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INVESTIGATION OF STRUCTURE OF GRAPHITE  
FIBERS WITH THE GOAL OF IMPROVING THEIR  
MECHANICAL PROPERTIES

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INVESTIGATION OF STRUCTURE OF GRAPHITE FIBERS  
WITH GOAL OF IMPROVING THEIR MECHANICAL PROPERTIES

by

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The following approaches are recommended to be studied to upgrade the strength of graphite fibers:

- a. Expansion of fibers followed by impregnation of a laminating resin or metal.
- b. Exfoliation of fibers followed by compaction and hardening with a laminating resin or metal.
- c. Examination of exfoliated bulk graphite for possible conversion into high strength reinforcement.
- d. Strength of fibers which are intercalated with nonvolatile salts.

## FOREWORD

The work covered by this report was performed under Project 1T16215AH84; Materials; Plastics for Mobility Items and Systems.

The investigation was performed by S. Goldfein as directed by Emil J. York, Chief, Materials Engineering Division, Laboratory 4000.

The author's appreciation is extended to 1st Lt Patrick O'Brien for his supporting laboratory investigation and Mr. E. J. York, Chief, Materials Engineering Division for his encouragement and support.

## SUMMARY

The objective of this study was to develop data upon which a program could be based to improve the strength of graphite fibers. It was found that graphite fibers are composed of ribbons or sheets and contain many voids and flaws. The ribbons twist and undulate offering many opportunities for them to bend, fold over and cut each other. This condition explains their relatively poor tensile, compressive, shear and impact strengths. Similar conditions exist in "dry" areas in glass fiber reinforced composites and laminates. They are corrected by injection of resin into the area followed by hardening of the resin. Such an approach if used successfully with graphite fibers should strengthen them immeasurably. Exfoliated graphite has not been investigated as a source for high strength and modulus materials.

The following approaches are recommended to be studied to upgrade the strength of graphite fibers:

- a. Expansion of fibers followed by impregnation of a laminating resin or metal.
- b. Exfoliation of fibers followed by compaction and hardening with a laminating resin or metal.
- c. Examination of exfoliated bulk graphite for possible conversion into high strength reinforcement.
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INVESTIGATION OF STRUCTURE OF GRAPHITE FIBERS  
WITH GOAL OF IMPROVING THEIR MECHANICAL PROPERTIES

I. INTRODUCTION

1. Subject. The objective of this study was to collect data upon which a program might be used to improve the quality of graphite fibers.
2. Background. Graphite fibers, by virtue of their low density and ability to be formed with very high elastic moduli, have been studied extensively for use in composites with plastic or metallic matrices. There are a number of serious drawbacks which they possess however, which have prevented them from being utilized to any great extent in USAMERDC equipment.

Foremost of these limitations is price. The long treatment which organic fibers must undergo for conversion to graphite at elevated temperatures while under tension is costly. The higher the modulus the costlier is the end product. Another is the lack of significant advances in increasing the strain to failure of the fibers, particularly for higher modulus fibers.<sup>1</sup> This is another way of saying that the increase in tensile strength has not kept pace with the increase in modulus. In addition, as composites are made with fibers of increasing moduli, interlaminar shear strengths, impact strengths, and abrasion resistances seriously decrease.<sup>2</sup> The compressive strength of composites made with graphite fibers are not comparable with glass fiber reinforced plastics. There are substantial variations in the strength characteristics of carbon fibers.<sup>3</sup>

The most serious mechanical defect which graphite fibers and composites possess from the standpoint of utility in USAMERDC equipment is their relatively low impact strength. It is difficult to design around this in equipment which must sustain rugged treatment. In general for a homologous series of materials

$$S_I = K \frac{(S_t)^2}{E}$$

when  $S_I$  and  $S_t$  are the impact and tensile strengths respectively,  $K$  is a constant and  $E$  is the modulus of elasticity. It is evident that if the modulus is increased fourfold without any increase in the tensile strength, the impact strength will decrease fourfold. This appears to describe the situation with graphite fibers. Their strengths have not increased with a corresponding increase in modulus. When the glass fibers ( $E = 10 \times 10^6$  psi) are replaced in an epoxy composite by graphite fibers ( $E = 40 \times 10^6$  psi) the resulting impact strength is reduced to one-fourth. Since glass and graphite fibers do not belong to the same homologous series,  $K$  is not necessarily the same. Hence graphite fibers and their composites do not necessarily have to have lower moduli than their glass fiber counterparts.

## II INVESTIGATION

3. Procedure. It would appear that since the structure of graphite fiber changes as it becomes more graphitized and its modulus increased, a study of the structure should reveal clues as to what measures might be taken to correct its deficiencies.

A large number of investigations have been undertaken to elucidate the structure

of graphite fibers (1-23). Although this work is not yet complete or for that matter may never be complete, a picture of the structure has emerged which is consistent with all the evidence available.

4. Structure. Graphite fibers are essentially composites of crystallites, voids and organized and unorganized carbon.<sup>3</sup> For commercial production, the two precursor fibers most commonly used are polyacrylonitrile (PAN) and cellulose (rayon). In addition, fibers are available which are made from petroleum pitches. The structures of the precursors for these classes of carbon fibers are folded-chain crystalline, straight chain crystalline, and amorphous, respectively. Each class requires specific and unique processing in order to obtain the desired ultimate properties. Yet, in every case, the final fiber surface microstructures are remarkably similar and appear to be related to the processing rather than to the chemistry of the starting materials.<sup>3</sup>

"The basic building blocks for the carbon fiber microstructure are graphitic crystallites of varying sizes. Small crystallites, highly bridged, of about  $10^{\circ}\text{A}$  on up to about  $40^{\circ}\text{A}$  in width ( $L_c$ ), are characteristic of low and intermediate modulus fibers where the heat treatments are below  $1900$  to  $2000^{\circ}\text{C}$ . As these temperatures are exceeded and high modulus fibers are produced at up to  $2800$ - $2900^{\circ}\text{C}$ , the crystallites become more perfect with little bridging and lower internal surface, grow to an average width of about  $65^{\circ}$  to  $100^{\circ}\text{A}$ , with some as large as  $220^{\circ}\text{A}$ , and are generally aligned along the fiber axis. Thus, on the average, intermediate modulus fiber crystallites are composed of 5 to 12

layer planes. In the fiber cross section, these crystallites show frequent dislocations, a general waviness, folding, and even complete loops in the layer planes. Hot-stretched high modulus fibers also have some 20-30<sup>o</sup>A structure, the presence of which is not yet fully understood. Along with these normal size-variations, it has been found that the surface crystallites are larger than those in the bulk of the fiber.<sup>3</sup>

