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Technological Hurdles to the Application of Intercalated Graphite Fibers

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TECHNOLOGICAL HURDLES TO THE APPLICATION OF INTERCALATED GRAPHITE FIBERS

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SUMMARY

Before intercalated graphite fibers can be developed as an effective power material, there are several technological hurdles which must be overcome. These include the environmental stability, homogeneity and bulk properties, connection procedures, and costs. Strides have been made within the last several years in stability and homogeneity of intercalated graphite fibers. Bulk properties and connection procedures are areas of active research now. Costs are still prohibitive for all but the most demanding applications. None of these problems, however, appear to be unsolvable, and their solution may result in widespread GIC application. The development of a relatively simple technology application, such as EMI shielding, would stimulate the solution of scale-up problems. Once this technology is developed, then more demanding applications, such as power bus bars, may be possible.

INTRODUCTION

Intercalated graphite fibers have been an interesting research topic for at least 20 years (ref. 1). One of the driving forces behind this research has been their immense technological promise. This promise is based on the fundamental properties of graphite, such as high tensile strength, negative coefficient of thermal expansion, high Young's modulus in the "a" direction, very high thermal conductivity, and low density, coupled with the gains in electrical conductivity from graphite intercalation compounds. In addition, intercalation of high modulus pitch-based fibers has little effect on the fiber's mechanical properties (ref. 2). This is illustrated in table I, which compares the mechanical properties of composites made from pristine and Br₂ intercalated P-100 fibers.

However, even after 20 years, intercalated graphite fibers remain basically an interesting research topic. They have not replaced copper and aluminum as industry's favored electrical conductors. They have not, in fact, found any widespread technological use as electrical conductors. It is legitimate at this juncture to ask why. What stands in the way of utilizing these materials in existing and future technologies? And perhaps more importantly, can the problems be overcome?

PROPERTIES OF INTERCALATED GRAPHITE

A comparison of the properties of typical graphite intercalation compounds (GIC's) with metals commonly used in electrical applications reveals resistivities of the same order, but much lower densities, higher strengths, and higher moduli (table II). The fact that their resistivities are somewhat higher than copper was glossed over in earlier work by emphasizing instead the density-resistivity product (ref. 5). For applications where weight is a premium (such

as aircraft and spacecraft) this figure of merit seemed reasonable, and when this figure of merit for GIC's was compared to that of copper the results were encouraging.

If, however, the calculations would have been compared to that for aluminum, they would have noted that the figure of merit for aluminum is less than 40 percent that of copper and lower than most GIC's. One would be justified then, in questioning why aluminum has not replaced copper as the wiring standard, at least in aircraft and spacecraft. The answer is that there is more to choosing a material for an application than its resistivity and its density.

In the case of aluminum there is a technological problem which, except for major power buses in high performance aircraft, prevents its widespread use. Aluminum spontaneously forms aluminum oxide in the presence of air. Aluminum oxide is an effective electrical insulator, especially when a thick layer is formed (as in anodized aluminum). At first glance this might seem an advantage since a high temperature insulation could be easily added to the wires. Unfortunately, this same oxide layer makes aluminum unsolderable. The resulting difficulty in making strong, low resistance contacts limits its use for power buses in most applications.

Intercalated graphite fibers also have technological problems which must be overcome if they are to succeed in the market. Difficulties are encountered in the areas of chemical stability, homogeneity, bulk properties, electrical termination, and cost.

STABILITY OF INTERCALATED FIBERS

One of the most severe problems with GIC's is their instability. Much of the earliest work used Group IA (alkali metals) elements as intercalates because they form intercalation compounds easily and with complete (or nearly complete) charge transfer. Unfortunately, when these compounds are exposed to air they react very quickly with oxygen and water, and the intercalation compound is destroyed. Perhaps the first intercalation compound to be described as stable in air was that with FeCl_3 (ref. 6). This was later joined by CuCl_2 (ref. 5), SbCl_5 (ref. 7), and other transition metal chloride intercalates. Unfortunately, it was found that the intercalation compounds of these materials formed with commercially available graphite fibers, such as Amoco P-100, slowly degrade over time if they are stored in ambient laboratory air (fig. 1).

It was suspected that water was reacting with the fibers, and so the effect would increase with increasing humidity. In the extreme (100 percent humidity at 60 °C) the reaction with transition metal chlorides was devastating (refs. 8 to 10) (fig. 2). However, it was found that the residue compounds of halogen intercalates (Br_2 , ICl , IBr) were little affected by these conditions, and in fact P-100 fibers intercalated with Br_2 show no degradation at high humidities, or even submerged in water for long periods of time (years) (ref. 8).

The fibers would not, in most instances, be used by themselves but as part of a composite with a resin or metal matrix. This requires that the fibers be stable, not only at ambient conditions, but also in the processing environments used to form the composites. These will vary with the matrix material used. The most common resins for structural graphite are epoxy resins with typical

cure temperatures of 350 °F (175 °C) and cure times of several hours. Only Br₂ (ref. 9) (fig. 3), and perhaps CdCl₂ (ref. 11) intercalation compounds have shown stability at those temperatures.

