivity was 200 ± 25 μΩ-cm. This corresponds to a four-point resistivity of about 140 μΩ-cm (ref. 13). This is nearly a factor of five higher than expected. Although we have no explanation for this, the factor of five has been observed before (ref. 14). The addition of the Br_2 intercalated Pyrograf III fibers did not improve the resistivity, but in fact appears to have increased it. We speculate that the Pyrograf III may be hindering the fiber to fiber contacts of the Pyrograf I fibers.

Table II.—Composite Properties

Composite Type	Fiber Vol, %	Thickness,mm	Resistivity, μΩ-cm	$\rho_{pristine}$, ρ
Pristine	>50	1.58±.02	552±20	1.0
IBr + epoxy	59.7	1.07±.03	218±21	2.5
IBr + Pyro III + epoxy	50.8	0.40±.01	270±35	2.0
IBr + RS-3	>60	0.69±.04	174±29	3.2

CONCLUSIONS

The intercalation reaction of IBr into VGCF was investigated as a means to create a low density, high strength, and at least moderately conductive composite material for applications such as electromagnetic interference shielding covers for spacecraft. A high Z intercalate was used as the protection of components from ionizing radiation can thus be enhanced also. This involved performing a time and temperature study, using resistivity as a criterion. In addition, the reaction was scaled up three orders of magnitude from an initial yield of 20 - 70 mg to 20 - 40 grams of intercalated fibers in order to produce enough material to make test coupons. The optimum conditions for the scaled up reaction were 114 °C for 72 hr, yielding a fiber with a resistivity of 8.7 ± 2 μΩ-cm. The thermal stability of these fibers was poor, with degradation occurring at temperatures as low as 40 °C in air. Composites were fabricated using both polycyanate and room temperature cure epoxy resins. Composite resistivity was 200 ± 30 μΩ-cm, as measured by contactless conductivity measurements, about a factor of five higher than would be expected from a simple rule of mixtures. The addition of 1.0 percent Br_2 intercalated microfibers increased the resistivity of the composites by more than 20 percent.

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13. ABSTRACT (*Maximum 200 words*)

Composites using vapor-grown carbon fibers (VGCF), the most conductive of the carbon fiber types, are attractive for applications where low density, high strength, and at least moderate conductivity are required, such as electromagnetic interference shielding covers for spacecraft. The conductivity can be enhanced another order of magnitude by intercalation of the VGCF. If a high Z intercalate is used, the protection of components from ionizing radiation can be enhanced also. Thus, the intercalation of VGCF with IBr is reported. Since composite testing is required to verify properties, the intercalation reaction optimization, stability of the intercalation compound, scale-up of the intercalation reaction, composite fabrication, and resistivity of the resulting composites is also reported. The optimum conditions for low resistivity and uniformity for the scaled up reaction (20–30 g of product) were 114 °C for at least 72 hr, yielding a fiber with a resistivity of 8.7±2 μΩ-cm. The thermal stability of these fibers was poor, with degradation occurring at temperatures as low as 40 °C in air, though they were insensitive to water vapor. Composite resistivity was 200±30 μΩ-cm, as measured by contactless conductivity measurements, about a factor of five higher than would be expected from a simple rule of mixtures. The addition of 1.0 percent Br_2 intercalated microfibers increased the resistivity of the composites by more than 20 percent.

14. SUBJECT TERMS

Intercalated; Composites; Carbon fiber; Resistivity; Iodine bromine; IBr

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