" The crystalline carbon fiber microstructure develops as the precursor fibers are first stabilized at 200-300<sup>o</sup>C in air and then carbonized and graphitized at the appropriate conditions to give the desired modulus. During this process carbonaceous material is formed and collects as slowly undulating ribbons of turbostratic graphitic layer planes. These may be as large as 60<sup>o</sup>A in width and of widely varying, irregular lateral dimensions. Frequently the ribbons are several thousands of Angstroms in length ( 10<sup>4</sup> to 10<sup>5</sup> A): of the same magnitude as the chain lengths for medium to high molecular weight polymers. The ribbon is further characterized by a combination of straight and curved sections with the straight sections being as long as 321 A. It is the extent of these straight sections which defines the crystallite size and is directly related to the fiber modulus.<sup>3</sup>

" Several such ribbons, running parallel, coalesce to form microfibrils which are wrinkled and have imperfect packing but which do have short-range order in the packing of adjacent stacks of ribbons. These microfibrils may be 100<sup>o</sup>A in diameter and they are aligned parallel to the fiber axis. In intermediate modulus fibers, the various layer planes may be at angles of from 20 to 40<sup>o</sup> to each other; therefore, it is considered probable that some ribbons run from one

microfibril to another, tying them together. In the high modulus fibers the layer planes develop into overlapping and interlinking lattices."

"Coexisting with the crystallites, to form a two-phase microstructure, are voids or pores. These voids are thought to be created at the intersections of mis-oriented crystallites. In the intermediate modulus fibers they are numerous and about as wide as the crystallites. As the crystallite size increases (as the heat treatment temperature increases), pore size increases and the number of pores decreases. Then, in the high modulus fibers they become individual pores, needle-like in shape, with sharp edges, and longer than the crystallites. The volume fraction of voids actually increases with processing temperature: from 0.01-0.03 at 1000°C to 0.18-0.25 at 2000°C to 0.15-0.33 at 2900°C. It decreases, however, if the fiber is hot-stretched. In fibers treated at 2800 to 3000°C such voids can be as much as 200-300 Å in length with cross sections of 15-20 Å and they form an inaccessible porosity. Again, the average void cross section is a function of the heat treatment and is independent of the fiber precursor material. Such voids have variable specific internal surfaces: 50-100 m<sup>2</sup>/cm<sup>3</sup> at 1000°C, 700-900 m<sup>2</sup>/cm<sup>3</sup> at 2000°C, and 200-700 m<sup>2</sup>/cm<sup>3</sup> at 2900°C. Within the microfibril structure the voids take on the same orientation as the crystallites and the orientation is close to that of the straight portions of the ribbon.<sup>3</sup>

"Crystallite interlayer spacings of from 3.36 to 3.63 Å have been reported. By comparison, the spacing in natural graphite is 3.35 Å while that in turbo-static graphite is 3.44 Å. The normal range for cellulose-based fibers is given as from 3.42 to 3.44 Å. In acrylic-based carbon fibers two groups of

spacings have been measured: One from 3.35 to 3.40<sup>o</sup>A for a high modulus, highly graphitic, highly oriented phase and the other from 3.41 to 3.44<sup>o</sup>A for a low modulus unoriented phase. Still other measurements have indicated interlayer spacings ranging from 3.41 up to 3.47<sup>o</sup>A for intermediate to high modulus fibers. Although these reported values are quite variable, it is apparent that interlayer spacings tend to diminish in size with increasing processing temperature. As with crystallite size, the interlayer spacings also vary as one goes from the fiber surface inward, the interior spacings being the greater. For example, Thornel 50 has been reported to have surface interlayer spacings of 3.44<sup>o</sup>A, while in the fiber bulk they are 3.461 A. The same reference gives these measurements as 3.41 A and 3.478 A, respectively, for Morganite I fiber. )

“ The microfibrils, in turn, collect into bundles of nonplanar graphitic layers called fibrils. These fibrils vary in diameter from 250 to 1000<sup>o</sup>A, run parallel to the fiber axis, and seem to have no ends. Alignment between fibrils is not perfect: there is a combination of paralleling and interweaving of fibrils to give a three-dimensional microstructure. As a result, the "c" direction of the crystallites is aligned with the fiber radii and the "a-b" planes with the fiber circumference, thus presenting fiber surfaces primarily of basal planes at an angular spread of about +10<sup>o</sup>. )

“ In addition to the lattice microstructure, there is a gross structure in carbon fibers. Cavities and inclusions, sometimes greater than 0.1  $\mu$  in size, have been noted at a frequency of from 0.03 to 2.3 flaws/mm, having a Poisson's type distribution. These inclusions are seen in all types of fibers and

probably stem from inhomogeneities such as separated copolymer or dirt in the original fiber precursor. Cavities are most frequently seen only in fibers heat treated above 1800°C.

There are evidences that the formation of cavities may be attributed to expanding gases generated by the decomposition of organic inclusions. These cavities are often large enough to cause local increases in the fiber diameter. Also, it is thought that small cracks are associated with these cavities and that the cracks project into the lamellar structure which is seen to surround these cavities. Such flaws are believed to be related to spinning problems. Some cavities are elongated and have substantial length. One type seen at this Laboratory appeared to be continuous holes down the centers of the fibers. These holes are believed to be the result of insufficient oxidation during the stabilization process."

"Surface flaws are also quite prevalent. Most often they are evidenced by the fracture pattern in a fiber cross section. In these cases the fracture will have its origin at the surface flaw. Such flaws can be traced back to the precursor fiber spinning process. The presence of airborne particulate matter which collects on the fiber surface is probably the principal cause of their occurrence."

