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Graphite Fiber Intercalation: Basic Properties of Copper Chloride Intercalated Fibers
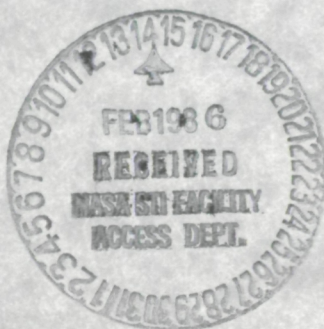
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GRAPHITE FIBER INTERCALATION: BASIC PROPERTIES OF

COPPER CHLORIDE INTERCALATED FIBERS

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SUMMARY

In situ resistance measurements were used to follow the intercalation of copper chloride in pitch-based fibers. Subsequent single fiber resistivity measurements revealed a large range of resistivities, from 13 to 160 $\mu\Omega$-cm. Additional density measurements revealed a bimodal distribution of mass densities. The denser fibers had lower resistivities and corresponded to the stage III compound identified by x-ray diffraction. Neither resistivity nor density correlated with diameter. Both energy dispersive spectroscopy and mass density data suggested that excess chlorine resided in the intercalated fiber, resulting in a stoichiometry of $C_{4.9n}CuCl_{2.5}$ (where n is the stage number) for the denser fibers. Finally, thermogravimetric analysis showed a 33 percent loss in mass upon heating to 700 °C. This loss in mass was attributed to loss of both chlorine and carbon.

INTRODUCTION

Graphite fiber intercalation may prove to be beneficial to the aerospace industry in the fabrication of lightweight, high-strength, electrically conductive composite materials. Before fabricating composites, the basic properties of these new graphite fibers must first be characterized. One of the graphite intercalation compounds which is currently under investigation is the copper chloride graphite intercalation compound.

Copper chloride graphite intercalation compounds have been studied in several different graphite fiber host systems. Endo et al. (ref. 1) have intercalated copper chloride in benzene derived fibers and have reported a room temperature resistivity of 9 $\mu\Omega$-cm. They have also reported that these benzene derived copper chloride intercalated fibers were stable in argon atmospheres up to 200 °C for at least 12 hr. Oshima et al. (refs. 2 and 3) have successfully intercalated copper chloride in pitch-based carbon fibers. Electrical resistivity values in the range of 12.9 to 38.3 $\mu\Omega$-cm were reported for a mixed-stage fiber product. These pitch-based copper chloride intercalated fibers exhibited thermal stability up to 177 °C in air. Gaier and Jaworske (ref. 4) have documented the long-term stability of similar copper chloride intercalated pitch-based fibers under both ambient laboratory conditions and vacuum. Low electrical resistivity combined with favorable thermal stability

make copper chloride intercalated graphite fibers a promising candidate for use in aerospace applications where there is an absence of moisture.

This paper presents the results of three sets of experiments designed to study the basic properties of copper chloride intercalated P-100 fibers. First, in situ resistivity measurements obtained during a $CuCl_2$ intercalation of a single fiber will be presented. These results give some indication of the dynamics of the $CuCl_2$ intercalation process. Next, results from diameter, resistivity, density, and energy dispersive spectroscopy measurements on single fibers will be summarized and correlated. These single fiber measurements serve to characterize the copper chloride intercalated fiber product in terms of variability from one fiber to the next. Finally, x-ray and thermogravimetry results will be discussed. These results are applied to bulk quantities of fibers and help to determine the characteristics of the intercalated fiber product.

METHODS

Union Carbide pitch-based P-100 graphite fibers were chosen for this study because they were commercially available and came in spools of arbitrary length. Their initial resistivity was 250 $\mu\Omega$-cm (ref. 5). The P-100 fibers were also known to intercalate copper chloride (refs. 2 and 3).

The anhydrous cupric chloride (Noah Chemical Co.) was placed in an all-glass ampoule along with the P-100 fibers. The $CuCl_2$ was then dried under vacuum overnight at 100 °C to remove any residual water. After drying, the ampoule was sealed off under vacuum, and the intercalation was initiated by heating the ampoule incrementally to 480 °C. At this temperature, the vapor pressure of $CuCl_2$, Cl_2, and Cu_3Cl_3 was estimated to be 0.32, 16.77, and 1.70 torr, respectively (ref. 6). The furnace was held at this temperature for 16 hr, more than twice as long as suggested by Oshima et al. (ref. 2 and 3).

