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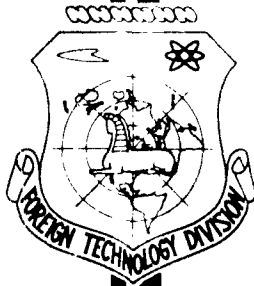
FIBROUS CARBON

By

V. Yu. Nikolayev and V. I. Kasatochkin

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FIBROUS CARBON

V. Yu. Nikolayev and V. I. Kasatochkin .

During the slow pyrolysis of carbon-containing gases, elastic fibrous forms of carbon and graphitic fibers form on the walls of the reaction spaces. Fibrous carbon is very promising as a heat-insulating and filtering material in corrosive media. In an inert and reduction atmosphere it can be used up to a temperature of 2760° [1].

One of the ways of using carbon fiber is as a filler for a large number of plastic materials and elastomers, for example rubber. Mats made out of carbon fiber possess great heat-resistance and small bulk weight and consequently can serve as convenient insulating materials which protect against the effect of high temperatures. Rigid carbon fiber, apparently, can also be used in various areas of electrical engineering.

The method of obtaining fibrous material is by passing a gas-forming hydrocarbon, preferably diluted, through the cracking zone at a temperature of 1150-1450° for not less than 0.4 seconds and then precipitating the carbon fibers on one or several inert surfaces located in the cracking zone. By adding to the hydrocarbon small amounts (0.3 - 4%) of hydrogen sulfide and by diluting the initial

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hydrocarbons with hydrogen, the yield of carbon fiber rapidly increases and reaches 30% with respect to the carbon contained in the initial gas. For example, when using a hydrogen and methane mixture, the best results are obtained at those hydrocarbon concentrations at which the carbon content in a liter of diluted gas at 300° equals 0.05-0.02 g. The use of hydrogen sulfide or substances which form it is recommended in such a concentration that the weight of the H₂S present or being formed constitutes more than 2% of the weight of the carbon contained in the hydrocarbon.

The carbon fiber is produced in various forms depending on the temperature, the configuration of the gas flow, its velocity while flowing past the collecting surface, the number of catalysts, and other factors. It is possible to obtain long single threads from the carbon, and also powder-like particles composed of very small threads. Carbon is also precipitated in the form of "whiskers" from parallel fibers, in the form of a flexible felt-like material (so called mats) from interwoven threads, and in the form of a felt-like material, whose flexibility can gradually be decreased until finally rigid solid particles of carbon of different bulk weight are obtained. The diameter of the threads varies from a fraction of a micron to 100μ, and the length varies in relation to the size, exceeding the diameter several times over and amounting to as much as several centimeters.

X-ray photographs of the carbon material confirm that the threads consist of carbon oriented to a certain degree and partially graphitized [2].

The formation of a fibrous form of carbon was observed a relatively long time ago. As early as 1890 [3] it was reported that when cyanogen is passed through a porcelain pipe containing powdered cryolite heated to cherry redness, thin fibers were obtained as a

result of the decomposition. The fibers had a certain elasticity and contaminated the paper.

In 1903 [4] it was learned that carbon wool consisted mainly of thin cylindrical threads with random interweaving. The thread length reached 5 cm and the diameter varied within 0.03-0.15 mm, the thinnest thread being 0.002 mm.

Intensive study of this process began comparatively recently. Fibrous forms of carbon were obtained by cracking methane diluted with nitrogen and by cracking carbon monoxide with hydrogen on an iron surface at 1000° [5]. The carbon precipitated in the form of fibers more or less parallel to each other. An X-ray analysis of the structure showed that the carbon is present in the form of the common amorphous variety (the space between the carbon lattices is approximately 3.5 Å, the carbon axis being perpendicular to the fiber axis, i.e., the hexagonal base planes are more or less parallel to a fiber axis with a maximum declination of approximately 30°). As far as we know, this is the first example of fibrous instruction from lamellar units.

In the pyrolysis of propane for the purpose of investigating lustrous carbon produced on fused quartz glass at 930° [6], at the point where the propane enters the hot zone of the reaction tube, gray-lustrous needles 8 mm in length and with a diameter of 0.1 mm precipitated on the lustrous carbon flakes. An X-ray diffraction study revealed that the needle crystals were identical in size with the flakes of lustrous carbon. The base planes of the "crystallites" of lustrous carbon lie parallel to the film surface, while the base planes of the needles lie parallel to the needle axis.