"Generally the density of carbon fibers ranges from 1.5 to 2.0 g/cm<sup>3</sup> which is much less than that of natural graphite (2.25 g/cm<sup>3</sup>) but approaches that of pyrolytic graphite (2.0-2.1 g/cm<sup>3</sup>). It is also recognized that there is an anisotropic density fluctuation in all fibers. This fluctuation decreases

with increasing processing temperature, as the ribbon stacks become more ordered, and increasing with hot stretching. It may be due to interlayer spacing variation or to layer size variation, or to ribbon irregularities, or to all three."

"The model for carbon fibers having (1) fibrils oriented parallel to the fiber axis, (2) crystallites generally oriented parallel to the fiber surface but with tilt and twist components, and (3) a definite flaw population correlates fairly well with the modulus properties of carbon fibers. In general, the relationship between modulus and crystalline orientation holds for all fibers, and increases in modulus can be related to increases in the average length of the straight portions of the ribbon structure. However, this model describes only the surface layers of the fiber. To explain fiber internal structure, most investigators have assumed a "tree-ring" or "onion-skin" fiber cross-sectional method in which the crystallites are entirely circumferentially oriented. Others suggest a model consisting of a circumferentially oriented outer layer but with a more or less randomly oriented interior."

Knibbs studied the effect on the fiber morphology of varying the time of oxidation during the manufacture of high modulus PAN fibers. For short oxidation times, he found that the crystallites of the final product (when the fiber was viewed in cross section) were oriented approximately isotropically. A moderately long oxidation period gave a thin circumferentially oriented skin and a radially oriented interior. Further increase in the oxidation time gradually converted the radial structure to a circumferential one. Larsen, using Knibb's technique among others, also concluded that the PAN-based fiber is best des-

cribed as having a circumferentially oriented outer layer with a radially oriented interior. For the cellulose-based fibers he concluded that the crystallites are circumferentially oriented on the surface but randomly oriented in the interior. The thickness of this circumferential layer increases as the modulus of the fiber increases."

"As yet the cross-sectional models have not been correlated to any degree with the mechanical or physical properties of fibers. One study did conclude, based on the "tree-ring" model, that the radial strength of the fiber is weak and that this is the reason for the poor interlaminar shear strengths experienced in composites reinforced with untreated high modulus fibers. Thus, there is much more to be done toward elucidating the morphology of various carbon fibers and in correlating morphology with properties."<sup>3</sup>

It is concluded that carbon fibers consist of long undulating and twisting ribbons. "For fibers with a modulus of 6 million psi., there is an almost random orientation of these ribbons with respect to the fiber axis. The thickness of these ribbons is approximately 5 to 6 turbostratic layer planes with a width of probably 20<sup>o</sup>A. The ribbons are highly undulated and twisted to give an intertangled mass. This provides a tight coupling between ribbons, and the fibers are observed to be surface flaw sensitive. Only a small portion of the surface appears to be covered with basal plane, and interfacial bonding to the matrix should be good. Flaw sensitivity should give a high gage length effect with tensile strength, with short gage lengths giving much higher strengths. Combined with good bonding, this should give a high "translation" of fiber strength (measured at 1 inch gage lengths) to the composite.

For fibers with a 40 million modulus, the ribbons are typically about 13 layer planes thick and  $40^{\circ}\text{A}$  wide. The amplitude of the undulation is greater than the wavelength, and about three-quarters of the basal planes are aligned within  $30^{\circ}$  of the fiber axis. Some variation in preferred orientation from the surface of the fiber to the center is observed, with the surface having a higher preferred orientation. There is also some radial preferred orientation with basal planes orienting parallel to the fiber surface. The radial preferred orientation in the center of the fiber depends on the processing, but is lower than the surface. The variation in axial preferred orientation and the radial preferred orientation will give rise to residual stresses upon cool-down. These stresses are compressive axially and circumferentially at the surface, and tensile radially. The combination of radial residual stress with a lowered tangling of the ribbons gives poorer coupling between ribbons. Fractography also reveals rougher fracture surfaces. The lack of coupling of the ribbons and the compressive surface stresses also appear to make the fiber somewhat less flaw sensitive. However, tensile strength gage length effects are still relatively large. The fiber surface is starting to be covered with appreciable amounts of basal plane, but the resin bonding to the surface is still relatively high. The variation in axial preferred orientation will also cause the outside sheath of the fiber to carry more of the load. The interaction of this non-uniform loading with the residual stress has not been determined. The good bonding and high tensile strength at short gage lengths should give good "translation" of 1 inch gage length fiber strength to the composite.

For 50 million psi fiber, the ribbons remain turbostratic and increase to about 20 layers thick and  $70^{\circ}\text{A}$  wide. The undulation amplitude drops to less than the wavelength, and three quarters of basal planes are oriented within  $12^{\circ}$  of the fiber axis. Less tangling is observed between ribbons, and a high radial preferred orientation is observed with the basal planes parallel to the surface. The residual stress which develops from the radial preferred orientation becomes high enough to cause internal fracture, which, with less tangling of the ribbons, gives poor coupling between ribbons. Fiber fracture surfaces become quite rough, and the tensile strength of the fiber often decreases. Flaw sensitivity and tensile strength gage length effects decrease. The surface of the fiber is predominately a faulted basal plane, and interfacial bond strength, without etching, will be low. Translation of fiber tensile strength, for surface treated specimens, to composite properties will be reduced as the strength at short gage length does not increase as rapidly as for flaw sensitive fibers of lower modulus.

Finally, for fibers of 100 million modulus, the ribbons are about 30 layers thick and  $90^{\circ}\text{A}$  wide, but ribbon fusion is evident in some samples. For one PAN sample, three dimensional ordering was observed, together with a preferred orientation of a-axes. The orientation was that expected if the orientation of carbon backbone of the PAN polymer was retained throughout processing. The ribbons are observed to have almost zero amplitude and are essentially parallel to the fiber axis. Very little tangling is also observed. Strong radial preferred orientation occurs, and the resulting residual stresses must give very poor coupling between ribbons. Fracture surfaces are extremely jagged.

The fiber surface consists of extremely smooth basal planes which would give poor bonding.