GIC fibers would also be exposed to organic chemicals when being processed with these same resins. While little work has been done in this area, Br₂ intercalation compounds have proven stable in various polar and nonpolar solvents for long periods of time (weeks) (ref. 12) (table III).

If a stable GIC can be formed, will it maintain its resistivity in the presence of high electric fields, or will the intercalate molecules begin to migrate, forming a fiber with a resistivity gradient? A GIC with a migratory intercalate could not be used with high power dc circuits. Fortunately, this has proved not to be a problem with promising GIC's to date (refs. 13 and 14) (fig. 4).

Some of the important parameters which affect the stability of intercalated graphite fibers have been identified. It has been shown that the degree of crystalline perfection is important. Fibers that are more highly ordered have been found to be more susceptible to degradation by high temperature and water vapor (ref. 9). In addition, the particle size is important, with larger particles having higher stability due, at least in part, to their lower surface to volume ratios (ref. 10).

HOMOGENEITY AND BULK PROPERTIES OF INTERCALATED FIBERS

Research on intercalated graphite fibers has generally been carried out on small bundles of fibers. These are typically one or several bundles of 2000 to 6000 filaments with lengths of a few inches at most. Typical weights are in the mg region. Any application will of course involve much larger quantities, so scale-up becomes an issue.

The first question about scale-up is the homogeneity of the intercalation reaction. These are not crystalline systems with well defined properties, so how closely does one fiber resemble another? There have been only a few studies to date which have attempted to answer this question. It appears that pitch-based fibers from a single batch have homogeneous properties within a few percent, and that the decrease in resistivity due to intercalation, at least by Br₂, occurs uniformly (ref. 15) (fig. 5). With vapor-grown fibers, however, large differences can be found between even the pristine fibers within a single batch (ref. 16) (fig. 6). The fiber diameter appears to be an important parameter here, with smaller diameter fibers having higher resistivity than their larger counterparts, probably because of increasing crystallite size with diameter (ref. 17) (fig. 7). Intercalation tends to accentuate those differences since the larger crystallites also intercalate to a greater extent (ref. 16).

Presumably the uniformity of the vapor-grown fibers will improve as the technology advances to the point of commercial availability. There may be advantages in some shielding applications however, to retaining a distribution in size and resistivity.

Although the uniformity of pitch-based fiber resistivity is quite high among fibers from the same batch, there are significant batch to batch variations. Formulations of pitch based fibers are driven by their mechanical properties. This is because mechanical properties drive the current and projected

markets. As a consequence, the formulations are constantly evolving towards an improvement in strength, modulus, and strain-to-failure. Maintenance of resistivity is not a priority and, unfortunately, the trend has been to increasing resistivity (table IV). Only a large potential market can make resistivity a priority, but that market is difficult to develop when the electrical properties are degrading.

Processing is a second issue that must be resolved. Can spools of fibers, or rolls of fiber mats, be intercalated in a manner that can be reasonably cost efficient? Again, with Br₂ as the intercalate and pitch-based fibers as the host, it has been found that uniform intercalation reactions will occur under conditions similar to those used for small samples (ref. 18). Diffusion rates require that considerably more time be allowed, but the reaction is facilitated by using liquid phase intercalation instead of vapor phase.

There has been very little research reported which characterizes the electrical properties of intercalated graphite fiber composites. Since the fibers will probably be applied in composite form, this is an area which requires further research. Initial indications are that the resistivity of the composite is not simply the resistivity of the fibers divided by the fill factor. Resistivities measured using both four-point and R.F. eddy current techniques, in uniaxial (ref. 19) and 0 - 90 woven crossply (ref. 20) epoxy composites revealed that the composite resistivities were from 2.5 to 4 times higher than expected from the fiber resistivities and the fill factors (table V). It should be noted that these composites had fiber loadings ranging from 48 to 64 percent, well above the expected percolation limit.

MAKING ELECTRICAL CONNECTIONS TO INTERCALATED GRAPHITE

As was illustrated above for the case of aluminum, connection procedures can be critical to the acceptance of new technology. This promises to be a particularly difficult problem for intercalated graphite fibers, because there must be connectivity between conventional conductors and the individual fibers embedded in the insulating matrix. The problem is compounded because, not only is each filament electrically isolated from those around it, but the transverse resistivity of the fibers (at least in the case of generally available pitch-based fibers) is also poor, perhaps two orders of magnitude higher than the axial resistivity (ref. 21) (fig. 8). Connections along the edge of the composite made with nickel foil show promise (fig. 9), but moisture must be excluded (which is difficult with most conventional polymers) or corrosion will degrade the contacts (ref. 22). If there is a slow leaching of intercalate, the corrosion will be accelerated because intercalates, by their nature, are highly reactive species.

There are additional difficulties when one composite section must be joined to another. Acceptable contacts might be achieved by using a metal foil and then either a pressure contact or metal bonding, but a better solution would be a good conductive adhesive.