Later [7] three types of pyrolytic carbon, including fibrous carbon, obtained at temperatures of 800-1300° on fused quartz were

investigated. All of these types are similar in "crystallite" dimensions to carbon obtained by charring (carbonization). The size of the "crystallites" increases with an increase in the precipitation temperature. It was noted that these types of carbon resemble graphite with respect to their capability of reacting with gaseous oxygen.

The structure of carbon formed by the decomposition of carbon monoxide on an iron catalyst was reported in 1952 by L. V. Radushkevich and V. M. Luk'yanovich [8]. They reported that carbon particles formed at a temperature of 600° had an elongated thread-like form. It was postulated that the thread-like nuclei, obviously consisting of iron carbides, were formed originally and that they grew transversely by means of carbon precipitation. The presence of intertwisted threads was noted. It was reported that similar fibers also grew on particles of thermal carbon black at a temperature of 1050° and in an atmosphere of CH₄, C₆H₆, or cyclohexane [9]. Also published were electron microphotographs of fibrous carbon formed by the interaction of carbon monoxide and iron oxide in brick work [10]. The fibers were produced at a temperature of 450° on various forms of iron oxides. An x-ray diffraction study revealed the presence of "amorphous" carbon. It was postulated that the catalyst of the reaction is either iron or an iron carbide formed on the oxide surface as a nucleus which produced the growth of fibrous carbon. The threads from the conglomerations of nuclei become twisted and, very likely, grow together into the form of the characteristic fiber. It was assumed that after precipitation began particles of the catalyst remained on the growing ends of the threads.

An investigation of the process of catalytic decomposition of carbon monoxide on crystals of copper, nickel, cobalt, silver, iron, chromium, molybdenum, and on electrolytic precipitants of palladium and rhodium

[11] revealed that one of the products of the reaction on iron, nickel, and cobalt at 500° was carbon. The other metals did not possess sufficient catalytic activity.

The structure of carbon precipitated from carbon monoxide on iron, cobalt and nickel at 390° was investigated with the aid of an electron microscope [12]. These precipitations were fibers with a diameter of 0.01-0.2 μ . The carbon fibers on nickel apparently consist almost entirely of little tubes or, less likely, of nonmeshed fibers. The precipitations on cobalt were solid fibers, little tubes, and twin fibers. Most of these fibers contain a dense nucleus of approximately the same diameter as the fiber in which they are located. The nuclei are located over approximately half the length of the corresponding fibers. The nuclei in precipitations formed on iron are discs, while those on cobalt are octahedrons. The nucleus in the precipitations on nickel apparently has a crystalline surface, but it was not possible to identify it. X-ray diffraction analyses of the powder shows that these nuclei consist of nickel with a face-centered cubic crystalline lattice and (or) nickel carbide with a tightly packed hexagonal crystalline lattice.

Ye. Ya. Rabinovich and A. V. Rodionov [13] investigated the decomposition of hydrocarbons in the temperature range 800-1250° on various surfaces. In this case the authors give the following conclusions:

- "1. Dispersed fibrous carbon formed by the pyrolysis of hydrocarbons is distinct from carbon black and lustrous carbon and is a variety of carbon which forms only on surfaces having specific catalytic properties.
2. The best catalytic action for the forming of fibrous carbon from hydrocarbons is possessed by metals of the iron subgroup. Quartz

is a catalyst only at temperatures higher than 1000°, if its surface layer is broken down by prolonged heating. Platinum has an insignificant catalytic action, while copper, porcelain, and impurity-free carbon do not catalyze this process at all.

3. Carbon threads contain in their structure dense inclusions analogous to the metal and metal-carbide nuclei present in carbon threads from carbon monoxide. The structure of the carbon threads from hydrocarbons and also the shape and number of dense inclusions in them depend on the nature of the metals which cause them to begin to form.

4. The intensive growth of dispersed fibrous carbon on the surface of metals of the iron subgroup is observed during the decomposition of mixtures of benzene vapor and nitrogen in the temperature range 830-930° and during the decomposition of methane in the temperature range 925-1000°.

5. The forming of carbon threads on an active surface is observed only when the reaction zone contains a high concentration of unstable intermediate products of decomposition of the hydrocarbons. In this respect the mechanics of the formation of dispersed fibrous carbon differ fundamentally from the mechanics of the formation of a carbon surface during thermal decomposition of hydrocarbons."

The structure of fibrous carbon obtained by various methods in recent years has been studied in the greatest detail [14-16]. The first article [14] dealt with the graphitic "whiskers" produced on the surface of a carbon fiber heated by an electrical current to a temperature of 2000-2500° in a methane atmosphere diluted by hydrogen to prevent carbon black from forming. In the very pure fibrous-structured graphite obtained, the graphitic planes are parallel to the fiber axis.