The strong similarities among carbon fibers of the same modulus, but from different precursors, were more noteworthy than the differences. Axial structures appeared very similar with catalytically processed fibers having somewhat larger  $l_c$  values; differences were more apparent in radial structures. In general, however, higher radial preferred orientations were associated with higher axial preferred orientations. Samples which appear to be strain-annealed, i.e., fibers which have been strained at high temperatures by an applied stress, show a somewhat poorer development of radial structure as might be expected. In this case, the applied stress causes the basal planes to align parallel to the applied stress.

Finally, improved carbon fibers are conceivably possible if axial alignment of ribbons can be accomplished with a minimum of radial alignment. This would minimize decoupling of ribbons.

Past work has shown that significant amounts of residual stress may be present in carbon fibers, and this will seriously affect flaw sensitivity. Secondly, the structural features responsible for residual stress make uniform loading of each fiber difficult, if not impossible. On a finer scale, little is known about the mechanisms of stress transfer among the rippled ribbon-shaped fibrils which compose all carbon fibers."<sup>1</sup>

Figure 1 shows the laminar structure of graphite. The ribbon or sheet like structures of graphite fibers are shown in Figures 2 and 3.<sup>1</sup> Figures 4-7

depict other possible structures using the same data.<sup>23</sup> All of the postulated structures have in common a ribbon or sheet-like structure containing irregularities, voids and discontinuities. The increase in strength and modulus due to hot-stretching of the fiber is shown in Figure 8. This can only result from the increased alignment of the ribbons.<sup>23</sup> When a plot of the density of various fibers versus their moduli is drawn (Figure 9) it is evident that a higher modulus resulted from a greater conversion from unorganized carbon to ordered graphite. The concentration of voids decreases with an increase in modulus.

## 5. Alteration of Structure.

a. Reverse sputtering. This process was used to etch away the graphite fiber to elucidate the structure.<sup>1</sup> It peeled away the graphite layer by layer.

b. Plasma etch technique. Plasma etching was used<sup>2</sup> to etch away both resin and fiber in composites. Examination by a scanning electron microscope later was used to view the fibrous structure.

c. Chemical treatment.

(1) Liquid ammonia. Treatment with liquid ammonia<sup>24</sup> has been used to expand the distance between the layers of graphite to such an extent that atoms and ions have been able to be placed between them. This process has been used to make intercalation compounds of graphite and the alkali metals.<sup>25</sup> (Figure 10).

(2) Halogens. It is well established that bulk graphite crystals exhibit exfoliation and can increase in size by factors as large as 10 to 100.<sup>26</sup>

Experiments have been shown recently<sup>27</sup> that graphite fibers can be affected to such an extent that they become plasticized and undergo a similar expansion. This plasticization is lost when the chemical agent (liquid bromine) is removed by heat. The fiber then returns to its initial form but new shape e.g. spiral instead of straight. (Figure 11).

(3) Salts, Anhydrous ferric chloride has been found to react with graphite to form interlaminary compounds.<sup>28</sup>

(4) Ternary compound. A graphite-aluminum bromide-bromine ternary system interlaminary compound has been found.<sup>29</sup>

(5) Calcium. Molten calcium has been found to react with graphite to form various compounds.<sup>30</sup>

(6) Carbon Fiber Surface Treatments. (Oxidation Treatment) Common liquid oxidation treatments are nitric acid boil, electrolytic oxidation with sodium hydroxide, and sodium hypochlorite.<sup>31</sup>

(7) Macroscopic flaws. The effect of surface flaws on ultimate tensile strength was investigated by etching away the fiber surface (Figure 12.)<sup>23</sup> Surface flaws reduce the ultimate tensile strength of fibers heat treated below 1250C but after treatment at higher temperatures, there is no correlation between surface flaws and strength. Considerable work has been done on fibers heat treated to 1000 C, and in the absence of surface flaws, all failures examined initiated at internal flaws.

### III DISCUSSION

6. Discussion. There appears to be general agreement among the many investigators regarding the crystallite ribbon or sheet-like structure of graphite fibers. There are a large number of techniques which have been used in the past to alter the structures. Intercalation compounds have been formed and ions and metals have been placed between the planes constituting the graphite. In the bulk, graphite defoliates with an expansion ratio of 10-100 times the original volume. In the fibrous form, the treatment with HF followed by bromine plasticizes the fiber to such an extent that it expands and can be flexed with impunity. (Figure 11) This process offers promise for permitting familiar textile operations to be carried out on them without mechanical damage. In the plasticized state, the fiber yarns are appreciably more flexible than in their unplasticized state, and the yarns can be made to take shapes which would cause the unplasticized material to break. On removal of the bromine, the yarn will retain a portion of the shape imparted to them.

It is clear that the halogen treatment is sufficiently versatile so as to separate the layers to any extent in bulk and in the fibrous form to push them so far apart that the interlaminar forces are extremely weakened. Under these conditions, catalyzed laminating resins such as epoxy and polyesters should be able to position themselves between the layers. After setting of the resin a composite could be formed of the resin and graphite fibrils or layers. This could greatly strengthen the fibers. Some type of bonding must occur between the graphite layers and intercalated materials.<sup>25</sup> Salts such as  $\text{FeCl}_3$  or  $\text{AlBr}_3$ , being non-volatile, might strengthen the structure in a similar fashion

to cross-linking agents with thermosetting resins such as polyester and epoxies.

Defoliation of bulk graphite should be examined as a possible low cost method of forming fibers.

Since carbon fibers are microporous,<sup>14</sup> this condition should be exploited as a possible means of penetration of the fiber by laminating resins without the halogen treatment.

Processes such as reverse electroplating in which the anode is cleaned by reversing the current, should be studied. This might present alternate methods of peeling off the skin and allowing entry by resins or metals by opening up hitherto inaccessible voids.

Techniques such as ultrasonics should be studied as thermal mechanical approaches to altering the structure of the fibers.

#### IV CONCLUSIONS AND RECOMMENDATIONS

7. Conclusions. It is concluded that graphite fibers are composed of ribbons or sheets and contain many voids and flaws. The ribbons twist and undulate offering many opportunities for them to bend, fold over and cut each other. This condition explains their relatively poor tensile, compressive, shear and impact strengths. Similar conditions exist in "dry" areas in glass fiber reinforced composites and laminates. They are corrected by injection of resin into the area followed by hardening of the resin. Such an approach, if used usccessfully with graphite fibers, should strengthen them immeasurably.

