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ABSTRACT

Laminar composites have been fabricated from pristine and bromine intercalated Amoco P-55, P-75, and P-100 graphite fibers and Hysol-Grafil EAG101-1 film epoxy. The thickness and r.f. eddy current resistivity of several samples were measured at grid points and averaged point by point to obtain final values. Although the values obtained this way have high precision (< 3 percent deviation), the resistivity values appear to be 20 to 90 percent higher than resistivities measured on high aspect ratio samples using multi-point techniques, and by those predicted by theory. The temperature dependence of the resistivity indicates that the fibers are neither damaged nor deintercalated by the composite fabrication process. The resistivity of the composites is a function of sample thickness (i.e. resin content). Composite resistivity is dominated by fiber resistivity, so lowering the resistivity of the fibers, either through increased graphitization or intercalation, results in a lower composite resistivity. A modification of the simple rule of mixtures model appears to predict the conductivity of high aspect ratio samples measured along a fiber direction, but a directional dependence appears which is not predicted by the theory. The "resistivity" of these materials is clearly more complex than that of homogeneous materials.

INTRODUCTION

There has been a great deal of experimental work in the last several years to characterize the electrical properties of graphite intercalation compounds (GIC's). The motivation for much of this work has been the eventual use of these compounds as electrical conductors. This goal appears to be within reach, as resistivities similar to that of silver have been obtained with AsF_5 intercalated highly graphitized fibers[1]. At this stage in the technological development of these materials there is a growing realization that improving on the fundamental properties of GIC's is a necessary but insufficient condition for their application[2].

Most schemes for utilizing intercalated graphite fibers envision embedding them either in a metal or polymer matrix. There have been several studies in which the electrical properties of either chopped or short lengths of pristine carbon fibers have been mixed with a polymeric resin[3,4,5]. There have, however, been problems reported when intercalated graphite fibers are chopped[6] or milled[7] which appear to be due to intercalate diffusing out of the fibers[8]. But a more serious problem is that the resistivity of this type of composite is fundamentally limited by percolation of current through the matrix, and so even the lowest resistivities reported for this type of composite are on the order of Ω-cm. For many power applications, such as cabling and antennae, metal-like resistivities ($\mu\Omega$-cm) will be required.

Perhaps a more effective approach to obtaining low resistivity graphite fiber composites is to utilize laminar composites, similar to those which have been so effective in providing light-weight, high-strength, high-modulus materials to the aerospace industry. Pitch-based graphite fibers have much lower resistivities than the PAN-based carbon fibers which are routinely used for strength. Only a few studies have been published where the electrical properties of pristine pitch-based graphite fibers in epoxy resin composites were studied[9,10,11]. In these cases the composites studied were small unidirectional composites prepared by hand. In addition, some authors have used intercalated graphite fibers. Once again the data base is small, and only small, hand laid up, unidirectional composites were fabricated and tested[12,13].

In this study we have extended this approach to produce electrically conducting graphite fiber - polymeric resin composites by using woven graphite fiber fabrics and standard fabrication techniques. We have used three grades of pitch based graphite fibers, Amoco P-55, P-75, and P-100, and their bromine intercalation compounds. In addition we have addressed the problem of characterizing the resistivity of the composites, which is complicated by their anisotropy.

METHODS AND MATERIALS

Six types of composites, which differed in their fiber composition, were fabricated for this study. Thornel P-55, P-75, and P-100 pitch-based graphite fibers, manufactured by Amoco, were chosen because they form stable, well characterized intercalation compounds with bromine[14], and are commercially available in spools of indefinite length. The fibers

range in resistivity from 950 $\mu\Omega$-cm, for pristine P-55, to 50 $\mu\Omega$-cm, for bromine intercalated P-100.