In addition to the bundle of P-100 fibers, a single P-100 fiber was mounted on four platinum leads and sealed in a side arm of the ampoule. The leads were connected to a Keithley model 220 constant current source and a Keithley model 181 nanovoltmeter in the conventional four-point configuration. Voltage data were obtained at 100 μA (every 30 sec), and were stored on an IBM computer for retrieval later.

Before terminating the intercalation, the temperature of the furnace was incremented to 540 °C to see if a further decrease in fiber resistance would occur. No significant change was observed. The ampoule was then allowed to cool, and the fibers were removed. No special precautions were taken for the storage of the intercalated fibers in the ambient laboratory atmosphere.

Forty-six single fibers from the bundle of intercalated P-100 fibers were mounted on four-point sample holders for resistance measurements. Again, the current for the four-point probe was 100 μA. One piece of each fiber was broken off and used for diameter and energy dispersive spectroscopy (EDS) measurements. Another piece of each fiber, approximately 2 to 3 mm long, was placed in a calibrated bromoform and carbon tetrachloride (Fischer Scientific) density gradient column. After each fiber descended to its equilibrium position, the density of that fiber was recorded.

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A Perkin-Elmer TGS-2 was used for the thermogravimetric analysis of bulk quantities of $CuCl_2$ intercalated fibers. The purge gas was nitrogen.

RESULTS AND DISCUSSION

In situ Resistance Measurements

The results of the in situ fiber resistance measurements are presented in figure 1. The first four plateaus represent the resistance ratios (resistance/ initial resistance, R/R_0) at the four temperature increments of 100, 200, 300, and 400 °C. The declines in R/R_0 indicate the negative temperature dependence of the resistance of the pristine fiber. At 480 °C, the shape of the curve is characterized by a substantial drop in resistance (signaling the onset of intercalation), followed by a more gradual decline. This behavior is similar to that observed for other intercalates, such as HNO_3 and Br_2 (ref. 7 and 8). After 16 hr at 480 °C, the in situ fiber was still showing a gradual decline in resistance. When the fiber was cooled, a positive temperature dependence of the resistance was observed, and at room temperature the final resistance was a factor of 3.4 lower than the original value. This result, taken alone, would suggest that the final room temperature resistivity of the copper chloride intercalated product was on the order of 70 $\mu\Omega$-cm.

The in situ measurement only represented one fiber; however, it was useful in following the progress of the intercalation reaction. The resistance data were collected for 16 hr, and about half of the observed decrease in resistance occurred within the first 1-1/2 hr.

Single Fiber Measurements

To determine the distribution of the copper chloride intercalated fiber resistivities, 46 single fibers from the bundle of fibers in the ampoule were mounted on four-point sample holders for resistance measurements, and pieces of each fiber were mounted on SEM sample holders for diameter measurements. The copper chloride intercalated P-100 fiber resistivities are summarized in the histogram shown in figure 2, along with resistivities obtained by Gaier (ref. 9) for pristine P-100 fibers. There is a surprisingly broad distribution of intercalated fiber resistivities, from 13 to 166 $\mu\Omega$-cm. Two of the fibers did not appear to intercalate at all. The peak of the skewed intercalated fiber distribution occurs at 20 $\mu\Omega$-cm (reduced from the pristine value by a factor of 13), while the average of the intercalated fiber distribution occurs at 60 $\mu\Omega$-cm (reduced from the pristine value by a factor of 4). The copper chloride intercalated fiber resistivity data presented here compare quite favorably with that of Oshima et al. (ref. 3), particularly at the lower end of the distribution.

Diameter measurements for both pristine (ref. 9) and copper chloride intercalated P-100 fibers are summarized in figure 3. Again, there is a surprisingly broad distribution in fiber diameters after intercalation, ranging from 9.2 to 17.0 μm. The average diameter of the intercalated fibers (± the standard deviation) is 12.1±2.1 μm, compared to 9.1±0.7 μm for the pristine P-100 fibers.

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The histogram in figure 4, summarizing the density results, reveals the reason for the large spread in the resistivity and diameter data. There were two distinct populations of copper chloride intercalated fibers, one with an average density of 2.25 ± 0.04 g/cm^3 and one with an average density of 2.53 ± 0.02 g/cm^3. Both populations were denser than their pristine counterpart, which had a density of 2.18 ± 0.02 g/cm^3. Apparently some fibers were intercalated more than others. It is difficult to determine the cause of this bimodal distribution. Presumably, all of the fibers were in the same chemical environment. There may be differences in the microstructure of the fiber, which were enhanced by copper chloride intercalation. However, Gaier (ref. 9) concludes that the pristine P-100 fibers are homogeneous to within 5 percent. Further experimental work is needed to understand the reason for this bimodal distribution.