Contact problems make it likely that most of the first uses of intercalated graphite will be for contactless applications. Since composites of intercalated graphite fibers are very reflective in the 3.95 to 5.85 GHz range (ref. 23), applications might include electro-magnetic interference (EMI) shielding and antenna reflector surfaces.

COSTS

The central question to the development of intercalated graphite fiber materials is one of cost versus benefit. The only commercially available fibers suitable for intercalation are either pitch-based or very high quality PAN fibers. Both of these are expensive. A premium must be paid for crystalline perfection in the fiber, and the costs rise more steeply as lower resistivities are required (fig. 10). Higher production volumes will certainly decrease fiber costs, but since the fiber synthesis processes are multi-step and complex, the prospects for an inexpensive material are bleak.

Intercalation, surprisingly, could actually be a means to lower the cost of conductive fibers. For example, we have used a low temperature process developed by Hung (ref. 25) to intercalate Amoco P-55 fibers with bromine. We had previously thought P-55 fibers were not graphitic enough to intercalate. The resulting fiber has a resistivity comparable to that of pristine P-100 fibers, but P-55 fibers only cost about 6 percent of the cost of P-100. It may prove to be less costly to use intercalated P-55 fibers than pristine P-100 to achieve the same resistivity.

Vapor grown fibers show promise of being manufactured inexpensively, especially if natural gas is used as a precursor. Additionally, new micro-whiskers have been developed which have the potential of being produced at costs comparable to carbon black (ref. 26).

The added costs due to intercalation depend upon which intercalate is chosen. The raw intercalates are, for the most part, common and inexpensive materials. If the intercalation reaction can be performed at room temperature (as with Br_2 and SbCl_5), the process is simplified. A complication of many intercalates is that they are hygroscopic, thus require additional drying or purification procedures. Perhaps forming the compound at the reaction site (FeCl_3 has, for example, been formed by passing Cl_2 over iron filings at the reaction site (ref. 27)) will be an effective strategy. In general the intercalation process need not add enormously to the cost of the fibers.

CONCLUSIONS

In summary, there are many problems associated with the development of intercalated graphite fibers as a cost effective power material. None of the problems discussed above, however, appear to be unsolvable, and their solution may result in widespread GIC application. The development of a relatively simple technology application, such as EMI shielding, would stimulate the solution of scale-up problems. Once this technology is developed, then the more demanding applications, such as power bus bars, may be possible.

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TABLE I. - PROPERTIES OF COMPOSITES MADE FROM PRISTINE
AND BROMINE INTERCALATED GRAPHITE FIBERS
[Refs. 2 to 4.]

| | Pristine P-100 | P-100 + Br ₂ |
|---------------------------------------------|----------------|-------------------------|
| Mechanical | | |
| Tensile modulus, GPa | 430±50 | 430±50 |
| Tensile strength, MPa | 840±110 | 840±50 |
| Poisson's ratio | 0.36±0.09 | 0.30±0.14 |
| Interlaminar shear, MPa | 26.2±1.0 | 30.9±0.8 |
| Bending modulus, GPa | 338±8 | 296±3 |
| Flexure strength, MPa | 540±30 | 560±10 |
| "a" axis CTE, ^a ppm/K | -2.1 | -1.8 |
| "c" axis CTE, ^a ppm/K | 41 | 41 |
| Density, g/cm ³ | 1.852 | 1.873 |
| Transport | | |
| Axial resistivity, μΩ-cm | 459±17 | 89±4 |
| Transverse resistivity, mΩ-cm | 528 | 617 |
| Thermal conductivity, ^b W/m-K | 170 | 170 |

^aCoefficient of thermal expansion.

^bCalculated from fiber value from ref. 4 and fiber volume of ref. 2.

TABLE II. - PROPERTIES OF POTENTIAL POWER MATERIALS

| | Strength, MPa | Modulus, GPa | Density, g/cc | Resistivity, μΩ-cm | RD |
|--------------------------------------|------------------|-----------------|------------------|-----------------------|------|
| Copper ^a | 420 | --- | 8.92 | 1.77 | 15.8 |
| Aluminum ^a | 210 | --- | 2.70 | 2.82 | 7.6 |
| T-300 ^b | 3200 | 228 | 1.76 | 2210 | 3890 |
| P-100 ^c | 840 | 433 | 2.18 | 250 | 545 |
| P-100 + Br ₂ ^c | 840 | 427 | 2.30 | 50 | 160 |
| VGF + Br ₂ ^d | 1000 | 200 | 2.35 | 8 | 18.8 |

^aRef. 28.

^bRef. 27.

^cRef. 2.

^dRef. 30.