The fibers were woven into a cloth prior to intercalation. Six-thousand filament strands of P-55 and P-75 fibers were woven into an 11 by 11 yarns/inch (4.3 by 4.3 yarns/cm) five-harness satin weave by Mutual Industries (Philadelphia, PA). This resulted in an areal weight of about 300 g/m^2. Because the P-100 fibers are more brittle than the P-55 and P-75, they were handled differently. Two-thousand filament strands were woven 10 by 10 yarns/inch (4 by 4 yarns/cm) in a plain weave pattern by Fabric Development, Inc. (Quakertown, PA). This resulted in a looser fabric with an areal weight of about 270 g/m^2, but very little filament breakage.

Four woven cloth samples of each fiber type were cut into 38 x 38 inch (96 x 96 cm) pieces and rolled to fit inside 4 inch (10 cm) glass tubes. The ends of the tubes were sealed with teflon covered rubber stoppers. Enough bromine was added to soak the entire cloth with a small amount of excess (about 500 ml). The tubes were rolled periodically so that the entire surface of each sample was submerged several times under the bromine. Capillary action caused the bromine to soak into the cloth. Thus the conditions were intermediate between a vapor phase and a liquid phase intercalation. Hung and Long have shown that cooling to near 0 ° C is necessary for bromine intercalation of P-55 fibers[15], so in that case the tube was submerged in an ice bath.

After four to seven days, the bromine was cleared out of the tube using a stream of air. This process required another two to four days, depending upon the amount of excess liquid bromine remaining after the intercalation reaction. The fabric was very stiff and brittle at the end of this process because of residues left by the liquid bromine. To remove the fabrics from the tubes, they were rinsed with bromoform until the residual solvent was nearly colorless. Residual bromine intercalation compounds have been shown to be stable in bromoform for at least several days[7] thus indicating there was no fear of deintercalation by this process. The bromoform lubricated the graphite filaments, leaving the cloth very pliable. The cloth was then removed from the reaction tube and laid out on a flat surface to dry. Unfortunately, much of the cloth's stiffness returned after the cloth dried. Sample filaments were taken from several locations on each cloth sample, and their resistivity was measured to ensure that the reaction was successful.

At room temperature under bromine atmosphere, there is a bromine mass uptake of about of about 44 percent of the carbon mass[16], or about 130 grams of bromine per cloth. The final fiber has about 18 percent bromine by mass, or about 55 grams per cloth. Although the vast majority of the bromine will degas from the fibers within a day[17], there is noticeable degassing for a week after they are removed from the tubes.

The cloths were packaged flat and shipped to Rohr Industries (Chula Vista, CA) where the composite samples were fabricated using Hysol-Grafil EAG101-1 film epoxy. Alternate layers of film and cloth were built up to four plies in the same orientation (0°_4) with a target composition of 68 percent fiber fill. The resulting laminates were cured at 350 ° F (175 ° C) for 1.5 to 2 hours. Previous studies have shown that the bromine intercalated fibers are indefinitely stable in air at these temperatures.

The resin density was 1.265 g/cm^3. The fiber densities were measured using a density gradient technique[18]. The laminate physical properties are summarized in Table I. It is interesting to note that the void content is substantially lower in the bromine intercalated samples. This may be due to improved wetting of the fiber by the resin. The laminates were cut into squares 2.25 inches (5.7 cm) on a side. About twenty of these test coupons were made and characterized for each of the fiber types (120 in all).

For the purpose of mapping the thickness and resistivity of the coupons, grids were superimposed on them dividing them into eight mm squares. Thickness measurements were made at each of the 49 grid crossings. Resistivity measurements were disregarded for the edge grid lines, so there were 25 resistivity measurements for each coupon.

Thickness measurements were made to nearest 0.005 mm with a digital micrometer. These were used to assess the laminate quality, and to set the thickness indicator on the contactless conductivity probe.

Resistivity measurements were made using a modified LEI 1010A conductivity probe (Leheighton Electronics, Inc., Leheighton, PA) in the bulk conductivity mode. The 1010A was modified by Leheighton Electronics to operate at 55.55 kHz (as opposed to the standard frequency of 1000 kHz) in order to increase the penetration depth of the probe. Verification of the accuracy of the instrument was provided by standards provided by both Leheighton Electronics and by the National Institute of Standards and Technology.