Energy dispersive spectroscopy data were collected on each of the 46 fibers. Although this technique does not yield absolute values because of the differences in sample orientation and distance from the detector, the relative concentrations of copper and chlorine were clearly indicated. The copper and chlorine data are summarized in figure 5. The linear correlation between copper and chlorine indicates that there was no enrichment of chlorine between the two different density populations of $CuCl_2$ intercalated fibers. Therefore, the denser population does not have a greater fraction of chlorine than the lighter population. The data, however, do suggest a stoichiometric excess of chlorine in both populations, as suggested by the slope of the line in figure 5.

To glean the stoichiometry from the EDS data, a cuprous chloride standard (J.T. Baker Chemical Co.) was subjected to similar EDS analysis. The results indicate that the EDS system was more sensitive to chlorine than copper by a factor of 1.34. To estimate the ratio of copper to chlorine in the intercalated fiber product, the slope of figure 5 was divided by the 1.34 correction factor. The corrected stoichiometry obtained in this manner was $CuCl_{2.51}$. The chlorine in excess of $CuCl_2$ was attributed to atomic or molecular chlorine trapped in the intercalation compound along with the cupric chloride.

Excess chlorine has been observed before in other metal chloride graphite intercalation compounds. For example, Dzurus and Hennig (ref. 10) have studied the graphite - aluminum chloride - chlorine system in detail. Quantitative analysis of various stage I and stage II compounds have revealed a 3:1 ratio of $AlCl_3$ to Cl. This translates to a stoichiometry of $C_nAlCl_{3.3}$, where n is a function of the stage.

All of the values for diameter, resistivity, density, copper concentration, and chlorine concentration are summarized in table I. Various combinations of these data were examined for correlation. No clear-cut correlation was observed between diameter and resistivity, or diameter and density; however, there was a grouping of data in the resistivity versus density plot, as shown in figure 6. All of the denser fibers had a resistivity value near 20 $\mu\Omega$-cm while the lighter fibers varied between 50 and 160 $\mu\Omega$-cm. The denser group of fibers may all be of similar stage or composition while the lighter fibers may represent a mixture of higher stages or less concentrated compositions.

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Bulk Fiber Measurements

The stage of the copper chloride intercalated fiber product was determined by x-ray diffraction. The analysis of a small bundle of fibers showed a fairly strong pristine peak and two slightly broader peaks, identified as stage III peaks. This result leads one to believe that the fibers with higher density and lower resistivity were intercalated to stage III, while the lighter fibers were only partially intercalated to some higher, unknown mixed stage.

Next, the stoichiometric ratio of chlorine (x) in the intercalation compound was calculated by comparing measured densities with known lattice spacings. This was done by using the measured density of the pristine fibers (2.18 ± 0.02 g/cm^3), the measured density of the intercalated fibers (2.53 ± 0.02 g/cm^3), the known stage of the intercalated fibers (n = 3), the known lattice spacings for both the pristine and copper chloride intercalated compounds (3.35 ± 0.01 Å and 9.40 ± 0.01 Å, respectively, ref. 11), and the currently accepted stoichiometry between carbon and copper in the copper chloride intercalation compound ($C_{4.9n}$ $CuCl_x$, ref. 12). Using equation (1), a value of 2.49 ± 0.06 was obtained for the stoichiometric ratio of chlorine.

$$\frac{\left\{\dfrac{\text{(mass of C + mass of Cu + mass of Cl)}}{\text{intercalated lattice spacing}}\right\}}{\left\{\dfrac{\text{mass of C}}{\text{pristine lattice spacing}}\right\}} = \frac{\text{density of intercalated fiber}}{\text{density of pristine fiber}}$$

$$\frac{\left\{\dfrac{(4.9)\,(3)\,(12.01) + 63.54 + (x)\,(35.45)}{9.40 + (2)\,(3.35)}\right\}}{\left\{\dfrac{(4.9)\,(12.01)}{(3.35)}\right\}} = \frac{2.53}{2.18}$$

(1)

This calculated value compared quite favorably with the estimated value of 2.51, obtained from the EDS data.

