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**CONFIDENTIAL****CONCERNING INVESTIGATION OF THE GRAPHITIZATION PROCESS**

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[Some results of work on graphitization done by GIPKh (State Institute of Applied Chemistry) in 1932-1933 are included in this paper.]

1. All black modifications of carbon represent conglomerates of graphite crystals which may vary in size. There is no such thing as amorphous carbon. When atoms of carbon are formed as a result of the decomposition of organic substances, they immediately become oriented. By means of the free valencies of end atoms of crystals formed in this manner, adsorption of residues of decomposing hydrocarbons takes place. This direction of the process is determined by the properties of the carbon atom referred to as its "metallic quality" (1).

The process of graphitization must be regarded as one of the growth of small crystals of graphite to a larger size. The basic characteristics of this process were clarified in Arndt and Pollack's work on a number of carbonaceous materials (2). They came to the conclusion that in the process of graphitization (at 2450°), the recrystallization of carbon proceeds in a manner similar to that occurring in the case of metals (according to Tammann), and that differences in the ability to graphitize are due to

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differences in the initial structure of the carbonaceous materials. The lack of a tendency on the part of anthracite and charcoal to graphitize is due to an insufficient proximity of individual elements of the structure (i.e., an inadequate density). In the case of anthracite a layer structure, and in the case of charcoal a fibrous structure prevent graphitization. As a result of studying the graphitization of petroleum coke (3), Arnfeldt came to the conclusion that the growth of graphite crystals represents an instance of the growth of two-dimensional parallel layers forming a layer lattice. According to Arnfeldt, the process of graphitization is a slow crystallization under formation of typical layer lattices. As the rate of diffusion in solids is slow and the directive forces acting parallel to the layers of the future lattice are weak, the process of crystallization must be slow in this particular case.

By investigating the graphitization of a number of materials, Veselevskiy and Pertsov (4) have shown that the change of properties in the course of graphitization takes place in a particularly abrupt manner in a definite temperature interval. These authors consider that in the temperature interval in question "a transition of coal from a colloidally dispersed state to a coarsely dispersed state" takes place. Just as Arndt and Pollack, they are of the opinion that the process of graphitization proceeds according to laws governing the thermal recrystallization of polycrystalline substances.

The effect of ash constituents which according to Acheson promote graphitization (5) is denied by Veselovskiy and Pertsov, while Arndt and Pollack consider that they even may impede graphitization (2). Arnfeldt is of the opinion that diffusion of carbon is considerably impeded in the liquid phase, so that the presence of ash in a carbonaceous material has a favorable effect on graphitization.

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In the present paper some results on the graphitization of anthracite and other carbonaceous materials are reported. These results indicate that ash constituents actively participate in graphitization, and that furthermore their fusion and evaporation determine the sharpest change in a number of properties of the materials being graphitized. This applies particularly to anthracite.

2. Graphitization was carried out in <sup>4.1</sup> Tammann tubular furnace illustrated in Figure 1. The temperature was controlled by means of a Holborn-Kurlbaum optical pyrometer. The precision of temperature measurement at 2500° was  $\pm 30^\circ$ . In the samples under investigation, the specific electrical resistance expressed in ohms per centimeter [per unit cross-section] was determined; furthermore, the actual density, the ash content, and in some cases the ignition point. On a number of samples the X-ray diagrams were taken. Petroleum, foundry, and tar coke were submitted to graphitization as well as several grades of anthracite from the Donets Basin. Table 1 lists the specific resistances, densities, ash content, and ignition points for samples which have been heated at 800° for one hour. The resistance was determined on powders of -75+125 mesh on an Arndt apparatus (2) at a pressure of 115.8 kg/cm<sup>2</sup>.

Among the materials listed in that table, petroleum coke and tar coke were the most uniform, while the presence of inhomogeneous particles was apparent in the case of petroleum coke. The high resistance of tar coke in comparison with foundry coke and petroleum coke is apparently due to its lower temperature of formation (800°). All of the cokes had approximately the same density. The ash content is highest in foundry coke. The anthracites differ from coke in regard to density and particularly as far as electrical resistance is concerned. Their higher resistance is apparently due to the

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presence of organic substances which had not yet decomposed. Anthracites A. K. and No. 1 are distinguished by a fine layer structure which is barely perceptible to the eye. On breaking they form a smooth, slightly cockle-shaped surface.