TABLE III. - THE RESISTANCE RATIO (RESISTANCE/
INITIAL RESISTANCE) OF BROMINATED
P-100 GRAPHITE FIBERS STORED
IN SOLVENTS OVER TIME

| Solvent | 5 days | 24 days | 9 months |
|----------------------------|--------|----------|----------|
| Water | 1.00 | 1.00 | 1.00 |
| Methanol | 1.00 | 1.06±.03 | 1.04±.03 |
| Ethanol | 1.00 | 1.00 | 1.00 |
| 2-Propanol | 1.00 | 1.00 | 1.00 |
| Benzyl alcohol | 1.00 | 1.00 | 1.00 |
| Acetone | 1.00 | 1.00 | 1.03±.03 |
| Chloroform | 1.00 | 1.00 | 1.00 |
| Bromoform | 1.00 | (a) | (a) |
| Carbon- tetrachloride | 1.00 | (a) | (a) |
| 1,1,1-Tri- chloroethane | 1.00 | 1.00 | (a) |
| Butyl Acetate | 1.00 | 1.00 | 1.00 |
| Toluene | 1.00 | 1.00 | 1.02±.01 |

^aCarbon paint used to affix the samples
dissolved and fibers were lost.

TABLE IV. - RESISTIVITY OF PITCH-
BASED FIBER PURCHASED OVER TIME

| Year | P-55 | P-75 | P-100 | P-120 |
|------|------|------|-------|-------|
| 1984 | 910 | 510 | 250 | 250 |
| 1986 | 990 | 910 | --- | --- |
| 1987 | --- | 700 | 350 | --- |

TABLE V. - RF AND FOUR-POINT RESISTIVITIES OF COMPOSITES

| | RF eddy current, $\mu\Omega$ -cm | Four point, $\mu\Omega$ -cm | Calculated resistivity, $\mu\Omega$ -cm |
|-------------------------|-------------------------------------|--------------------------------|-----------------------------------------------|
| P-55 | 4100±140 | 4370 | 1700 |
| P-75 | 3800±130 | 4010 | 1400 |
| P-100 | 1950±150 | 1530 | 470 |
| P-55 + Br ₂ | 2990±80 | 2040 | 650 |
| P-75 + Br ₂ | 1070±35 | 1300 | 430 |
| P-100 + Br ₂ | 560±30 | 650 | 170 |

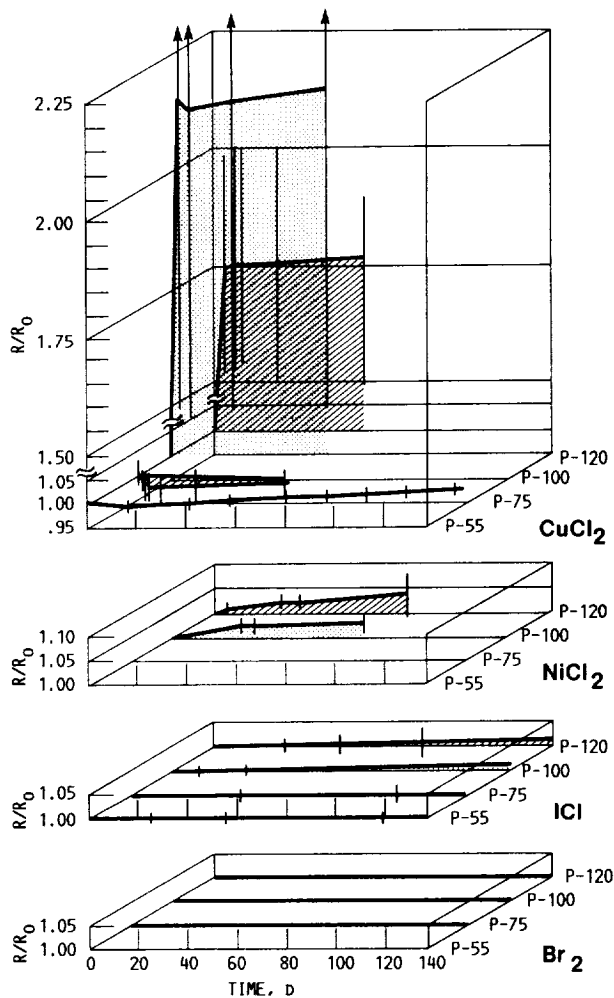


FIGURE 1. - RESISTANCE RATIO (RESISTANCE, R /INITIAL RESISTANCE, R_0) AS A FUNCTION OF TIME EXPOSED TO AMBIENT AIR FOR P-120, P-100, P-75, AND P-55 GRAPHITE FIBERS INTERCALATED WITH BROMINE, IODINE MONOCHLORIDE, COPPER (II) CHLORIDE, AND NICKEL (II) CHLORIDE. VERTICAL LINES INDICATE SAMPLE STANDARD DEVIATION.