In addition, a round robin of samples, including five samples of each fiber type which was used in this study, was held with Intercal Co. (Port Huron, MI) and Rohr Industries.

They each have R.F. Conductivity devices which are comparable, except that the Intercal instrument uses 1 kHz, and the Rohr instrument uses 3000 kHz. Although there were some variations among the three instruments, the values were similar.

Contour plots of the thickness and resistivity of each of the samples were generated using PC-MATLAB (The Math Works, Inc., Sherborn, MA). A linear extrapolation between measured points was used which results in artificially angular results (as in Figures 1 and 2). The thickness plots were contoured at 50 μm intervals. The resistivity plots were contoured at a level near 2σ, where σ is the standard deviation of the resistivity values in a single laminate. The values of the grid line intersections were averaged point by point to arrive at the final values. The average values were also contoured to aid the search for systematic measurement errors.

The four-point resistivities of the composites were measured with the current traveling along the plies (0°) and along the ply diagonal (45°). Making the correct electrical contact to these laminates is not straightforward. If contact is made to the resin there is a very large contact resistance and the voltage distribution of a thin layer of insulator is being measured. If the resin is sanded off, then the fibers which are being sampled are most likely damaged, and not representative of the sample. To try to overcome these difficulties, silver current contacts were painted across the cut ends of the composite as well as on the surfaces, and voltage contacts were painted across not only the top and bottom surfaces but also across the cut edges. With the cross-ply configuration, half of the fibers should carry the current into the middle of the sample.

The results from the four-point measurements of the first set of samples has been briefly described elsewhere[19], but in order to confirm those results, multi-point measurements were made. Samples with dimensions of 0.2 by 14 cm were cut, and current leads were wrapped around the ends and attached with silver paint which also covered the ends of the sample. Voltage contacts were made with a multiple point spring contact fixture. Resistivity was determined by fitting a straight line to the graphs of potential as a function of position. Additional samples of different widths were later measured to determine the effect of composite width of the measured resistivity.

The temperature dependence of the four-point resistivity was measured over the range of 1.6 to 300 K using a 19 Hz alternating current ranging from 5 μA for single fibers, up to as high as 2 mA for the larger composite samples with a lock-in amplifier (PAR model

HR-8) driving a digital voltmeter. Samples were surrounded by He gas to minimize the temperature rises from Joule heating. Temperatures were measured with a copper-constantan thermocouple above 77 K, and a carbon-glass resistance thermometer below that temperature.

RESULTS AND DISCUSSION

Analysis of the sample thickness confirmed the general high quality of the laminates. The standard deviation of the laminate thicknesses among samples ranged from 0.6 to 2.1 percent. There were, however, thickness variations within single samples which were as large as 15 percent from the mean. The variations in sample thickness are summarized in Table II. In order to try to identify systematic errors in the thickness measurements the average laminate thickness was also calculated by averaging each grid point through all samples of a given type. Contour plots of the average laminate thickness were then drawn. Contours shown in Figure 1 were drawn at the 2σ level. Although there appears to be a tendency for the measurements in the middle of the laminate to be somewhat thicker, variations are less than 2σ from the mean in all cases except P-75 + Br. Here the variations are just over 2σ.

The r.f. eddy current resistivities of the samples are summarized in Table III. The overall trend is for the composite resistivity to be dominated by the fiber type. Thus, the most conductive fibers (bromine intercalated P-100) result in the most conductive composites. The average composite r.f. eddy current resistivity for each fiber type was also calculated point by point in a way analogous to that described above for the average composite thickness. The results are shown in Figure 2. Note here that there is a definite trend of higher resistivity at the composite edges. This may be an indication that the edge effects are somewhat larger than anticipated by our measurement technique. The standard deviations are quite small however, ranging from about 1.5 to 3.3 percent.