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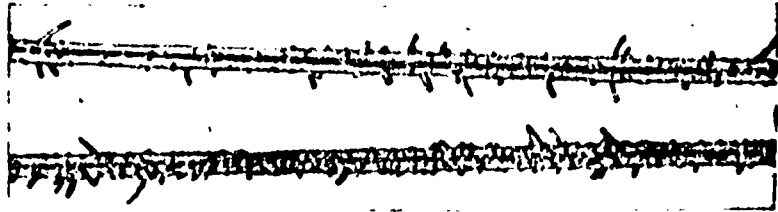


Fig. 1. Precipitation of graphite on a carbon fiber heated to 2000°C in acetylene.

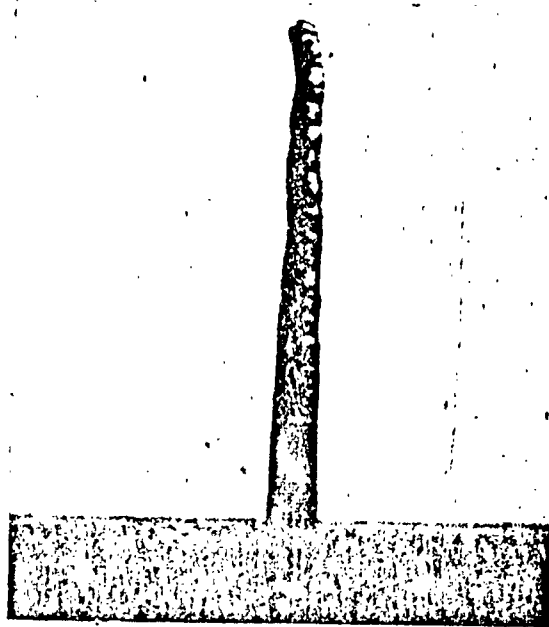


Fig. 2. Fibrous growth of graphite on a carbon fiber heated to 2000°C in acetylene.

For the study of the mechanics of the formation of graphite by precipitation of hydrocarbons the authors also used ethane and acetylene. Graphite formed from acetylene with the latter under a pressure of 1.3 mm Hg and the fiber temperature being 2000°C,

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precipitated in the form shown in Fig. 1.

A significant number of crystals grew more or less perpendicular to the axis of the original fiber. Figure 2 shows a particularly well-developed "whisker" (length 1.3 mm, thickness 0.1 mm) grown on a basic fiber with a diameter of 0.29 mm in 1 minute. X-ray dispersion photos of the basic fiber and of the "whisker" reveal that the "whisker" has the same fibrous structure as the basic fiber, the graphitic base planes being parallel to the fiber axis. Therefore the base planes in the "whisker" are oriented perpendicularly to the base planes of the basic fiber.

In Fig. 3 are curves showing the intensity of an X-ray reflected from the planes (002) and (100) as a function of the angle between the fiber axis and the normal to the surface formed by the original and the reflected rays.

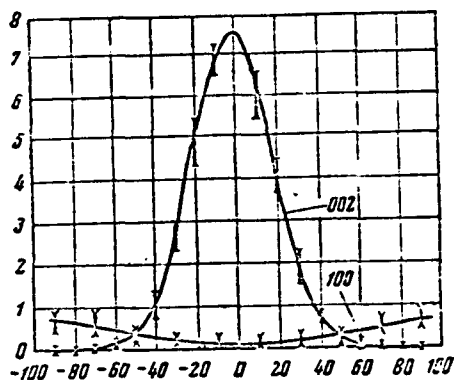


Fig. 3. The dependence of the intensity of X-rays reflected by planes 002 and 100 on the angle between the fiber axis and the normal to the surface formed by the original and the reflected rays.

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A fiber and a "whisker" were mounted in lucite and ground to the center of their thickness. In Figs. 4 and 5 are photos of micro-sections of the base and the top of a "whisker" in polarized light. The significant difference between the intensities of the light reflected from the "whisker" and the basic fiber confirm the results of X-ray diffraction dispersion that the orientation of the planes in the "whisker" is perpendicular to the orientation in the fiber. From the base to the top, the "whisker" is generally hollow and has the same cavity diameter; this indicates that it must grow either from the base or from the top. If a significant amount of carbon precipitates along its sides during the growing process, then a conical form is produced, particularly in those cases where the base is heated, and a temperature gradient related to radiational losses appears along the "whisker". The well-rounded smooth external appearance of the tip, the continuity of the cavity and of the parallel surface, and the continuous change in the orientation at the end of the "whisker", which can be seen by turning it with respect to the plane of polarization of the light, permits us to assume that the "whisker" grows from its base.