Several small bundles of $CuCl_2$ intercalated fibers were subjected to thermogravimetric analysis (TGA) in order to drive off the intercalate and determine the percentage of intercalate in the intercalation compound. A typical TGA curve, obtained from a 184 µg sample of fibers, is shown in figure 7. According to the TGA results, the observed mass loss is in the range of 31 to 37 percent, and occurs between 300 and 400 °C. There is some hint of a two step decomposition process, as suggested by the derivative curve shown in the lower portion of figure 7. From a speculative point of view, one of the decomposition steps may be due to the decomposition of the denser set of fibers while the other step may be due to the decomposition of the lighter set of fibers. The TGA results reported here differ from the TGA results reported by Davis, McGhie, and Wright (ref. 13) for stage II $CuCl_2$ inter- calated graphite powder. According to Davis et al., the mass loss in the pow- der is on the order of 50 percent and occurs primarily between 400 and 600 °C. Apparently, the graphite host not only plays an important role in the inter- calation process, it plays an important role in the decomposition process as well.

In addition to the nonhomogeneous nature of the bulk intercalated fiber product (as shown by the density data), another piece of information had to be considered during the interpretation of the TGA results. After heating the fibers under N_2 to 700 °C and observing a mass loss of approximately 33 percent, the fibers were subjected to EDS analysis to confirm the loss of intercalate. The surprising result of the subsequent EDS experiment was the presence of both copper and chlorine in fibers that were presumably deinter- calated. The EDS results on the heated fibers are presented in table II. The average intensity of the copper signal in the fibers that were heated is about the same as in those that were not heated (1300±152 counts versus 1800±1000 counts, respectively), suggesting no significant loss of copper. However, the average intensity of the chlorine signal in the fibers that were heated is significantly lower than in those that were not heated (2338±279 versus 5547± 3530 counts, respectively). Hence, the ratio of chlorine to copper is markedly different. The average values, corrected for differences in elemental sensi- tivity, suggest that the copper to chlorine ratio after heating to 700 °C is about 1:1.4. If the stoichiometry $C_{4.9(3)}$ $CuCl_{2.5}$ is valid prior to heat- ing, then the $Cl_{1.1}$ lost during heating accounts for about 12 percent of the mass loss. (However, only half of the fibers exhibited this stoichiometry, making 12 percent an upper limit and 6 percent a lower limit.) If there is no loss of copper, then the balance of the mass loss must be due to the loss of carbon. If there was a trace of oxygen in the nitrogen used to purge the TGA, then oxidation of the fiber may further cloud the issue. The specific mechan- ism by which carbon and chlorine are lost remains unclear, but it may involve chlorinated fragments of the carbon lattice. The TGA system is currently being refitted with a mass spectrometer to address this question.

CONCLUSIONS

Copper chloride intercalated P-100 fibers prepared at 480 °C exhibited resistivity values ranging from 13 to 160 $\mu\Omega$-cm, with an average resistivity of 60 $\mu\Omega$-cm. This corresponded to a four-fold reduction in fiber resistivity. Likewise, a large distribution in fiber diameters was observed, with values ranging from 9.2 to 17.0 μm. This large variation in the intercalated fiber product was apparently due to nonuniform intercalation of the bundle of fibers. Density measurements confirmed that some fibers had more intercalate than others. The density values formed two distributions, one with an average of 2.53±0.02 g/cm^3 and the other with an average of 2.25±0.04 g/cm^3. The denser fibers had lower resistivities and were identified by x-ray diffraction as the stage III compound. The lighter fibers were of some higher, unknown stage. Before copper chloride intercalated fibers are prepared in kilogram quantities for use in aerospace composites, additional improvements will be needed to increase the yield of the low-stage compound.

Energy dispersive spectroscopy measurements and independent calculations based on density and lattice spacing revealed that there was an excess of chlorine in the copper chloride intercalated fibers. Both methods gave a stoichiometry of $C_{4.9n}$ $CuCl_{2.5}$. Although excess chlorine has been reported in the graphite flake - aluminum chloride system before, we believe that this is the first indication of excess chlorine in the graphite fiber - copper chloride system. The excess chlorine may play an important role in the electrical performance of the intercalated fiber product.

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Finally, thermogravimetric analysis of the copper chloride intercalated fiber product clearly showed a mass loss of 33 percent. However, subsequent energy dispersive spectroscopy analysis showed a significant amount of copper remaining in the fibers. Hence, mass loss may not always indicate loss of intercalate.