An effort was made to address how accurately the r.f. eddy current resistivity measurements reflect the "true" resistivity of the sample. Although the r.f. eddy current technique has been shown to be insensitive to inhomogeneities parallel to the magnetic field (from ply to ply)[20], it does carry the implicit assumption that the sample is homogeneous normal to the field (within the ply). This assumption is certainly not valid for conductive fibers in an insulating matrix, and is probably not even valid within the fibers themselves.

Three approaches were taken in an attempt to verify the r.f. eddy current results: comparison with known conductivity standards, comparison with other r.f. eddy current instruments, and comparison with four-point resistivity measurements.

Standard reference material 1523, "Silicon Resistivity Standard for Eddy Current Testers", is a set of two homogeneously doped silicon standards, one with a resistivity of 1 Ω-cm, and the other with 0.01 Ω-cm. In addition, another doped silicon standard prepared and calibrated by the NSTM (but not a standard reference material) which had a resistivity of 0.0011 Ω-cm was measured. Our instrument measured all of the reference materials accurately. This established that our instrument was working properly, but not that the values in a heterogeneous material were correct.

A round-robin test was set up with Intercal Company (Port Huron, MI), where they have built an eddy current conductivity instrument which operates at 1.00 kHz, and with Rohr Industries, where they have built one which operates at 3000 kHz. As the frequency of the instrument increases, the penetration depth probed decreases. The penetration depth is also less for more conductive samples. Thus, the lower the frequency, the higher the conductivity that can be measured for a given sample depth. In all there were about 50 samples included in the round robin, most of which were graphite epoxy composite samples with various PAN, pitch, pristine, and intercalated fibers. The conductivity measurements tended to fall in frequency order with the Intercal instrument measuring slightly lower resistivities, and the Rohr instrument measuring higher. The actual values varied by 20 - 30 percent. Figure 3 shows a plot of the penetration depth as a function of resistivity for our instrument. Given the nominal sample thickness of about 1 mm, it can be seen that a composite resistivity greater than 20 μΩ-cm should be measurable. This is more than a factor of three more conductive than the most conductive fibers by themselves. The resistivity range of the composite samples measured is also indicated in the figure, and they are well within the measurable range for our instrument.

Having established that the r.f. eddy current instrument from Leheighton measured homogeneous materials correctly, and that the frequency is sufficiently low to determine reasonable values for the conductivity of heterogeneous materials, the eddy-current resistivity values were compared to those obtained using a four-point measurement technique, and those predicted by theory.

One would expect the composite resistivity might follow from a simple rule of mixtures calculation:

(1) $$\sigma_c = \sigma_f v_f + \sigma_m v_m$$

where σ_c, σ_f, and σ_m are the composite, fiber, and matrix conductivities respectively, and v_f and the v_m are the respective fiber and matrix volume fractions. For these composites $\sigma_f \gg \sigma_m$, and $v_f \approx v_m$ so the second term is negligible and to a good approximation:

(2) $$\sigma_c = \sigma_f v_f.$$

There is, however, a directionality to the conductivity owing to the systematic way in which the fibers are embedded in the matrix. For a uniaxial composite this directionality has been shown to be of the form:

(3) $$\sigma_\phi = \sigma_l \cos^2\phi + \sigma_t \sin^2\phi$$

where σ_l is the conductivity along the longitudinal (fiber axis) direction of the composite, σ_t is the conductivity along the transverse direction of the composite, and ϕ is the angle between the measurement and the longitudinal direction[9]. In a perpendicular biaxial composite with no interaction between the laminates the relation:

(4) $$\sigma_\phi = \sigma_l \cos^2\phi + \sigma_t \sin^2\phi + \sigma_{l'} \cos^2(\phi - \pi/2) + \sigma_{t'} \sin^2(\phi - \pi/2)$$

where $\sigma_{l'}$ and $\sigma_{t'}$ are the longitudinal and transverse components of the conductivity of the perpendicular fibers, would hold. For the composites measured in this study, $\sigma_l = \sigma_{l'} \gg \sigma_t = \sigma_{t'}$, thus the directionality disappears, and equation (4) reduces to:

(5) $$\sigma_\phi = \sigma_l = \sigma_c$$

Note, however, that σ_l is the conductivity of the fibers that are parallel to the measurement direction or, in the case of a 0 - 90 cloth measured in the longitudinal direction, one half the volume fraction. Thus, in terms of fiber conductivity:

$$\sigma_c = \sigma_f v_f/2 \tag{6}$$

In the real case of a woven fabric there certainly is interaction between the laminates, but these would also be expected to have no angular dependence.