For graphitization 50 grams of pieces having dimension<sub>s</sub> of 20-30 millimeters were used. A compressed package of this material was placed in the center of the carbon tube in such a manner that it occupied approximately 20 centimeters of the tube's length. Preliminary experiments established that through a distance of 20 centimeters the temperature gradient at 200° does not exceed 50°.

After charging the tube, the temperature was raised to the required point during 1 hour and the samples were then kept for 1 hour at the final temperature. Results of some experiments are listed in tables 2, 3, and 4. Table 3 cites for comparison the corresponding data on the initial materials.

The results of the experiments show that the initial materials can be divided into two groups on the basis of the resulting graphite's electrical resistance: cokes and anthracites. From this point of view, petroleum coke yields the best results. Tar coke and foundry coke are slightly inferior to petroleum coke. Tar coke exhibits a relatively high ash content after graphitization, which is apparently due to the fact that its pores are closed, so that the ash cannot volatilize and escape from larger pieces. As far as external appearance is concerned, the samples of coke are not changed perceptibly in the course of graphitization. They also do not undergo any mechanical changes. After graphitization the various types of coke acquire a silvery grey color, while remaining very hard.

The behavior of anthracites is entirely different. As distinguished from coke, anthracites after graphitization split into plates which swell perpendicularly to the layers, thus increasing the volume of the material by a factor of 4 to 6. Upon powdering graphitized anthracite yields a soft,

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sooty graphite resembling natural graphites [with respect to texture and structure], but differing from the latter in that it exhibits a higher reactivity. As can be seen from the tables, the specific electrical resistance of graphitized prepared from anthracite is very high, namely 4 times higher than the resistance of graphitized petroleum coke. This high resistance is due to the layer structure of the powder, the platelets of which lie parallel to each other when the resistance is measured. For that reason the resistance measured by this method is not characteristic for graphitized anthracite. The density of graphitized anthracite is not lower, but superior to that of the graphite obtained from coke. The ash content of anthracite remains pretty high after graphitization and amounts to ~1.0%. This circumstance is due to the difficult removal of ash from large pieces and to the swelling of these pieces. Experiments on the graphitization of fine anthracite powder in an Acheson furnace have shown that in this case the ash content drops to 0.1-0.3%. While swelling of fine anthracite powder also occurs, it is not so extensive because of the small size of the particles. It is noteworthy that fine anthracite powder on graphitization has a much lower resistance than graphitized coarse pieces (0.0140-0.0160 ohms per centimeter as compared with 0.0316 ohms per centimeter).

3. The peculiar behavior of anthracite in graphitization was noticed by the author of this paper in his earliest work (7). It was found that when the temperature is raised slowly in the graphitization of anthracite lumps in a furnace of the Acheson type (30 kilowatts) with a coke filling [or on a coke bed], the lumps of anthracite do not crack, but swell in a direction perpendicular to the layers, increasing in volume 4-6 times.

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The swollen <sup>are</sup> pieces of anthracite which have swollen as a result of graphitization. ~~This product is~~ extremely soft and elastic. As a result of receiving an impact in the direction across the layers, the swollen piece is flattened into a plate which is highly flexible and has mechanical properties resembling those of the graphitized threads obtained by Pirani and Fehse (8). Thin compressed plates obtained from swollen pieces can be rolled into tubes without breaking and can be freely cut with a knife. Determinations of the electrical resistance of a plate cut from a well-swollen piece showed that across the layers  $\rho = 0.0327$  ohms/cm, while along the layers  $\rho = 0.0064$  ohms/cm. These figures explain the high resistance of the product from anthracite powder, and also demonstrate that the resistance along the layers does not differ much from the resistance of graphitized coke. The determinations of electrical resistance were carried out on a plate from a swollen piece of anthracite without submitting the plate to any sort of treatment. Consequently the plate retained hollow spaces inside.