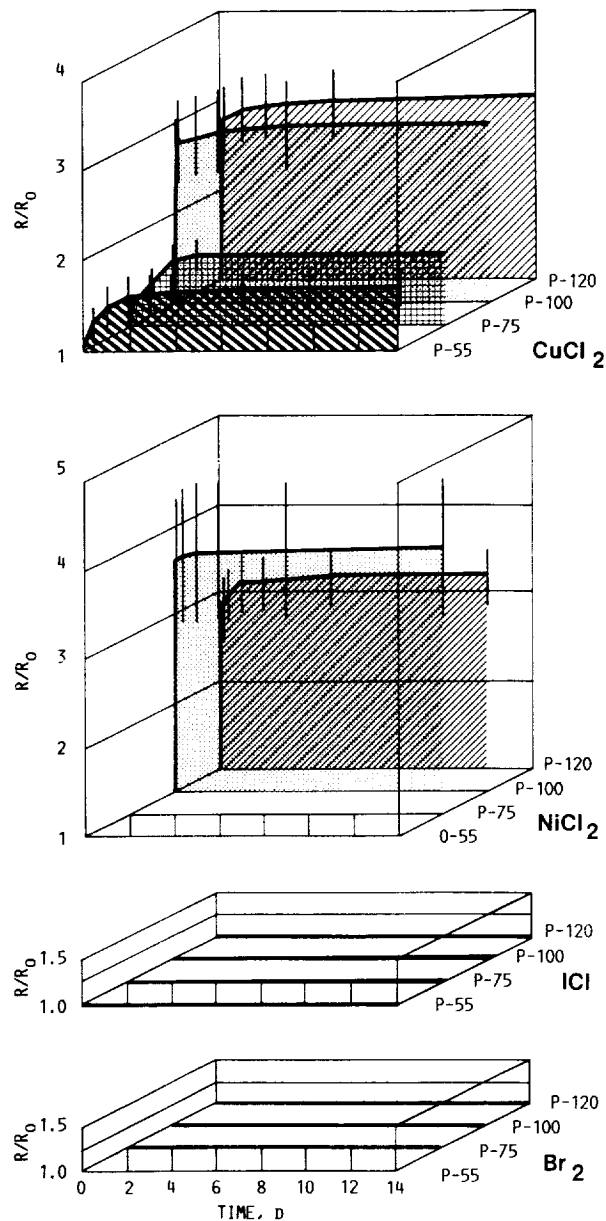


FIGURE 2. - RESISTANCE RATIO (RESISTANCE, R /INITIAL RESISTANCE, R_0) AS A FUNCTION OF TIME EXPOSED TO 100 PERCENT HUMIDITY AT 60 °C FOR P-120, P-100, P-75, AND P-55 GRAPHITE FIBERS INTERCALATED WITH BROMINE, IODINE MONOCHLORIDE, COPPER (II) CHLORIDE, AND NICKEL (II) CHLORIDE. VERTICAL LINES INDICATE SAMPLE STANDARD DEVIATION.