Exfoliated graphite has not been investigated as a source for high strength and modulus materials. This should be studied. Destruction of fibers by exfoliation to obtain a source of high strength ribbons for laminating purposes should also be investigated.

8. Recommendations. It is recommended that the following approaches should be studied to upgrade the strength of graphite fibers:

a. Expansion of fibers followed by impregnation of a laminating resin or metal.

b. Exfoliation of fibers followed by compaction and hardening with a laminating resin or metal.

c. Examination of exfoliated bulk graphite for possible conversion into high strength reinforcement.

d. Determination of strength of fibers which are intercalated with non-volatile materials.

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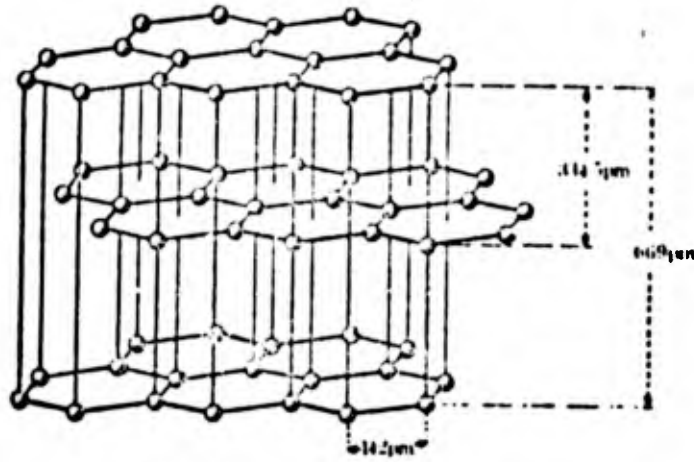
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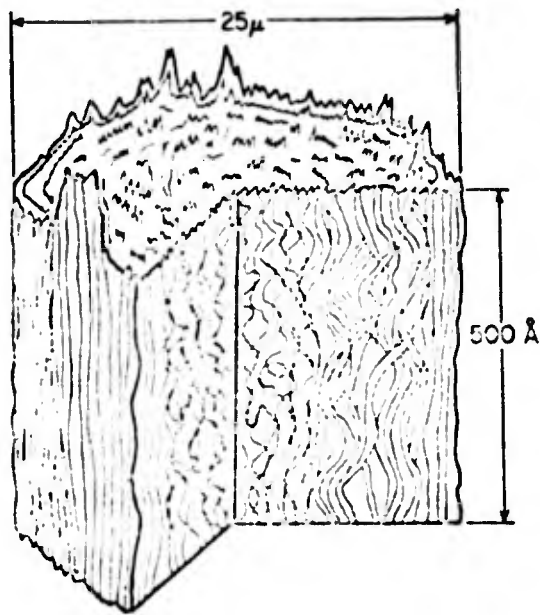
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THE STRUCTURE OF GRAPHITE