As has been indicated above, anthracite cracks into plates during graphitization in a Tammann furnace. The plates formed in this manner then swell. A number of experiments has shown that the reason for this is an excessively high rate of temperature increase. When anthracite of grade A. K. is heated in a Tammann furnace, it begins to crack at 1980-2000°. When the experiment was completed at that temperature, solid lumps covered with cracks were obtained. When anthracite was heated up to 2800°, particularly strong cracking was observed in the range of 2100-2300°. As a result of this cracking, separate silvery-grey plates are obtained, but they are still hard and brittle. When the temperature is further raised to 2550-2650°, cracking ceases and swollen plates which have become soft and resilient are obtained. By conducting a number of experiments on A. K. No. 1 anthracite, it was established that swelling and softening of this type of anthracite

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takes place only when the temperature is no lower than 2500-2600°.

The results of these experiments are cited in Table 5. ~~Figure 9 shows the changes in the density and ash content of anthracite which occur in graphitization. Figure 6 and 8 show the surface of plates obtained by graphitizing anthracite at 2300° and 2600°, respectively. Figure 7 shows how a plate from anthracite was blown up from the inside until it practically assumed the shape of a sphere after graphitization.~~

As can be seen from the cited data, only the specific electrical resistance is changed when anthracite is heated from 1200° to 2000°, while other properties are affected very slightly. However, the change in the resistance is due to failure to hold the sample at 1200° for a sufficient length of time: when anthracite is kept for 6 hours at 1200°, a drop of the resistance down to 0.0580 ohms/cm results (9). Thus, the actual change of the resistance in the temperature range under consideration must also be insignificant. As mentioned above, the appearance of anthracite does not change very perceptibly in this temperature range. On the other hand, an increase of the temperature by only 300° beyond this range (from 2000° to 2300°) brings about very noticeable changes in the anthracite. Table 5 shows that the ash content is strongly reduced (from 3.9 to 1.4%) while the density is strongly increased (cf. Figure 9). To this period corresponds a pronounced change in the appearance of the anthracite: there is extensive cracking. With a further increase of the temperature, modification of the properties of the anthracite continues, but this modification is no longer intensive. Cracking of the anthracite stops and is replaced by swelling. It is obvious that in this particular range ( $T^{\circ} > 2300^{\circ}$ ) the brittleness of the anthracite disappears and it becomes resilient, so that the swelling brought about by the internal pressure of ash vapors can take place. ~~This blowing up of the layers can be clearly seen in figures 6 and 7.~~



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~~Figure 8 shows a 650 times magnified view of the surface of a layer of graphitized anthracite treated at 2600°. Under the microscope this surface appears uneven and semispherical convexities are visible on it. At the same time the surface is very smooth, as if it were polished, and reflects light strongly.~~  
 The surface of graphitized anthracite treated at 2600°. Under the microscope this surface appears uneven and semispherical convexities are visible on it. At the same time the surface is very smooth, as if it were polished, and reflects light strongly.

The sharp changes in the properties of anthracite in the range 2000-2300° are accompanied by a transition of the ash constituents into a molten and evaporized condition. The presence of the ash constituents in this state creates favorable conditions for the recrystallization of carbon, which explains the abrupt change of properties mentioned above. (cf. Figure 9). This type of graphitization is characteristic not only for anthracite, but also for a number of varieties of coke, as was shown by Veselovskiy and Pertsov (4). It has been shown (9) that even in the graphitization of electrodes made of petroleum coke, the electrical resistance changes to an insignificant extent up to 2000°, and that only in the range 2000-3000°, the resistance drops from 0.0050 ohms per centimeter to 0.0024 ohms per centimeter (cf. Figure 10). This drop is accompanied by a sharp reduction of the ash content. It is obvious that the observed abrupt change in the properties of a carbonaceous material that is being graphitized depends on the nature of the ash constituents, and that this change consequently may take place in various temperature ranges. In view of the fact that most carbonaceous materials have approximately the same ash composition, the temperature ranges in question do not differ much from each other.

When anthracite was partly de-ashed (down to an ash content of 0.9%), the swelling was strongly reduced, and a fairly hard and brittle product was obtained, although the temperature of graphitization was 2650°. This graphitized anthracite resembles graphite obtained at 2300° from anthracite which had not been de-ashed.