Graphite formed from precipitation of hydrocarbons has a very smooth surface. The products of the original decomposition of hydrocarbons move rather freely over the smooth graphitic planes forming these surfaces. This freedom of movement is very likely due to the fact that the forces perpendicular to the graphitic planes are comparatively weak van der Waals forces. Atoms build up into a lattice when they reach the edges of the planes, where they can be held by strong chemical bonds. Graphitic crystals obtained by the decomposition of hydrocarbons will therefore be more likely to grow by the expansion of the planes in a transverse direction than by the formation

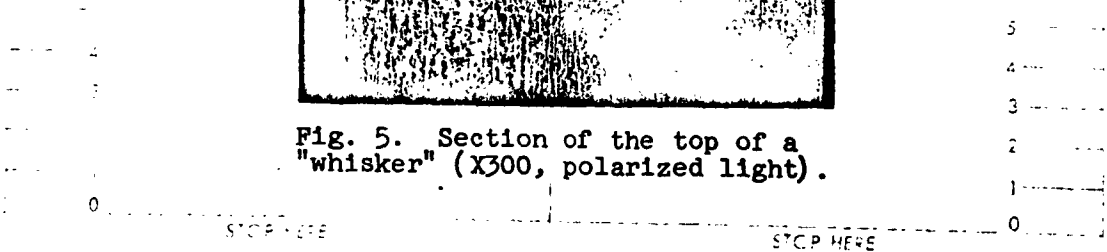
of nuclei of new planes, which is confirmed by the fibrous structure.



Fig. 4. Section of the base of a "whisker" (X300, polarized light).



Fig. 5. Section of the top of a "whisker" (X300, polarized light).



Meyer assumed that "whiskers" occur as a result of the forming of a boundary with a large angle between the "disruption" (i.e., a particle of carbon black precipitated out of the gaseous phase) and the graphite layer lying below. Carbon atoms obtained by the dissociation of the gas, since they possess great surface mobility, can be captured on a line where the border emerges onto the surface and diffuse into points where they can be combined into a lattice.

The high-energy boundary, which is the cause of the growth of a "whisker" according to this model, was very likely created by the growth of a disruption propagating approximately perpendicular to the fiber surface, as shown in Fig. 6 a. This disruption, was covered by the graphite planes, as shown in the figure. The rival covering of the undisturbed layer parallel to the fiber axis causes the formation of a 45° boundary line between the normal and the "disrupted" growth.

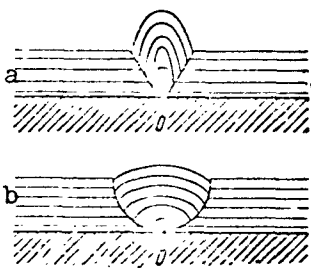


Fig. 6. Scheme of the formation of "breakage" nuclei. a) "whisker" nucleus with a high-energy boundary; b) defect with a low-energy boundary.

The disruption of the growth of a covering, the basic development of which occurs parallel to the fiber surface, leads to a parabolic boundary between the normal and the "disrupted" growth of a carbon

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surface, as shown by the scheme in Fig. 6b. The angle between the graphite planes of the basic covering and the site of the "disruption", decreases with an increase in the "Disruption" thus excluding the growth mechanism suggested for a "whisker". "Disruptions" of this type were actually observed during the growth of a fiber in a methane atmosphere. Figure 7 shows the cross-section of such a region with defects photographed in polarized light and magnified by one hundred. The figure clearly shows several defects corresponding to Fig. 6b, and one large defect corresponding to Fig. 6a. A similar picture is observed in polarized light with respect to the change in the orientation of the crystallites at the site of a defect.

Later, fibers of 100 to 0.2 mm in thickness [15] were investigated. The investigation was conducted in polarized light on an ordinary microscope, on an electron microscope, and by diffraction of X-rays and electrons. Graphite fibers were obtained in a nitrogen atmosphere saturated with N-heptane at room temperature. The gas was passed through a quartz tube containing small pieces of iron and equipped with a thermocouple. The temperature in the tube mid section was maintained at 1000° with the aid of an electric furnace.

The investigation under a microscope in reflected polarized light in the hottest zone of the furnace disclosed not only (50 mm), thick (up to 0.2 mm), and straight threads, which had already been observed by other authors, but also spiral threads, some of which were significantly larger than the threads reported previously (Figs. 8a and b).