The average four-point resistivities for each of the composite types are listed in Table III. They are in reasonably good agreement with the r.f. eddy current results, but clearly higher than the values calculated from the rule of mixtures argument presented above. Further, when composite samples with the cloth angled 45° to the current flow were measured, the resistivity was substantially higher. With the exception of internal and external contact resistance subtleties, this should be a straightforward measurement, and so the fact that the results are so far from the rule of mixtures model is troubling.

Perhaps the fibers were damaged during the laminate fabrication process. If there was substantial damage done to the fibers, not only would the fiber resistivity increase, but its temperature dependence would change also. As Figure 4a illustrates, the temperature dependence of the resistivity, however, was found to be very similar to that of the pristine fibers. In addition, the temperature dependence of bromine intercalated fibers (which is substantially different from that of the pristine fibers) and the composites made from the intercalated fiber are also similar (Figure 4b), indicating that the intercalated fibers were not significantly either deintercalated or damaged.

In an attempt to resolve whether the additional resistance observed was real, multi-point measurements were made on high aspect ratio pristine P-100 and Br_2 intercalated P-100 fibers composites. The resulting resistivities along the fiber directions agree quite well with rule of mixtures model predictions as shown in Table III. However, by this technique the composites still had a definite directionality to their conductivity as can be seen by comparing the samples cut along the fiber axis (0°) with those cut at a 45° angle. The 45° cut composite resistivities actually agree fairly well with those from the four-point tests. It was also found that as the width of the 45° samples is increased, the effects of the directionality diminished. The directionality appears to be related to the fiber to fiber

contact resistance. Especially in the case of high aspect ratio samples, current cannot be transported very far along the voltage gradient before it must be transferred to another fiber. This is in contrast to the current traveling along a fiber aligned with the voltage gradient, which need not be transferred to another fiber. Because of the large fiber to fiber contact resistance, equation (4) above does not hold. This may indeed call into question whether equation (3) truly holds for a uniaxial composite.

The r.f. eddy current resistivity is not directional in character, and is insensitive to probe contact resistance, but probably is sensitive to fiber to fiber contact resistance. This has been confirmed by the fact that composites made with woven fabric have lower resistivities than uniaxial composites of similar fiber fill. This leads us to believe that the eddy currents are set up on a scale large enough that they must pass through both fiber and resin.

As the eddy current circulates through the inhomogeneous medium of the composite it will be dominated by the component which parallels the conducting fibers. Thus we might expect that the r.f. eddy current conductivity might be predicted by using the average conductivity in any given direction. Only that component of the fibers which is parallel to the direction of interest will contribute to the conductivity. The result of this type of analysis are the same as was demonstrated above, with the final result being equation (6).

The measured resistivity, however is about a factor of two yet larger than this. Perhaps as the eddy currents are forced through the fiber-resin interfaces there are additional components which complicate the measurement. It is interesting, and perhaps fortuitous that the r.f. eddy current resistivities and the four-point (0°) resistivities are roughly the same. Clearly, more study of these systems is required.

The r.f. eddy current resistivity is found to be a linear function of laminate thickness. Specific features in the thickness contour plots often have corresponding resistivity features. This can be attributed to local variations in the fiber fill. If the fiber cloth is of uniform thickness, then any thickness variations in the composite will be due to an excess or dearth of resin. An excess of resin will increase percolation lengths and raise the resistivity, and a dearth of resin will have the opposite effect. This effect can be clearly seen in Figure 5.