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4. An X-ray investigation of the products was carried out by Ya. L. Khurgin at the Leningrad Industrial Institute. The samples were ground into a powder and after addition of dextrin past<sup>e</sup> were pressed by means of a capillary into rods having a diameter of 0.4 mm or 0.62 mm. The exposures were made in Debye chambers with a drum of 15.6 mm diameter. Hadding tubes with iron anticathod<sup>e</sup>s were used. The exposure was continued for 20-24 hours.

In Table 6 are cited values for the dimensions of crystals resulting from petroleum coke and anthracite after the material in question had been heated at the temperature indicated in the table. Figure 11 shows the results of a photometric examination of the X-ray diagrams by means of an automatically registering Moll microphotometer. The results listed in the table show that in the graphitization of anthracite a particularly strong growth of crystals sets in at 2300°; i.e., this growth begins in a temperature range in which a sharp modification of most of the properties of anthracite takes place and the removal of ash starts. At 2600° the crystals become still larger.

One can see from the photometric curves that reflexion (002) is doubled for samples c, d, and f. This phenomenon, which had already been observed by Ebert, is explained by the fact that the (001) planes are slippage planes in the case of graphite. When the rods are pressed out of the capillaries, the resulting orientation is such that reflexion may take place only from (001) planes which are near the surface. This results in a doubling of the line. Thus the doubling of lines indicates that there are sizeable crystals which have well-developed slippage planes.

In the case of anthracite this doubling is first observed in the sample graphitized at 2300° and is especially pronounced in the sample graphitized at 2600° (Figure 11, d). The same doubling of the line (002) occurs in Acheson graphite (Figure 11, f).

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Figure 11 also shows the gradual increase of the intensity of lines with progressing graphitization. Lines (002), (201), and (200) [orthohexagonal (rhombic) indexing] are already sufficiently clear in the first samples. In the case of the sample graphitized at 2300° (Figure 11, C), the intensity of lines (004), (020), (021), and (006) increases considerably.

The cracking of anthracite into plates, the swelling of these plates, and the resulting elastic properties have led to the assumption that in the process of graphitization the crystals are oriented with their basal planes along the anthracite layers. Pirani and Fehse (8) demonstrated that oriented graphite crystals are responsible for the high flexibility of threads prepared from acetyl cellulose. Turner and Anderson (11) showed that in some regions of anthracite there is orientation of crystals.

In order to investigate the orientation of graphite crystals in anthracite layers, X-ray diagrams were taken in the following manner. A sample having the shape of a plate was cut from the anthracite perpendicularly to the layers. This sample was affixed with an adhesive in a Laue chamber. Hadding tubes with iron anticathodes were used as a source of X-rays. The resulting X-ray pictures confirmed the assumption which had been made in regard to the orientation of graphite crystals. The incomplete ring which is visible in all four pictures of Figure 12 is formed by the reflexion of  $K_{1,2}Fe$  from plane (001) in the second order (002). The second ring of a smaller diameter which appears in the pictures of Figure 12 is due to the reflexion of  $K_{3,4}Fe$  from the same planes. The total data indicate the following structure. The hexagonal axes which are perpendicular to the (002) planes are not all perpendicular to the anthracite layers, but disposed within a conus. The axis of this conus is perpendicular to the anthracite layers, while the inner angle formed by its tip is different for different samples (cf. Table 7). The distribution of the axes within the conus shows a frequency increasing towards the axis of the conus. Furthermore in samples 1

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and 4 of Table 7 the ring is complete, which indicates the presence of a certain number of crystals oriented at random. The orientation is most pronounced in the case of sample 3. The decrease of the degree of orientation with increasing temperatures is due to the beginning of a loosening (swelling) of the anthracite layers, which has a disorienting effect on the latter. Nevertheless, one can see that in samples 2, 3, and 4 the majority of crystals are oriented along the layers.

5. The experimental results show that cokes and anthracites yield substantially different kinds of graphite. On the basis of electrical resistance, the best graphite is yielded by petroleum coke and the worst by anthracite. The high electrical resistance of graphitized anthracite is due to an extensive loosening of the structure during the period of swelling and also to an increased resistance in the direction perpendicular to the layers.

The reduction of the <sup>e</sup>degree of swelling of anthracite by preliminary powdering leads to a lower electrical resistance of the resulting graphite. One may eliminate the swelling of anthracite almost completely by graphitizing an electrode pressed from the powder rather than the powder as such. The author of this paper has shown that with fine powders electrodes made from anthracite have the same resistance as electrodes made from petroleum coke, or even a lower resistance than the latter (9).