The pressure change did not noticeably influence the formation of threads in this case; therefore it can be assumed that their growth is a result of several defects in the catalyst particle. In this

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process the fairly characteristic threads shown in Figs. 8b, d and e were obtained; no satisfactory explanation for their formation has been found. The investigation samples were prepared by mounting them in polystyrene and then grinding them down with a diamond paste. In agreement with previous X-ray studies [3-5], it was discovered that axis C of the graphite lattice is approximately perpendicular to the fiber axis (deviating by only 10°). At the same time, in other cases the threads contain spherical segments with axis C directed radially from the center (Figs. 9a, b).

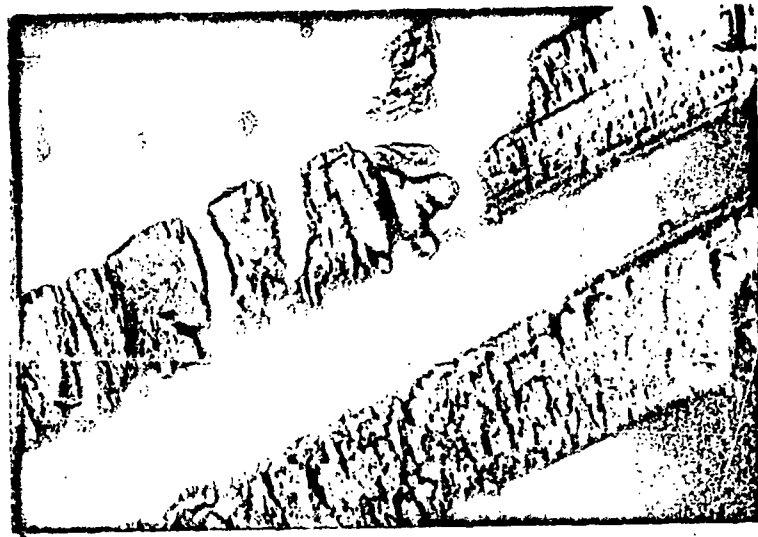
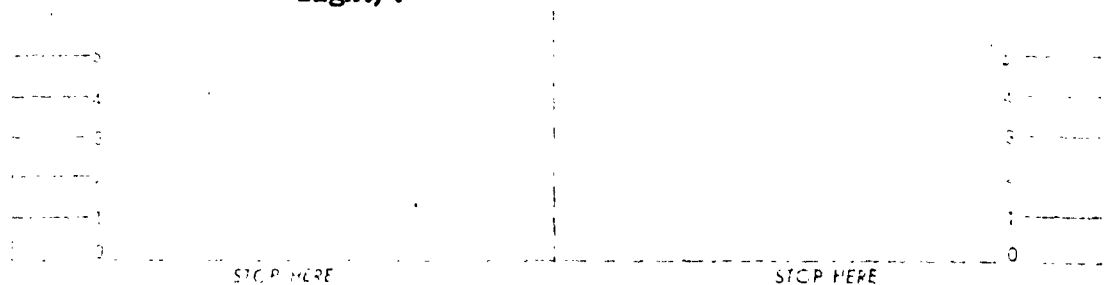


Fig. 7. Cross section of "damaged" region of a fiber covered with graphite during the decomposition of methane (X100, polarized light).



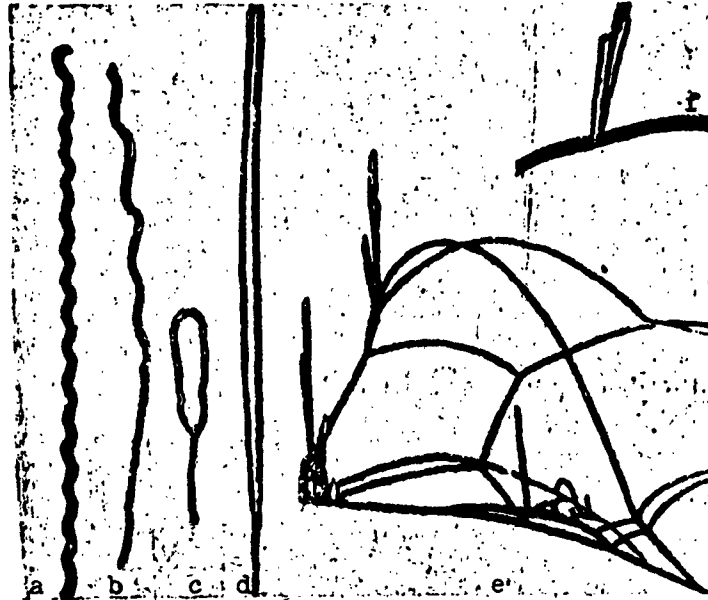


Fig. 8. Graphite fibers.
a) X120; b,c, and d) X16; e) X8;
f) X9.6.