It is not clear that the multi-point resistivity measured along a fiber axis is the characteristic resistivity that can be utilized in application. The r.f. eddy-current resistivity may be more useful in applications such as electro-magnetic interference shielding, and

antenna fabrication. Perhaps the anisotropy of the composites may be useful in some applications. Clearly, the "resistivity" of a composite sample is more subtle than that of a homogeneous material, and its anisotropy and dependence upon measurement technique must be understood before there will be large scale acceptance for electrical applications.

CONCLUSIONS

Bromine intercalation of pitch-based graphite fiber cloths woven by standard methods has been scaled up to 38 inch (96 cm) widths. Laminar composites have been fabricated from both commercially available pristine and bromine intercalated graphite fibers using standard techniques. Temperature dependence of the composite resistivities indicate that neither the pristine nor the intercalated fibers are damaged during the composite fabrication process.

A procedure is proposed for measuring the resistivity of composite samples using the r.f. eddy current method. The thickness and resistivity of several samples is measured at grid points which are then averaged point by point to obtain the final value. Resistivity values obtained this way had a high precision, but the resistivity values appear to be high by 20 to 90 percent over resistivities measures on high aspect ratio samples using multi-point techniques, and by that predicted by theory.

The resistivity of the composites was found to be a function of sample thickness (i.e. resin content). Composite resistivity is dominated by fiber resistivity, so lowering the resistivity of the fibers, either through increased graphitization or intercalation, results in lowering the composite resistivity. The resistivity of composites of the most highly graphitized and bromine intercalated graphite fibers is significantly lower than that of standard graphite epoxy composite materials.

A modification on the simple rule of mixtures model appears to predict the conductivity of high aspect ratio samples aligned along a fiber direction. If the lay-up of fibers is symmetric, as in the 0-90° composites in this study, one-half of fiber volume percent must be used to account for the one-dimensionality of the fiber conductivity. Unlike that might be expected from earlier models based on uniaxial composites, the directionality of the composite conductivity appears to be dependent upon the exact geometry. At least for the case of high aspect ratio composites with the plies aligned at 45°, there is a strong

dependence of the conductivity on the ply direction. The "resistivity" of these materials is clearly more complex than that of homogeneous materials.

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Table I -- Laminate Physical Properties

	P-55		P-75		P-100	
	pris	Br_2	pris	Br_2	pris	Br_2
Fiber Volume Percent	56.2	54.0	58.7	58.5	64.0	48.0
Resin Weight Percent	28.0	31.0	27.6	30.6	24.3	38.7
Void Volume Percent	6.5	3.8	5.0	0.0	2.9	0.0
Fiber Density, g/cm^3	2.182	2.214	2.059	2.141	2.039	2.293
Resin Density, g/cm^3	1.265	1.265	1.265	1.265	1.265	1.265

Table II -- Laminate Thicknesses

P-55		P-75		P-100	
pris	Br_2	pris	Br_2	pris	Br_2
Average Laminate Thickness, mm					
.963	.992	1.00	1.03	.748	.895
Standard Deviation, mm					
.022	.006	.014	.022	.015	.008

Table III -- Laminate Resistivities

P-55		P-75		P-100	
pris	Br_2	pris	Br_2	pris	Br_2
Fiber Resistivty, μΩ-cm					
950	370	760	280	320	67
Calculated Resistivity, μΩ-cm					
3380	1370	2590	960	1030	280
R.F. Eddy Current Resistivity, μΩ-cm					
4320	2020	3920	1190	2010	490
Four-Point Resistivity (0°), μΩ-cm					
4370	2040	4010	1300	1530	650
Multi-point Resistivity (0°), μΩ-cm					
				1150	250
Four Point Resistivity (45°), μΩ-cm					
4880	2420	5620	1710	3620	1660
Multi-point Resistivity (45°), μΩ-cm					
				1400	360