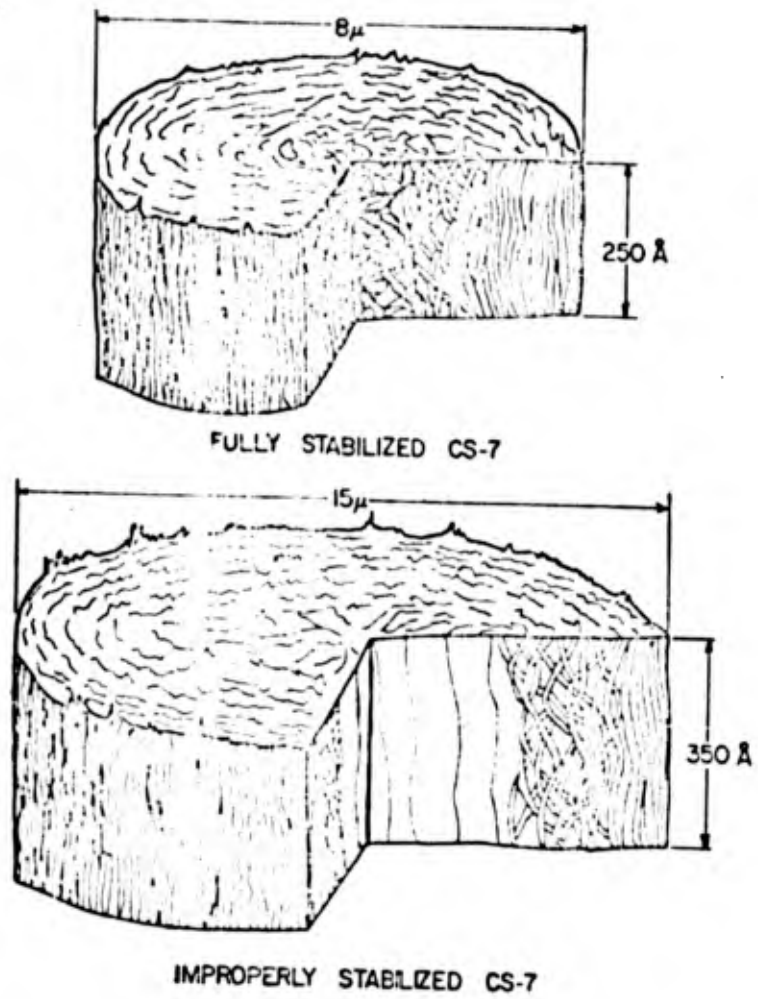
FIGURE 1



GE. Monofilament

Three-Dimensional Structural Model

FIGURE 2



Three-Dimensional Structural Model, CS-7

FIGURE 3

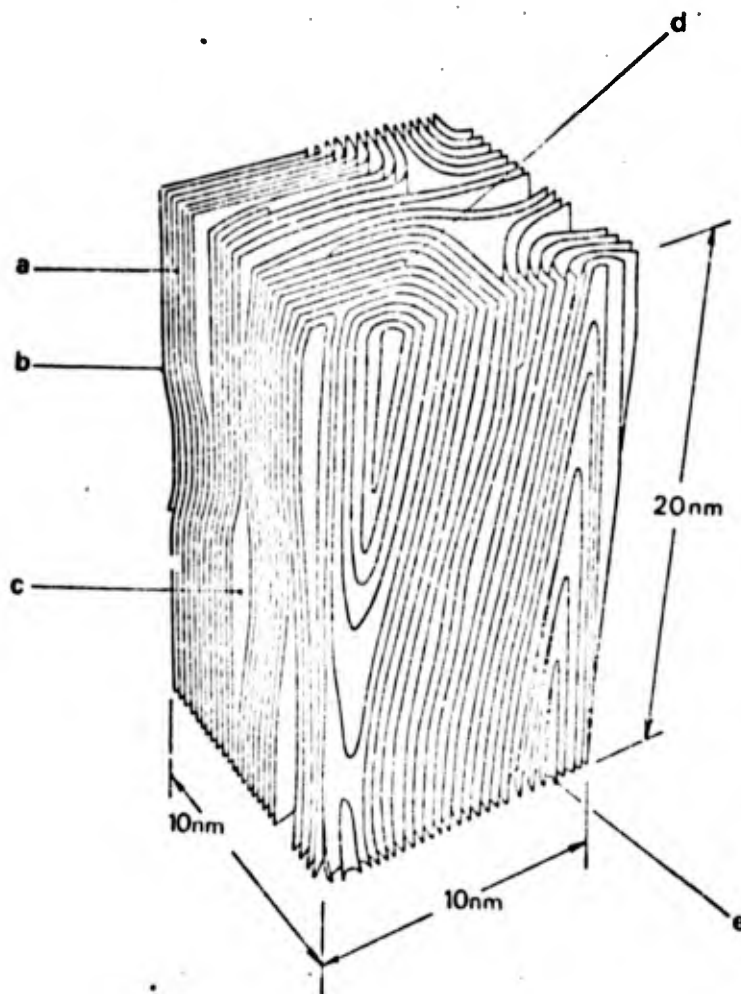


FIGURE 4

**A THREE DIMENSIONAL MODEL OF CARBON FIBRE STRUCTURES**

- Apparent features are:
- (a) Planar areas possibly containing AB nearest neighbour ordering
  - (b) Boundary
  - (c) Void
  - (d) Edge dislocation
  - (e) Bending about two axes (Gillin 40)

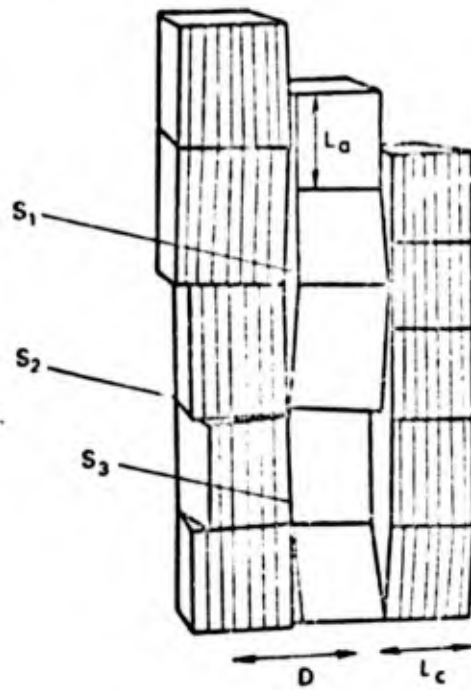
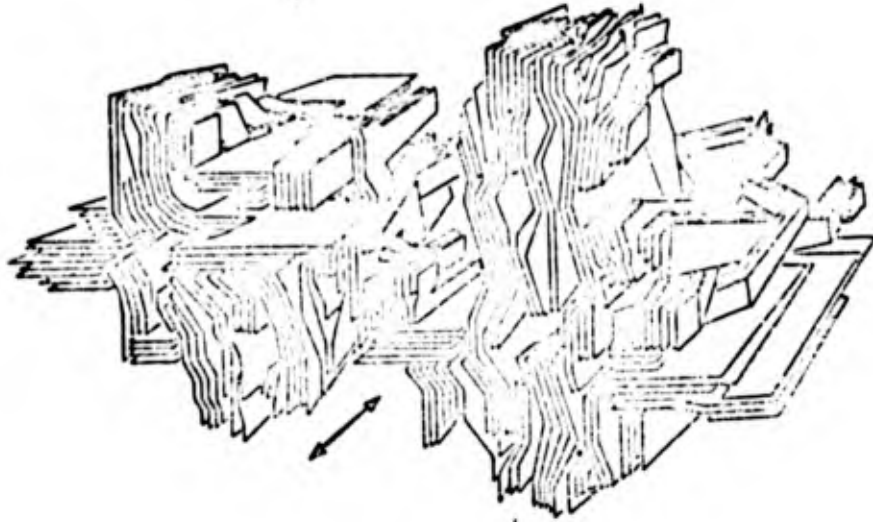