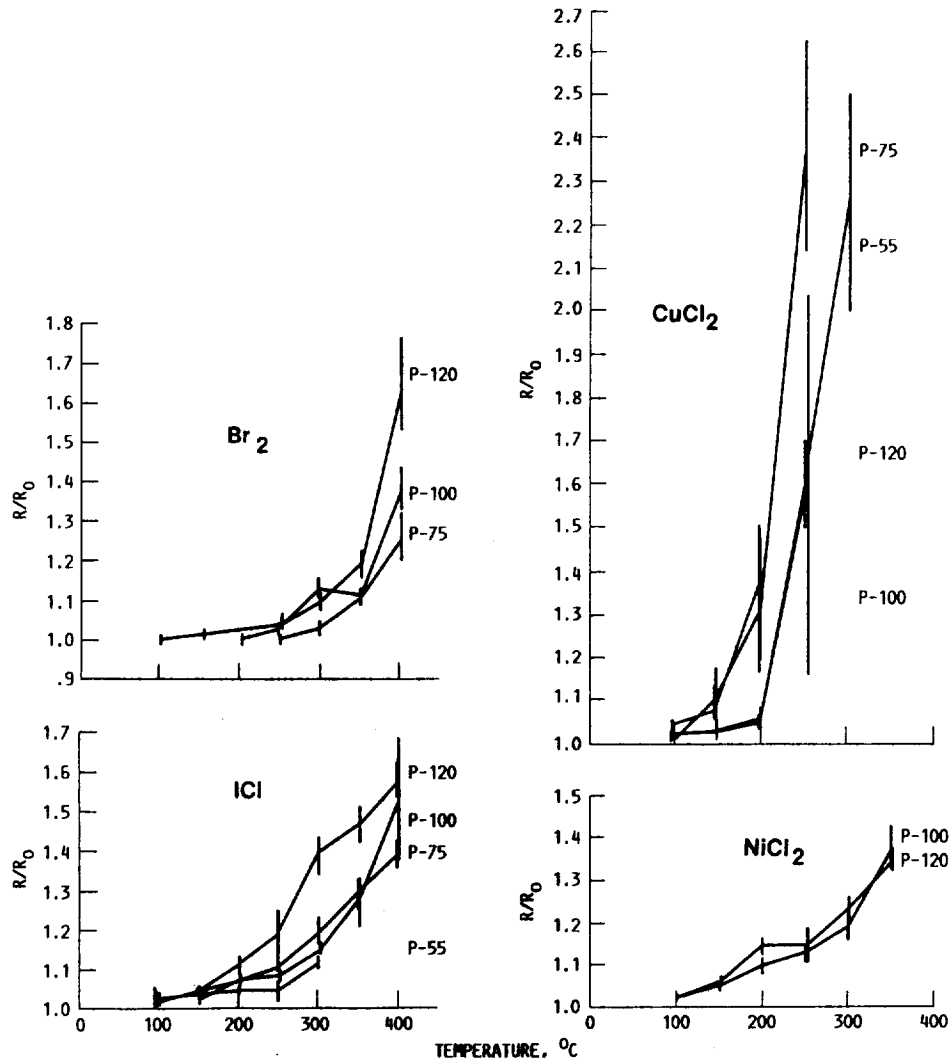


FIGURE 3. - AVERAGE IN RESISTANCE RATIO AS A FUNCTION OF TEMPERATURE FOR P-55, P-75, P-100, AND P-120 GRAPHITE FIBERS WITH VARIOUS INTERCALATES: (A) BROMINE, (B) IODINE MONOCHLORIDE, (C) COPPER (II) CHLORIDE, NICKEL (II) CHLORIDE.

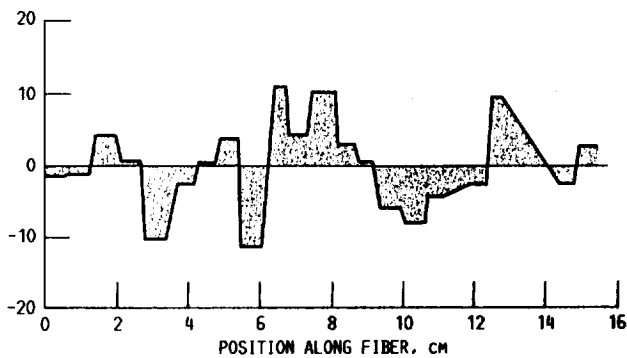


FIGURE 4. - EFFECT OF HIGH CURRENT DENSITY UPON THE RESISTANCE OF A SINGLE BROMINE INTERCALATED FIBER. GRAPH SHOWS THE DEVIATION FROM THE MEAN OF THE RESISTANCE OF 21 REGIONS ALONG THE FIBER LENGTH.

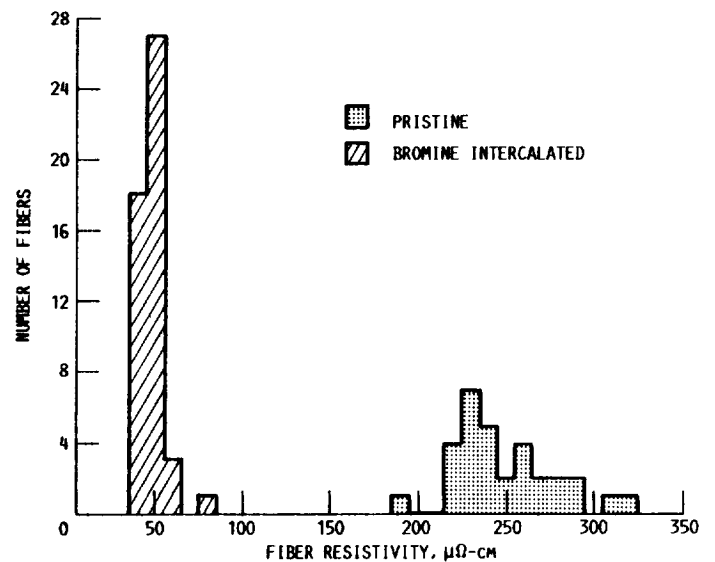


FIGURE 5. - DISTRIBUTION OF PRISTINE AND BROMINE INTERCALATED P-100 FIBER RESISTIVITIES.