ACKNOWLEDGMENTS

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TABLE I. - SUMMARY OF DIAMETER, RESISTIVITY, DENSITY COPPER, CONCENTRATION, AND CHLORINE CONCENTRATION DATA

Diameter, μm	Resistivity, μΩ-cm	Density, g/cm³	Copper, counts at 8.0 keV	Chlorine, counts at 2.6 keV
9.1	88	2.249	612	1497
9.2	89	2.221	835	3253
9.3	14	2.544	512	1692
9.4	16	2.548	2049	6647
9.7	114	2.218	1325	1601
9.7	120	2.228	410	1253
9.8	28	2.251	464	981
10.1	166	2.198	976	1025
10.2	---	2.545	2197	5735
10.4	53	2.448	2221	7505
10.5	157	2.230	982	3583
10.5	100	2.248	----	----
10.5	13	-----	2674	9093
10.8	267	2.098	----	----
10.8	---	2.332	1685	3489
10.9	62	2.200	494	1211
10.9	122	2.236	958	3364
11.3	20	2.539	2652	8767
11.4	114	2.234	463	1554
11.5	50	2.279	471	1488
11.5	---	2.520	2479	5830
11.7	163	2.220	469	1585
11.7	---	-----	1841	5840
11.8	58	2.263	584	1900
11.8	27	2.512	2416	6200
11.9	25	2.554	1764	5658
11.9	16	2.526	2824	9457
12.0	20	2.550	3314	11365
12.1	158	2.246	2319	8800
12.2	24	2.312	1423	2913
13.0	23	2.505	2018	6453
13.0	24	2.509	1946	6477
13.0	163	2.221	632	2228
13.0	15	2.524	2719	8042
13.5	253	2.213	423	1298
13.6	23	2.542	2115	6475
13.8	80	2.327	2224	8006
13.9	22	2.539	3250	10950
14.4	70	-----	908	3102
14.9	22	2.539	3438	11616
15.0	24	2.550	3181	10596
15.3	23	2.530	2127	6910
15.5	23	2.535	3537	11758
16.2	29	2.501	3687	11541
16.5	32	2.350	----	-----
17.0	48	2.223	2828	9795

TABLE II. – ENERGY DISPERSIVE SPECTROSCOPY RESULTS FOR
THE COPPER AND CHLORINE CONCENTRATIONS IN
FIBERS HEATED TO 700 °C

Copper, counts at 8.0 keV	Chlorine, counts at 2.6 keV	Corrected chlorine[a] counts	Chlorine/copper
1520	2629	1962	1.291
1183	2315	1728	1.461
1394	2716	2027	1.454
1357	2362	1763	1.299
1066	2575	1922	1.803
1242	1937	1446	1.164
1314	2129	1589	1.209
1385	2027	1513	1.092
1323	2140	1597	1.207
1461	2726	2034	1.392
1334	2502	1867	1.400
1109	2004	1496	1.349
1038	2038	1521	1.465
1468	2636	1967	1.340