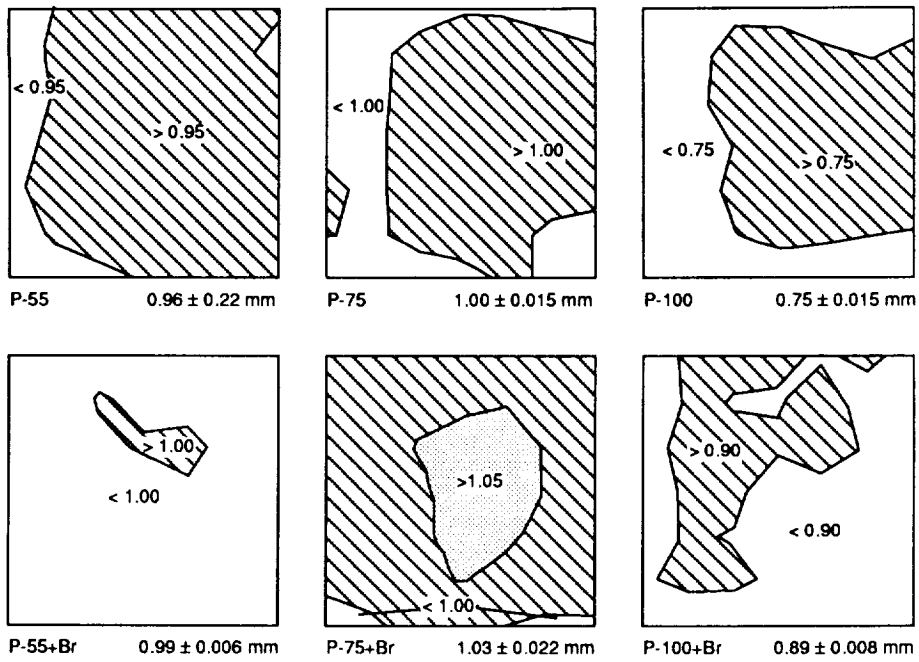


Figure 1. - Average laminate thickness.

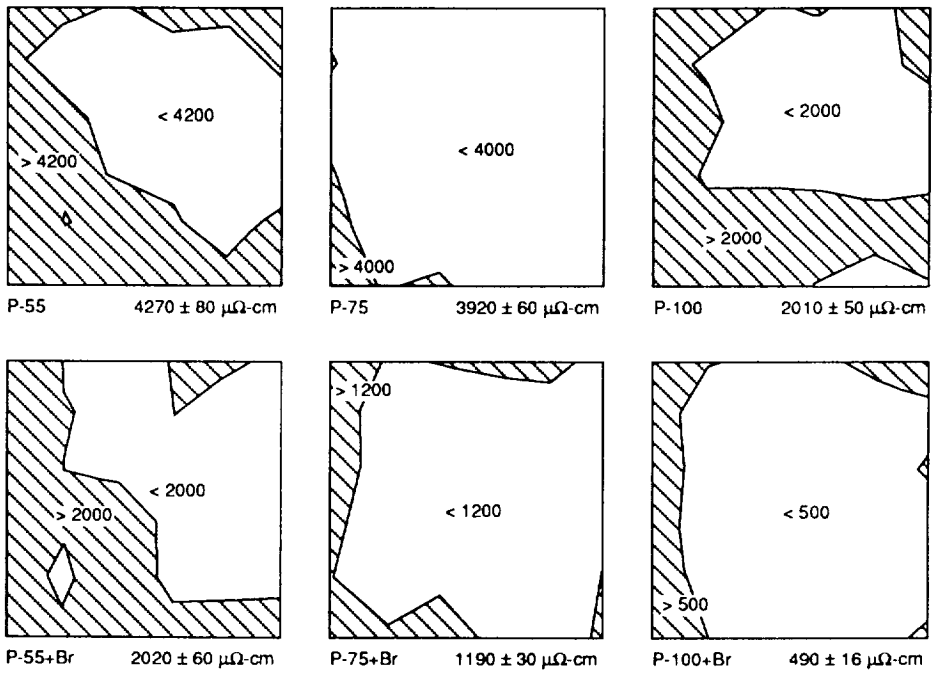


Figure 2. - Average laminate R.F. eddy current resistivity.