If the appearance of the graphite and its softness are considered, graphites made from anthracite are much better than those obtained from coke.

Published statements to the effect that anthracites yield a hard graphite (2, 6, 10) are due to the fact that the authors in their experiments did not use a temperature which was high enough to convert the anthracite into a soft graphite.

X-ray data show that crystals of approximately the same size are formed from both anthracite and coke as a result of graphitization.

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The differences are due in the main to oriented growth and oriented arrangement of crystals in anthracite. In coke there is no orientation of crystals whatever. For that reason the typical stages of graphitization exhibited by anthracite are absent in coke. The only exception is retort coke which, as shown by Arndt, Pollack, and Schaidhauf (12), also swells during graphitization.

Thus one sees that in general the properties of the produced graphite are determined by the properties of the starting material and its structure. In evaluating the quality of the graphite, one should not restrict oneself to the consideration of only one property such as the density, as is done by Arsem (6), or of the specific electrical resistance (Arndt), but also consider other properties like elasticity, greasiness, etc, bearing in mind that these properties may vary to an appreciable extent with different carbonaceous materials (different grades of anthracite, for instance).

One must distinguish two stages in the process of graphitization. The first ends at approximately 1200° and is determined by the growth and formation of graphite crystals. As shown in a number of investigations, these processes take place in the beginning at the expense of carbon formed in the decomposition of organic compounds contained in the carbonaceous materials. Later on the growth of crystals is due to other causes. According to Arndt and Pollack, the growth of crystals above 1200° proceeds at the expense of a recrystallization which is analogous to the recrystallization of metals as interpreted by Tammann. Veselovskiy and Pertsov substantially agree with this conclusion. Arndt and Pollack state that a laminated or fibrous structure of the starting material interferes with recrystallization, as can be observed on the examples of anthracite or charcoal. As far as ash is concerned, it is generally assumed that ash is of no consequence in graphitization, or else that it impedes graphitization by preventing the crystals from adhering to each other and merging.

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The results of the present investigation show that ash cannot be regarded as an inactive ingredient in the process of graphitization. Recrystallization of carbonaceous materials above 1200° cannot be considered independently of the evaporation of ash. Experiments have shown that ash actively participates in the process of graphitization as soon as it has assumed the liquid or gaseous state. This can be clearly seen in the graphitization of anthracite, which remains substantially unchanged up to 2000°, but undergoes sharp changes in the range 2000-2300° due to the fusion and evaporation of the ash in this temperature range.

The ash content diminishes in the temperature range in question, while the resistance of electrodes made from petroleum coke drops at the same time (cf. Figure 10). The presence of the liquid and gas phases of the ash constituents facilitates the diffusion of carbon. As a result of this, the growth of crystals is accelerated, which is confirmed by the data of the X-ray investigation.

At higher temperatures the ash content drops due to volatilization. However, the crystals had a sufficient time to grow during the period of evaporation of the ash. Subsequently the effect of the fused ash is less pronounced due to both the lowered ash content and the increased crystal size.

In view of the fact that the majority of carbonaceous materials contain approximately the same ash ingredients, the effect of the ash in all of them becomes noticeable in about the same temperature range. This applies to anthracite, electrodes from petroleum coke, and in Veselovskiy and Pertsov's experiments to petroleum coke, tar coke, gas coke, anthracites, charcoal, and other materials.

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While recrystallization of carbonaceous materials proceeds in the solid phase at temperatures reaching as high as 3000°, this recrystallization is very slow. The evaporation of small crystals of carbon and subsequent condensation of vapors on larger crystals in the temperature range 2000-3000° is extremely unlikely because of the low vapor pressure of carbon in that temperature range (cf. van Saar, Z. anorg. allgem. Ch. 1928; Kohn and Hikkel, Z. f. Phys. Vol. XXVII, 305).

According to Nishigama's data (13), increasing of the time of heating at a constant temperature in the graphitization of prepared carbon filaments begins to have an effect only at 3000°. For this reason the abrupt modification of properties of carbonaceous materials in the range near 2000° cannot be explained by recrystallization in the solid phase without taking into consideration the effect of the ash.