Fig. 9. Longitudinal cross section of a fiber, X960 (a); and a cross section at a small angle to the longitudinal direction of the fiber, on which the development of a spherical segment is clearly seen, X480 (b); the fiber axes are horizontal; the arrows point out the direction of Nicol's prisms.

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In addition, the end of each thread is of spherical shape (Figs. 8f and 10a).

It was established from the observations that thick threads form as a result of a two-stage process. It was assumed that at first a longitudinal growth of very thin fibers with a constant cross section occurs; later thin, growth is replaced by radial growth. The internal structure of thick and straight threads shows that in certain cases they were formed during radial growth of thin, spiral threads, while in other cases the original thread was apparently completely straight. The hypothesis of a two-stage growth is supported by the results of the observation of the points of intersection between two straight threads and their center axes, which pass through an immediately adjacent space. Thus it can be assumed that the two threads, being very thin, touched each other, and a subsequent radial growth bound them together. In the second growth stage (in the thickening process) sometimes new centers of growth are formed on the thread surface, yielding spherical segments perpendicular to the fiber axis.

These new centers are usually more developed on one side of the thread (Fig. 10a), which indicates that they are generated from certain substances moving in the gas flow (for example, particles of carbon black). Thus sometimes the structures shown in Fig. 10b are obtained. In observing under a microscope in polarized light a cross section of a very straight thread made at a sharp angle (Fig. 9 b), we did not succeed in examining the thin original thread in the center. This gave us reason to assume that the original thread is very thin and cannot be seen with an ordinary microscope, which is confirmed by microphotographs made with an electron microscope (Fig. 11), where the broken end of a thread can be seen. The thin rod

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sticking up along the center axis may be the original fiber. We recall that previously [12] it was assumed that graphite fibers were formed merely by a longitudinal growth from the base, the final cross-section being established during the formation of the nucleus. However, in order to explain the microphotograph of Fig. 11, apparently we must still acknowledge a two-stage mechanism of the growth of the fibers. Thick threads were discovered only in the hottest zone. Investigation under an electron microscope revealed that the entire fiber is a hollow tube with the diameter of the cavity varying appreciably. It is entirely obvious that they do not consist of the two parallel fibers, which were assumed as a possible explanation of the previous less clear photos taken on the electron microscope [10].

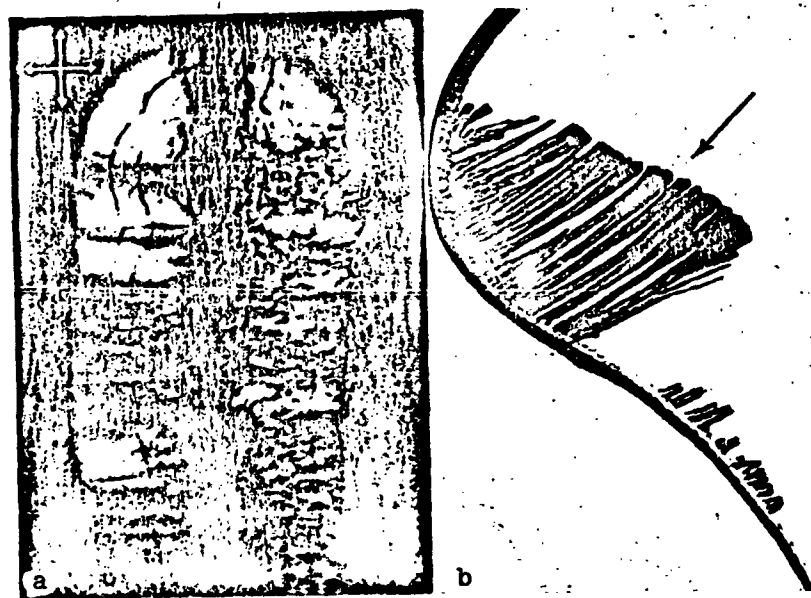


Fig. 10. Longitudinal cross section of the top of a fiber, X320 (a) and a fern-like growth of threads from a graphite fiber (b); the arrow indicates the direction of the gas flow, X16.