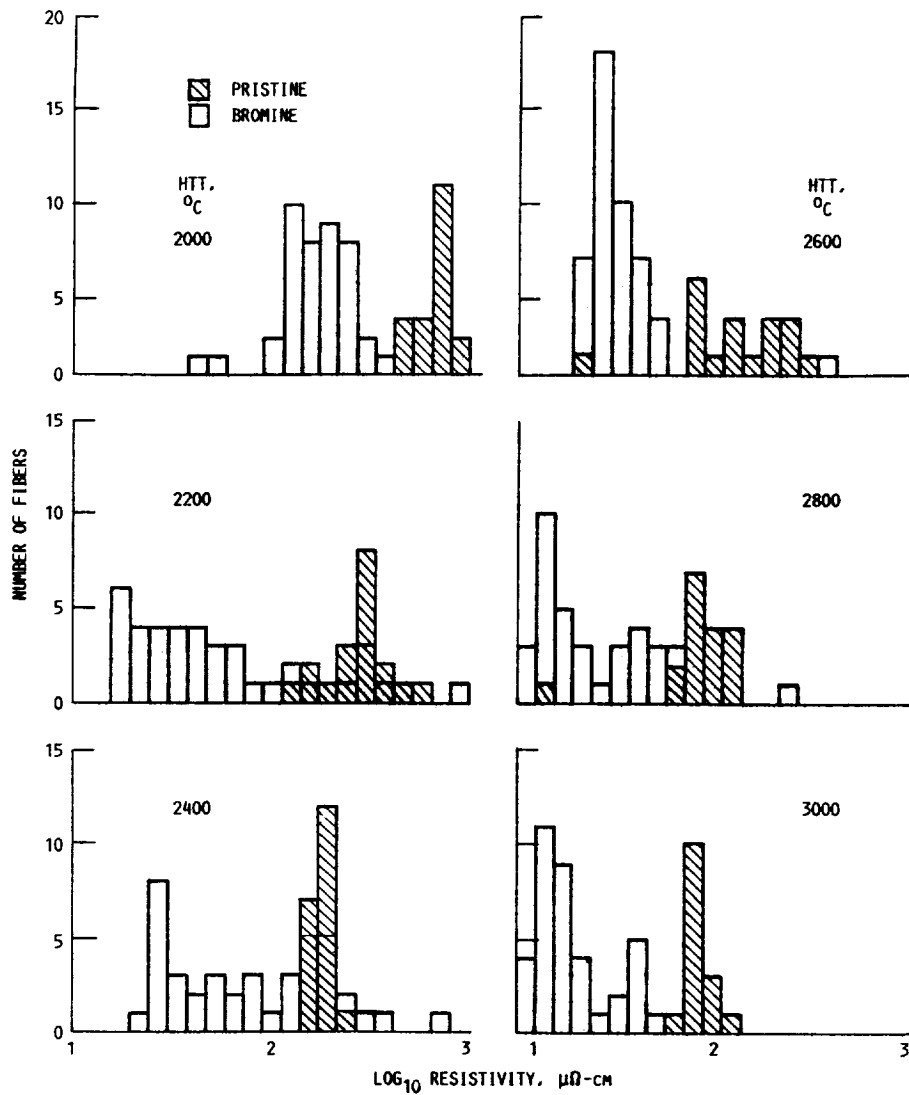


FIGURE 6. - RESISTIVITIES OF PRISTINE VAPOR-GROWN GRAPHITE FIBERS AT VARIOUS HEAT-TREATMENT TEMPERATURES AND THEIR RESPECTIVE BROMINATION COMPOUNDS.