FIGURE 5

SCHEMATIC, IDEALIZED DIAGRAM SUMMARIZING X-RAY DIFFRACTION EVIDENCE

$S_1$  Void;  $S_2$  subgrain twist boundary;  $S_3$  inter crystalline boundary



**FIGURE 6**

**AN ALTERNATIVE THREE-DIMENSIONAL MODEL OF THE CARBON FIBRE STRUCTURE**

**The arrow indicates the fibre axis**

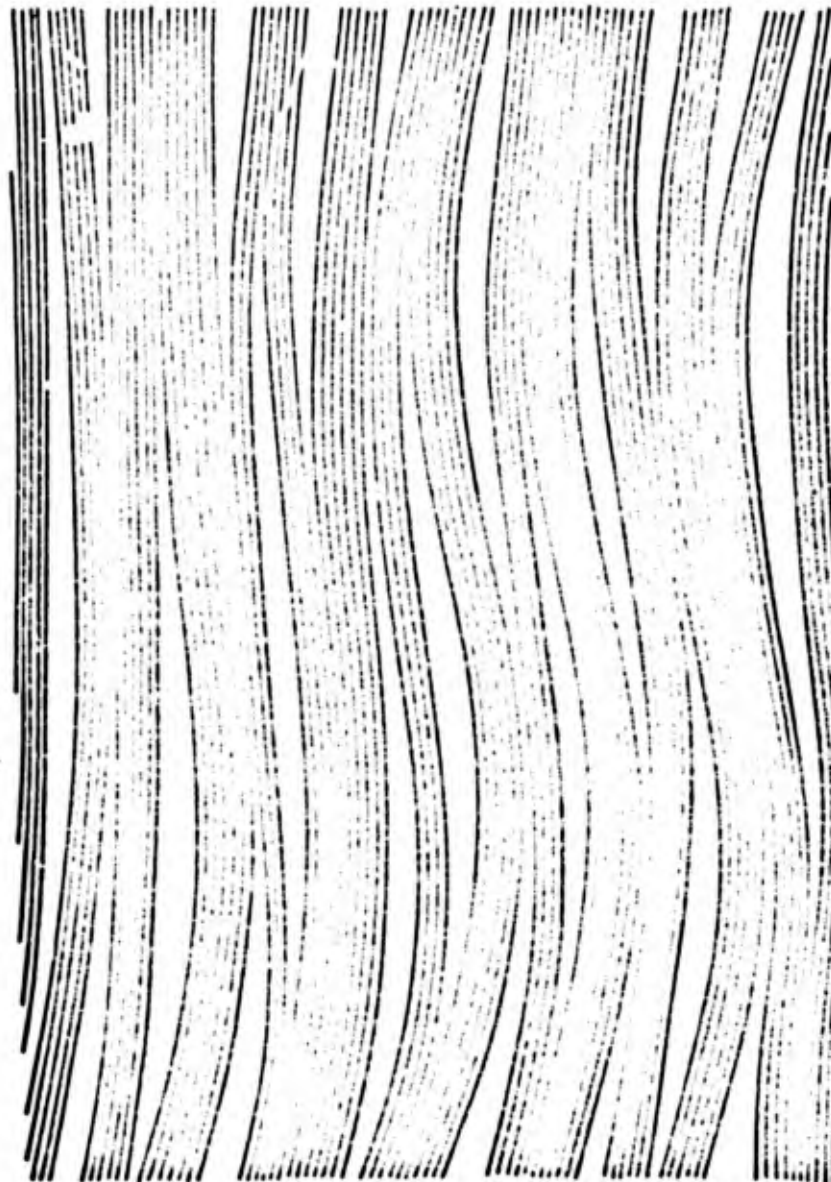


FIGURE 7

SCHEMATIC REPRESENTATION OF THE RIBBON STRUCTURE OF CARBON FIBRE

Stack size  $L$ , apparent layer size parallel to the fibre axis  $l$

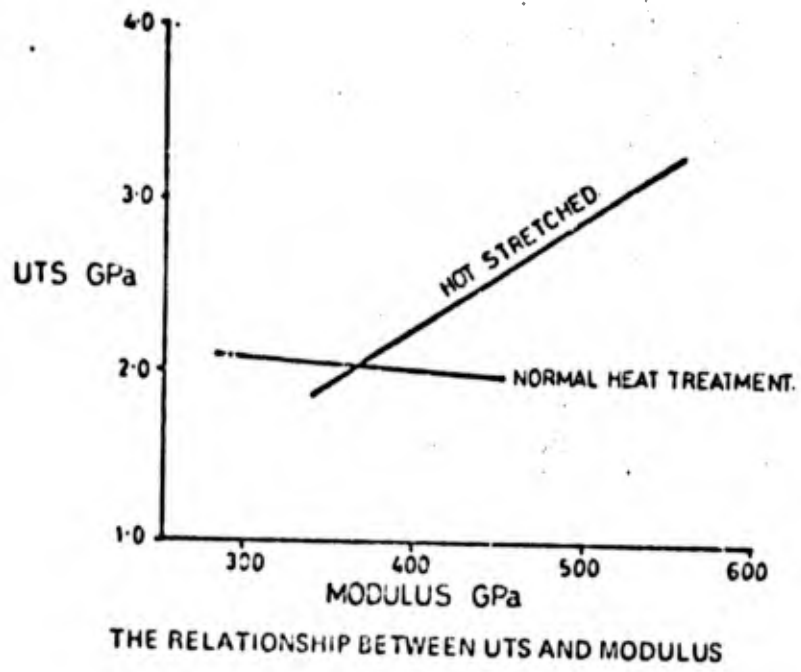
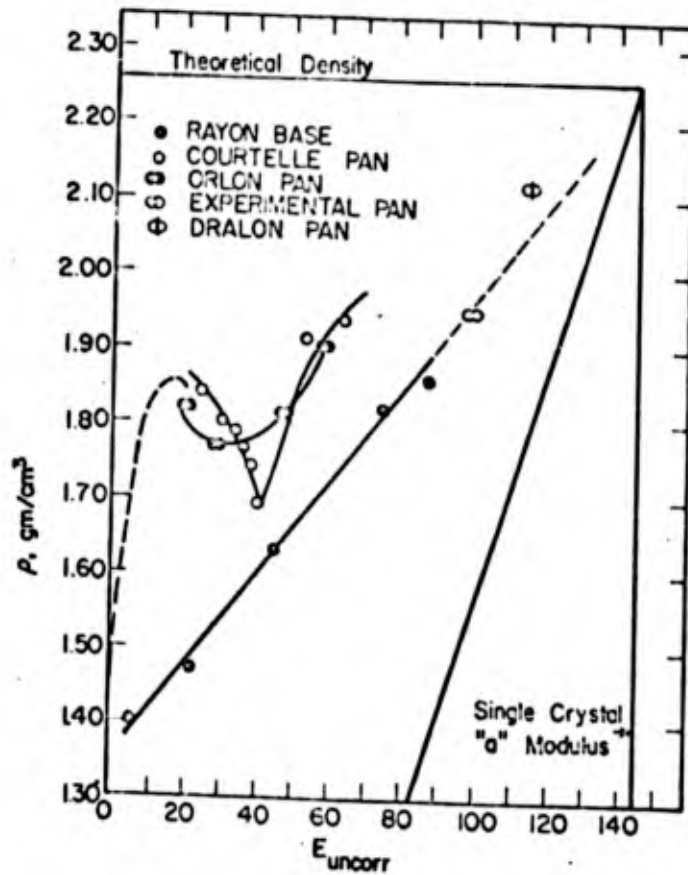