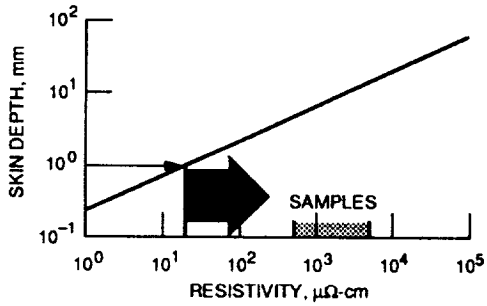
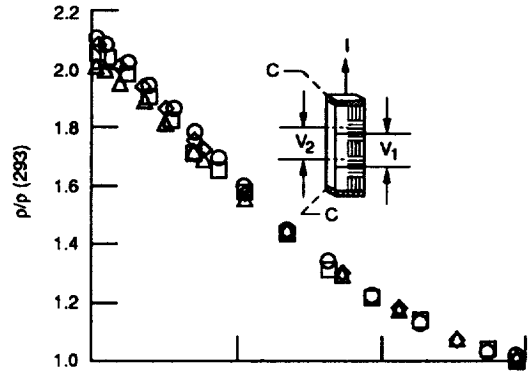
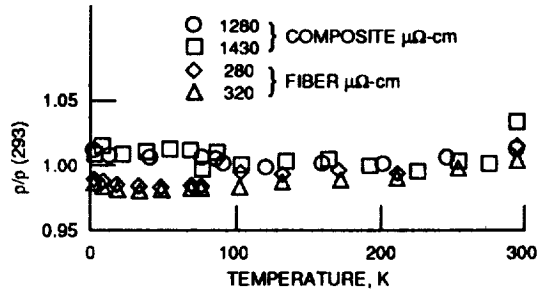


Figure 3. - Resistivity skin depth measurements using 55.55 kHz.



(a) P-100 pristine.



(b) P-100 brominated.

Figure 4. - Temperature dependence of the resistivity of pristine and bromine intercalated P-100 graphite fibers and fiber/epoxy composites.

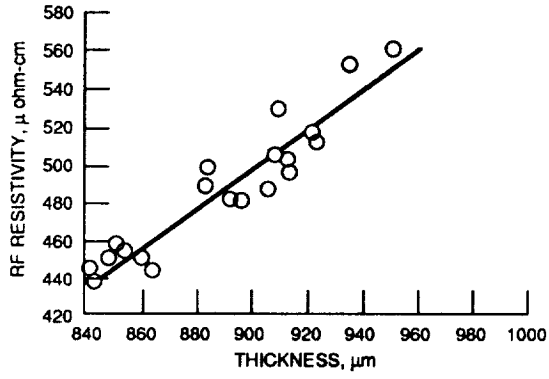


Figure 5. - Dependence of resistivity on thickness for P-100 + Br composites.

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16. Abstract

Laminar composites have been fabricated from pristine and bromine intercalated Amoco P-55, P-75, and P-100 graphite fibers and Hysol-Grafil EAG101-1 film epoxy. The thickness and r.f. eddy current resistivity of several samples were measured at grid points and averaged point by point to obtain final values. Although the values obtained this way have high precision (<3 percent deviation), the resistivity values appear to be 20 to 90 percent higher than resistivities measured on high aspect ratio samples using multi-point techniques, and by those predicted by theory. The temperature dependence of the resistivity indicates that the fibers are neither damaged nor deintercalated by the composite fabrication process. The resistivity of the composites is a function of sample thickness (i.e. resin content). Composite resistivity is dominated by fiber resistivity, so lowering the resistivity of the fibers, either through increased graphitization or intercalation, results in a lower composite resistivity. A modification of the simple rule of mixtures model appears to predict the conductivity of high aspect ratio samples measured along a fiber direction, but a directional dependence appears which is not predicted by the theory. The "resistivity" of these materials is clearly more complex than that of homogeneous materials.

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