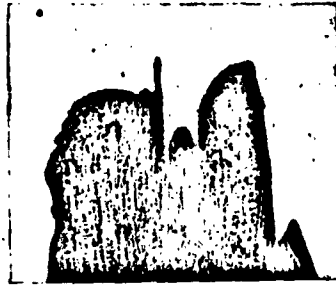


Fig. 11. The end of a broken fiber (X4500).

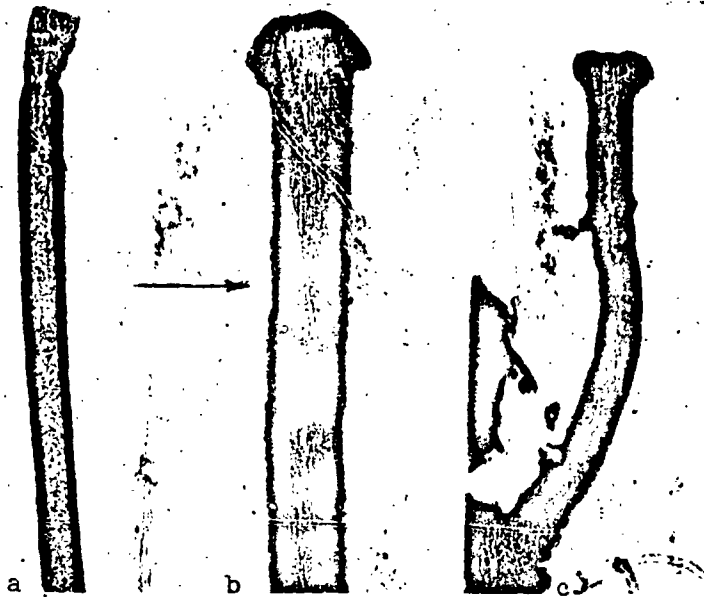


Fig. 12. Complete fibers.
a) without a catalyst; b and c) with catalyst particles (a,b - X75,000; c) X110,000).

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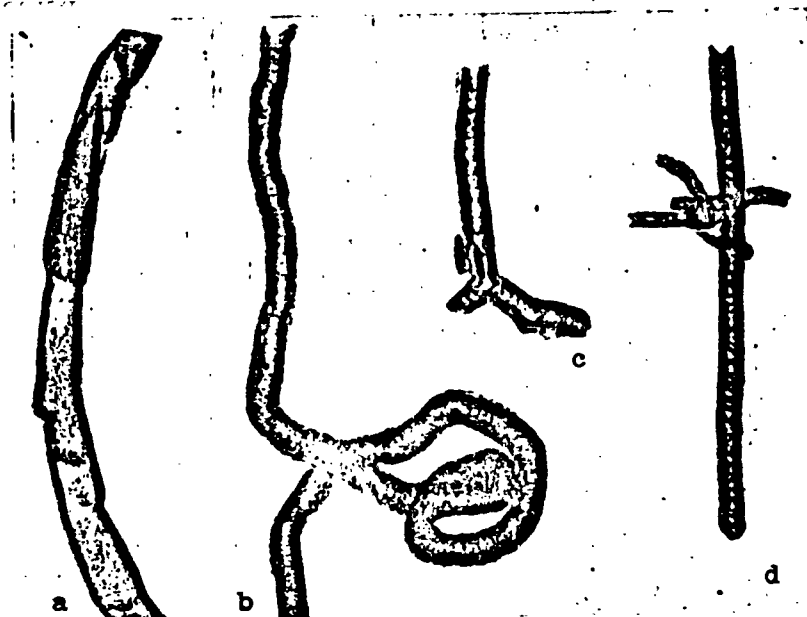


Fig. 13. Fibers, with crosspieces in the central cavity. (a - X40,000; b,c,d, -X75,000).

Figures 12b and c show fibers with particles of a dense material on the end (catalyst), as was assumed by a number of authors [8, 10, 11]. The lower half of the dense particle in Figs. 12b and c is conical, and apparently the diameter of the fiber cavity is determined by the shape of this cone. On the other hand, the conical shape itself may be a result of the growth of a graphite fiber from a catalytic particle. The assumption is made that the catalyst particle is moving in the growing fiber. In certain cases a considerable length of the cavity was filled with a thick material.

Figure 12 reveals an infrequent case where the tube is open and changes at the top into a very thin strip. Coiled bands were also observed. The existence of the bands was discovered only by graphite precipitation under high pressure in an electric arc [16].