FIGURE 8



Modulus vs. Density of a Wide Range of Carbon Fiber

FIGURE 9

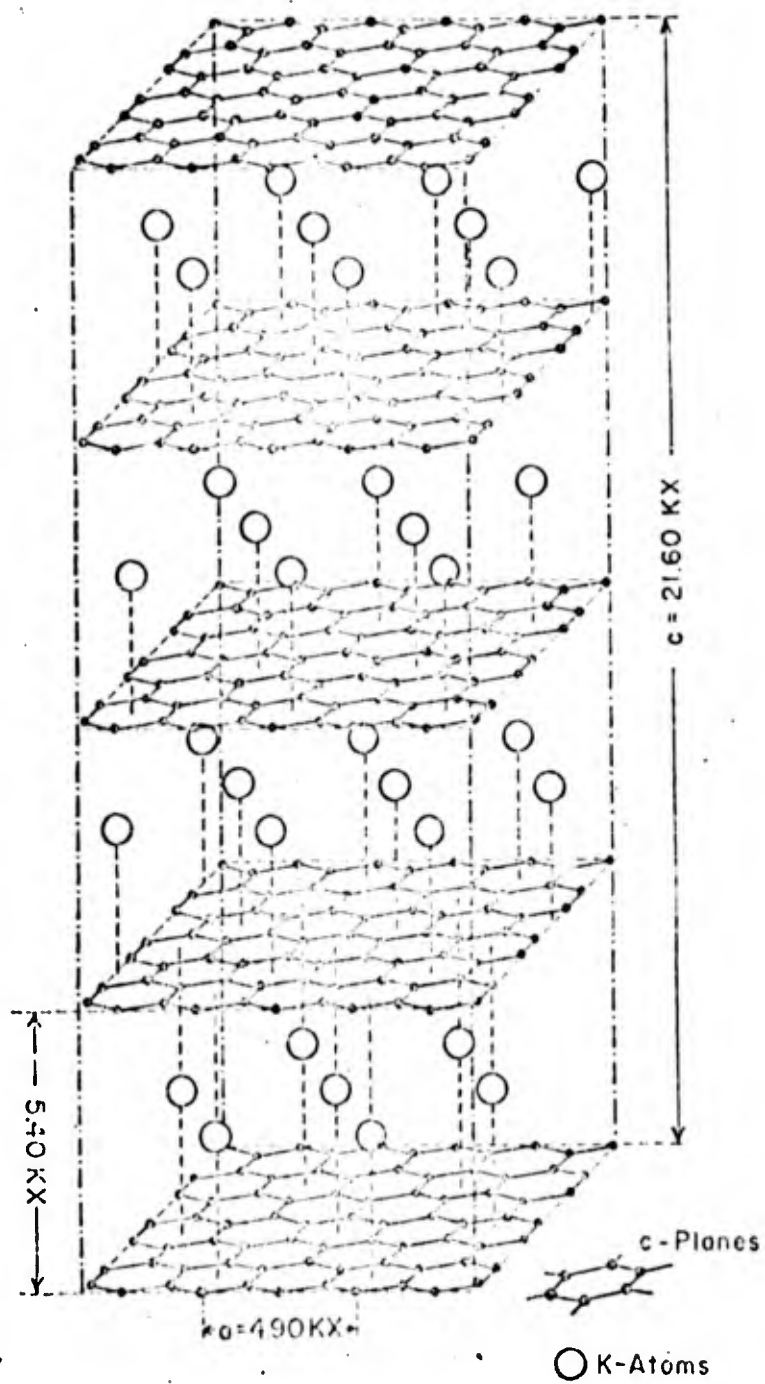
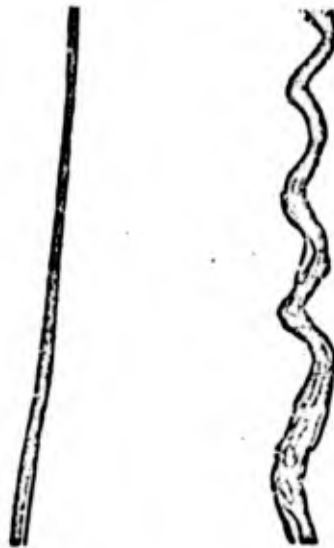


Figure 10 Graphite intercalation compound (Rudorf, W. U. of Tübingen  
Graphite intercalation compounds, Adv in Inorg. Chem. and  
Radiochem. 1959)

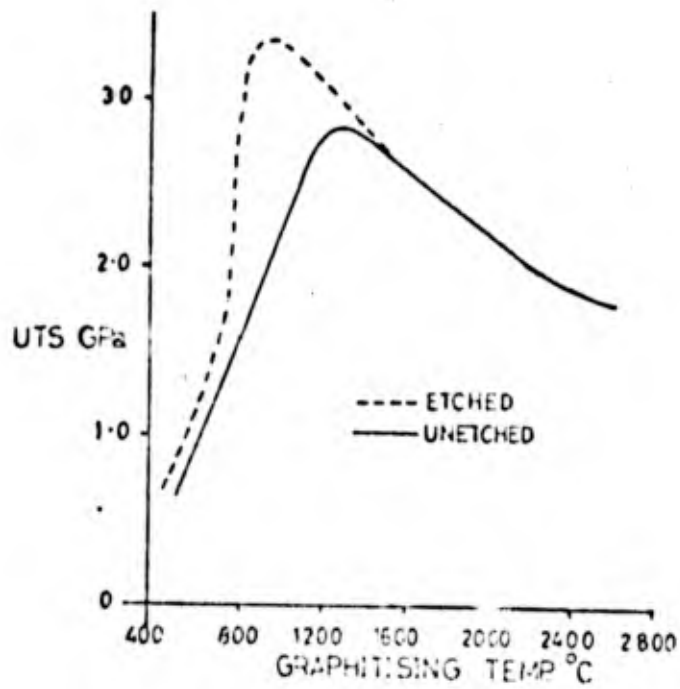


Left: untreated Thornel 390 fiber.  
Right: bromine treated Thornel 390 fiber.



Bromine treated Thornel 390 fiber  
wound on a Teflon mandrel. Note the sharp bends.

FIGURE 11 Plasticization effect on graphite fibers.



THE EFFECT OF SURFACE ETCHING ON UTS OF CARBON FIBRES WITH DIFFERENT HEAT TREATMENTS

FIGURE 12