[a]These chlorine values were corrected by dividing the
measured value by 1.34 (see text for details).
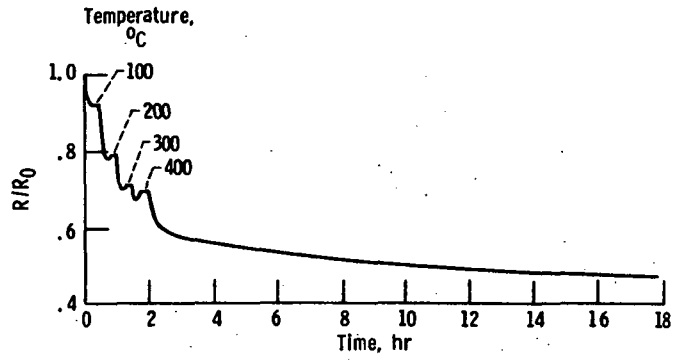
Figure 1. - Resistance ratio as a function of time, for a copper chloride intercalated P-100 fiber.
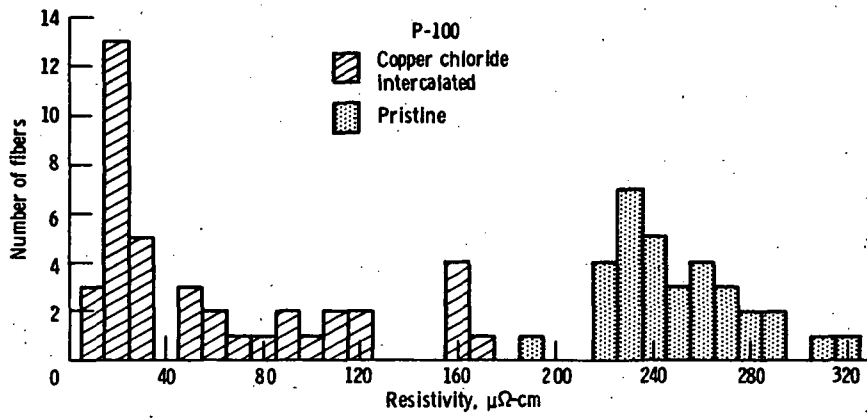
Figure 2. - Histogram showing distribution of pristine (ref. 9) and copper chloride intercalated P-100 fiber resistivities.
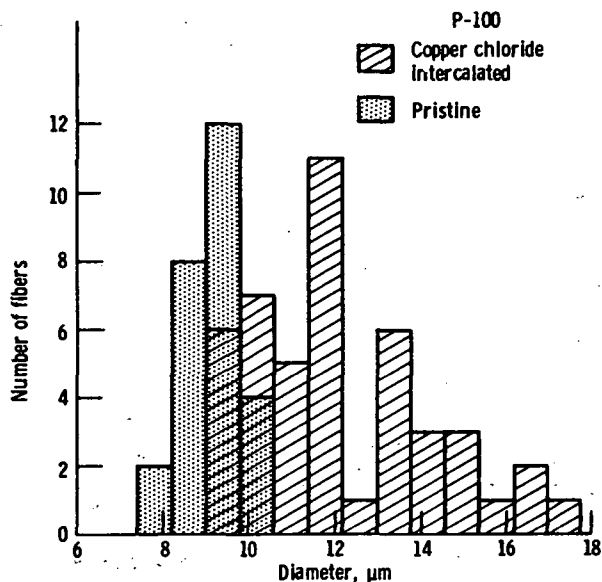
Figure 3. - Histogram showing distribution of pristine (ref. 9) and copper chloride intercalated P-100 fiber diameters.
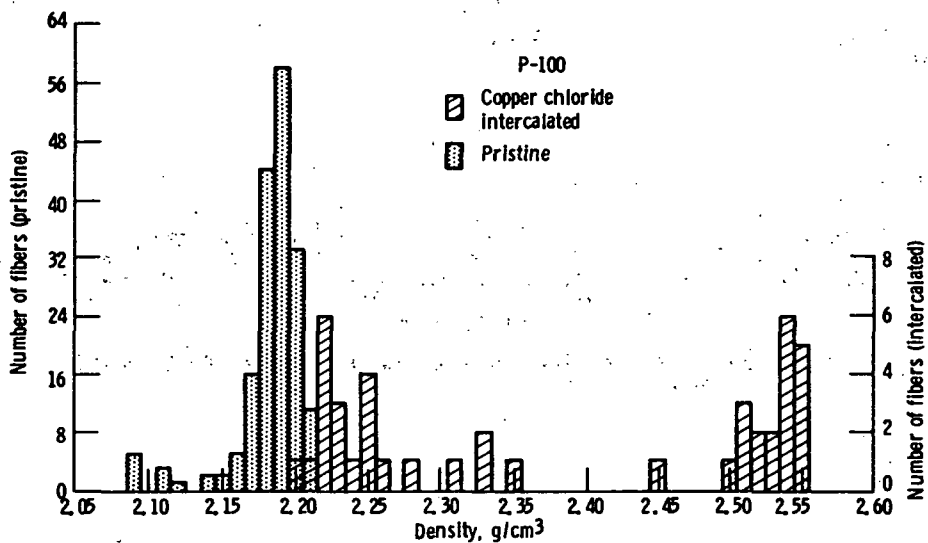