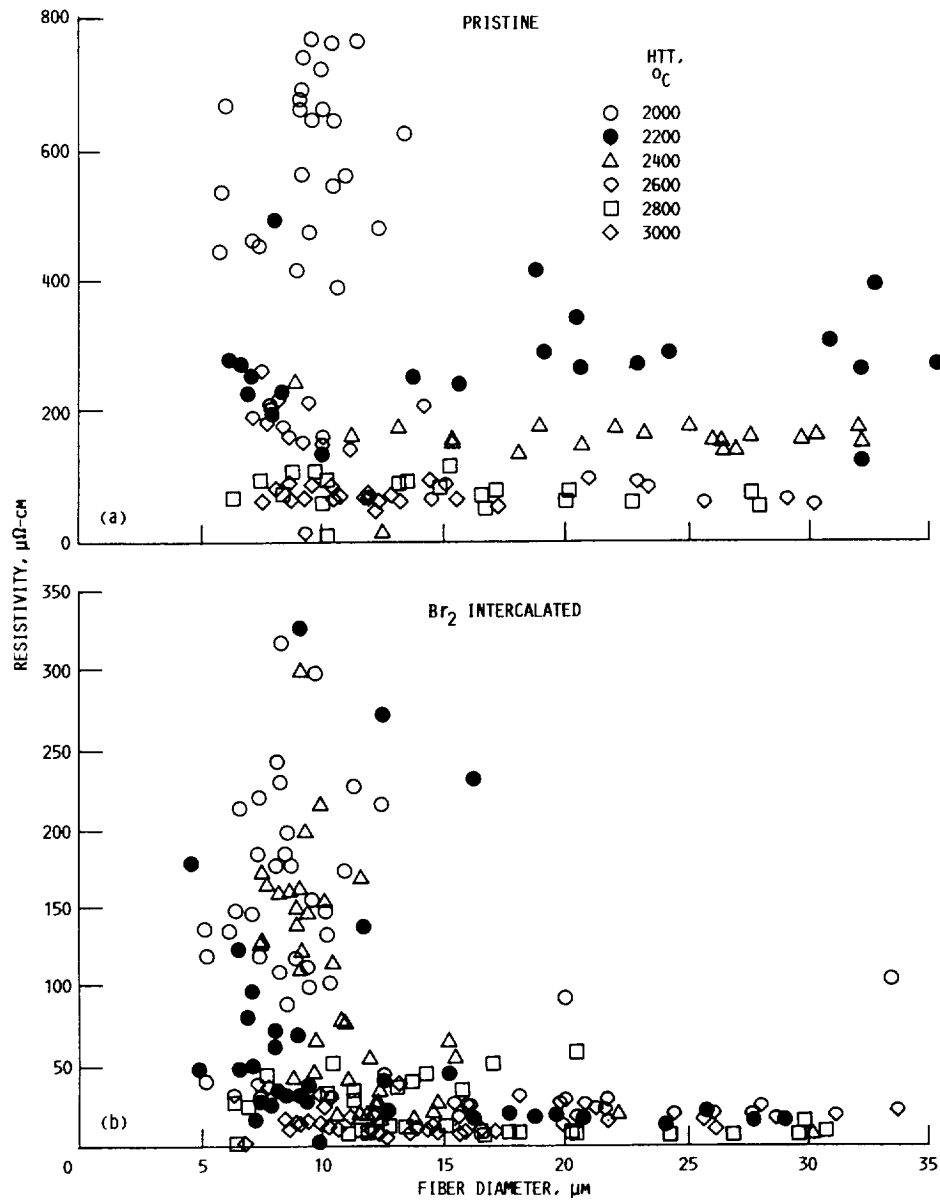


FIGURE 7. - INDIVIDUAL PRISTINE (A) AND BROMINE INTERCALATED (B) FIBER RESISTIVITIES AS A FUNCTION OF FIBER DIAMETER.

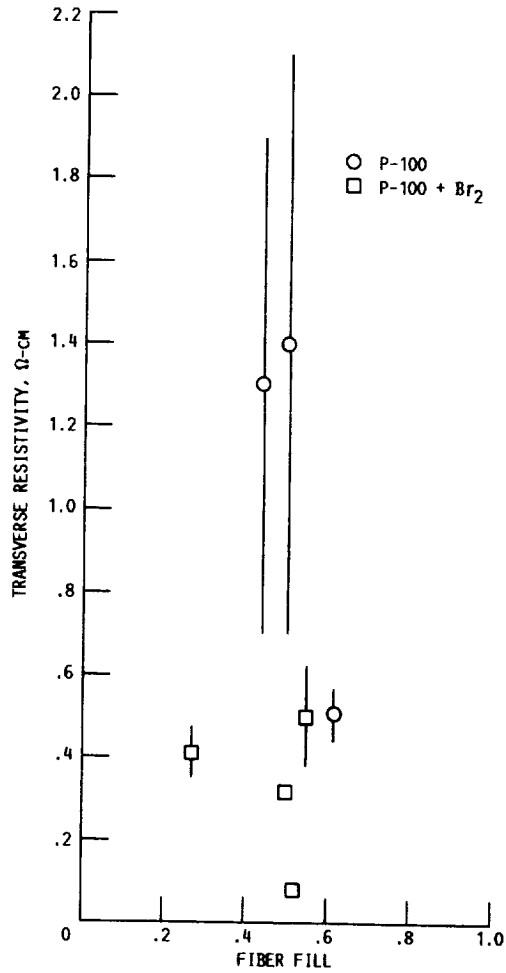


FIGURE 8. - TRANSVERSE RESISTIVITY OF COMPOSITES AT VARIOUS FIBER VOLUME FRACTIONS.

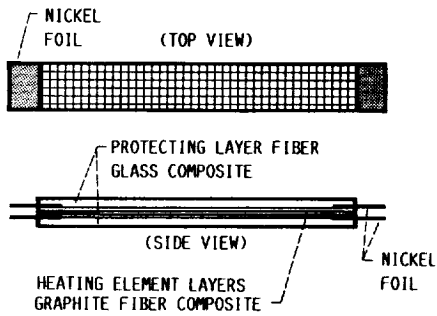


FIGURE 9. - METHOD OF MAKING ELECTRICAL CONTACTS TO GRAPHITE-EPOXY COMPOSITES.

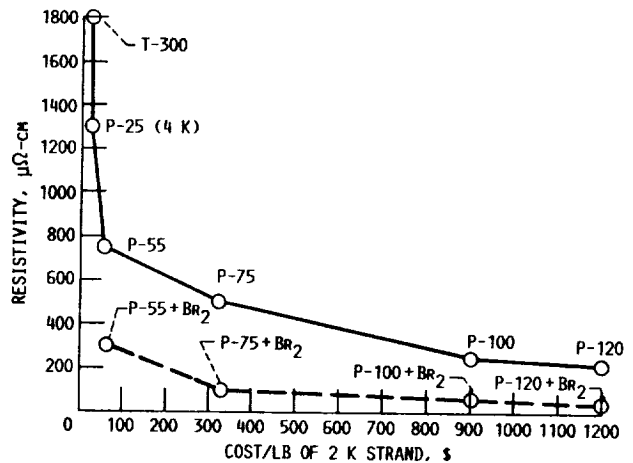


FIGURE 10. - RESISTIVITY OF SEVERAL GRADES OF PRISTINE AND BROMINE INTERCALATED GRAPHITE FIBERS AS A FUNCTION OF COST (1986).

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