It can be seen from Fig. 13 that the cavities in the threads can contain crosspieces at various intervals. From the shape of the crosspiece we can assume that they are thin layers covering a catalytic particle. The thickness of the crosspieces is approximately 20 Å. The fiber cavity is of conical shape at the top (Figs. 13b and d), indicating that at this place there was contact with the conical particles which were separated during the preparation of the threads. It can be seen from Fig. 13c that the ends of the two branches have the same shape as the lower end in Fig. 13d, which apparently was not attached to any dense particle. This observation is irreconcilable with the hypothesis of longitudinal growth of a fiber from the dense particles shown in Figs. 12b and c.

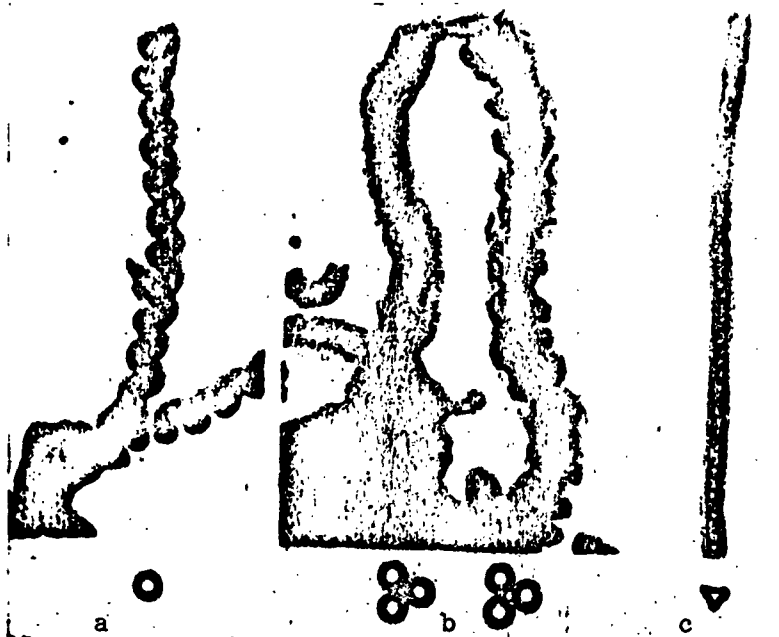


Fig. 14. Threads of fibers ($\times 40,000$)
a) individual spiral fiber; b) combination of three fibers, parallel and twisted; c) triangular fiber.

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Figure 14 shows an individual thread in the shape of a spiral and a combination of three threads which are sometimes coiled and sometimes completely parallel. The lower part of the picture shows the probable cross section of these threads, the shape of which depends on differences in the catalytic activity of the surface of the catalyst.

The significant intensity of the ring (002) in the electron-diffraction photo shows that the thin graphite threads are also formed from lamellar units laid in cylinders. Thus it is natural that they should contain a central cavity. It is characteristic that axis C has the same direction both in the thin fibers formed by longitudinal growth and in the thick threads, which are the result of subsequent radial growth. A measurement of the strength of the threads, made on a thread 12μ thick gave a tensile-strength value of 155 kg/mm^2 .

The extremely strong and elastic graphite "whiskers" obtained in a direct-current electric arc in an argon atmosphere under a pressure of 92 atm and at a temperature of 3700° K is reported in Bacon's article [16]. These "whiskers" are obtained as inclusions in a piece of graphite formed by diffusion of carbon vapor from the positive to the negative electrode. The diameter of this new form of graphite "whiskers", a form which has the properties of a single crystal along the axis, is from 1 to 5μ with a length of up to 3 cm. They consist of one or more tubes, and we assume that each tube is in the form of a coiled sheet of graphite layers extending continuously along the fiber axis with axis C exactly perpendicular to the fiber axis (Fig. 15).

These "whiskers" have a high degree of flexibility and a tensile strength amounting to as much as 2000 kg/mm^2 . Their Young's modulus

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equals $7 \cdot 10^{12}$ dyne/cm², while their electrical resistivity is approximately $65 \mu\Omega \cdot \text{cm}$, which is approximately equal to the value characteristic of a single crystal.

The structure of a "whisker" in the form of a coiled sheet of graphite layers (apparently consisting of several monolayers) is attractive from the standpoint that it explains simultaneously the thickening and elongation of a "whisker". In support of this structure is the fact that during the destruction of a "whisker" by a strong electric current, large sheets, parallel to each other, peel off around the periphery.

The great mechanical strength and elasticity and the corrosion and temperature stability of fibrous carbon material permits us to hope that wide use of it will be made in the national economy. For this reason it is necessary to devote special attention to the study of the structure, properties, and synthesis of various types of fibrous carbon.



Fig. 15. Model of a graphite "whisker" with a convolute structure.

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