Figure 4. - Histogram showing distribution of pristine and copper chloride intercalated P-100 fiber densities. (The number of pristine fibers tested was about four times the number of the copper chloride intercalated fibers, so the scale for the pristine fibers is increased by a factor of 4.)
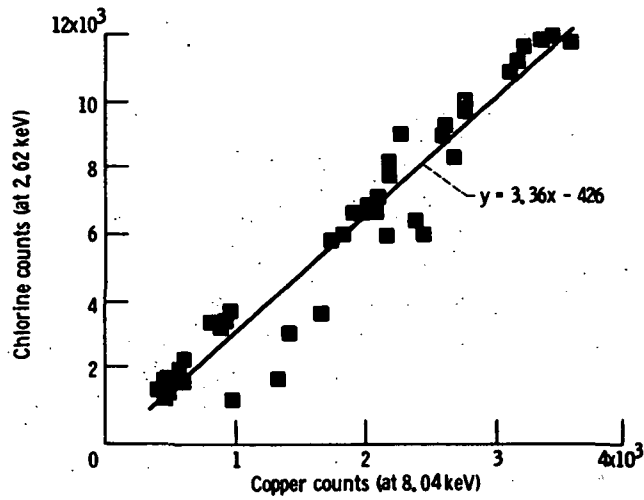
Figure 5. - Copper concentration versus chlorine concentration as determined by energy dispersive spectroscopy.
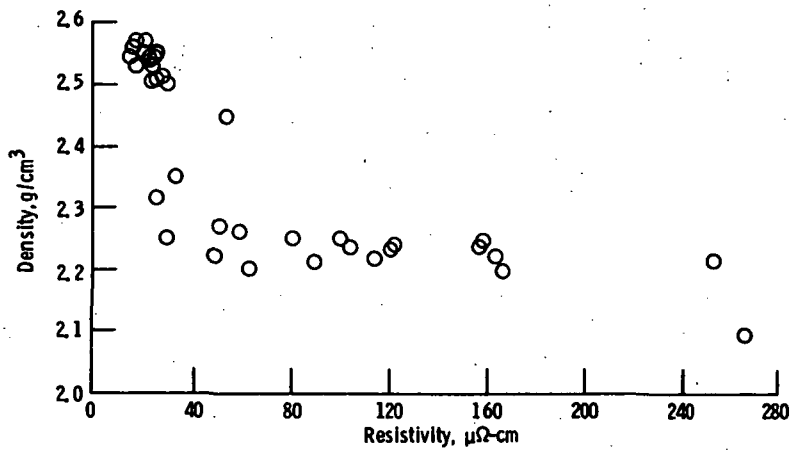

Figure 6. - Resistivity versus density for suite of copper chloride intercalated P-100 fibers.
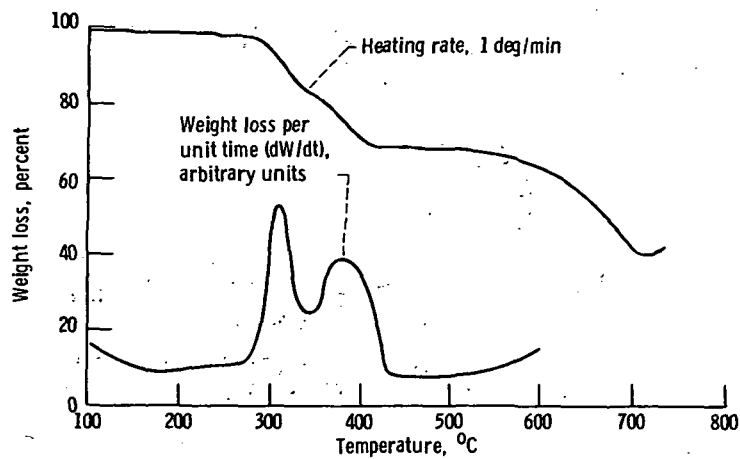
Figure 7. - Decomposition of copper chloride intercalated fibers, as determined by thermogravimetric analysis.

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16. Abstract

In situ resistance measurements were used to follow the intercalation of copper chloride in pitch-based fibers. Subsequent single fiber resistivity measurements revealed a large range of resistivities, from 13 to 160 $\mu\Omega$-cm. Additional density measurements revealed a bimodal distribution of mass densities. The denser fibers had lower resistivities and corresponded to the stage III compound identified by x-ray diffraction. Neither resistivity nor density correlated with diameter. Both energy dispersive spectroscopy and mass density data suggested that excess chlorine resided in the intercalated fiber, resulting in a stoichiometry of $C_{4.9n}CuCl_{2.5}$ (where n is the stage number) for the denser fibers. Finally, thermogravimetric analysis showed a 33 percent loss in mass upon heating to 700 °C. This loss in mass was attributed to loss of both chlorine and carbon.

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