In the light of the information given above, one may form the following ideas on the graphitization of anthracite. When anthracite is heated up to 1200°, decomposition of organic compounds takes place, leading to the growth of the original crystals up to dimensions amounting to scores of Ångströms. There are no substantial changes between 1200° and 2000° after all organic compounds have decomposed. When the temperature is raised further, the ash between the layers of anthracite first melts and then is transmitted into the gaseous state. Depending on the rate of temperature increase, the process may proceed in two different ways at this point. If the rate of temperature increase is high, the anthracite cracks into plates. If the temperature rises at a slow rate, swelling of the anthracite may take place. The fact that swollen pieces of anthracite remain hard when the experiment is terminated prematurely leads to the unavoidable conclusion that at some higher temperature in this range the graphitized anthracite becomes elastic. It is possible that diffusion of ash constituents into the layers promotes the formation of the elastic product.

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The presence of ash constituents at these temperatures facilitates the diffusion of carbon, so that an accelerated growth of graphite crystals takes place. Due to the directing influence of the anthracite layers, this growth proceeds along the surface of the layers, so that the X-ray diagrams taken during this period show the most pronounced degree of orientation (picture 3 of Figure 12). When the temperature is raised further, loosening of the anthracite layers occurs under simultaneous growth of the crystals, which at this stage are already oriented. Notwithstanding the greater size of crystals at this point, the degree of orientation is reduced owing to the bending of the thin layers. One may mention here that the presence of oriented crystals is conducive to crystal growth (8).

The elasticity [resilience] and greasiness which are characteristic of graphitized anthracite are due precisely to the orientation of crystals.

Partial de-ashing of anthracite reduces its swelling and the resulting graphite is hard. Under the circumstances one must conclude that the softness and elasticity of graphite are due to the ash constituents, which facilitate orientation in addition to expediting the growth of crystals. The graphitization of other carbonaceous materials proceeds basically along the same lines as the graphitization of anthracite. However, the properties of the starting material affect the process in a specific manner in each individual case.

From the technological standpoint, when the aim is to produce graphite powder, one must select the right kind of anthracite and then insure good swelling by raising the temperature appropriately and bringing the material up to the right temperature. In order to improve the mechanical properties of electrodes made from anthracite powder, one should use the finest anthracite powder and raise the temperature in the 2000-2300° range at a particularly slow rate. It is not advisable to raise the temperature above 2300°, because



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otherwise the electrodes may become too soft without any appreciable gain as far as improvement of electrical conductivity is concerned.

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- [ 7 tables and 4 figures and 1 last of figures follow ]

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Table 1. Properties of Carbonaceous Materials Submitted to Graphitization.

Carbonaceous material.	Specific electrical resistance $\rho \times 10^4$ ohms/cm	Density of 250 mesh powder.	Ignition point in °C of grains having dimensions of 0.5-1.0 mm.	Total ash content in %
Petroleum coke	760	1.859	480	1.65
Tar coke	1640	1.944	540	1.36
Foundry coke	1290	1.807	535	8.00
Anthracite from Nesvet'ev stratum of the mine imeni OGPU	18600	1.770	-	2.46
Anthracite "Kulak" (A.K.)	9500	1.643	475	4.21
Anthracite No. 1	10770	1.671	480	2.84

Table 2. Graphitization of Petroleum Coke.

Temperature of experiment in °C	Duration of experiment in minutes.	Specific electrical resistance in $\rho \times 10^4$ ohms/cm	Density	Ash content in percent
2400	60	80	2.137	0.30
2400	60	85	2.170	0.31
2550	60	90	2.178	0.51
2400	120	90	2.235	0.33

Table 3. Graphitization at 2400-2500°C.

Material.	Before graphitization:			After graphitization:		
	Specific electrical resistance $\rho \times 10^4$ ohms/cm	Density	Ash content in percent	Specific electrical resistance $\rho \times 10^4$ ohms/cm	Density	Ash content in percent.
Petroleum coke	760	1.859	1.85	85	2.170	0.31
Tar coke	1646	1.944	1.36	124	2.176	1.16
Foundry coke	1245	1.807	8.78	120	2.163	0.91
Nesvet'ev anthracite	13600	1.770	2.46	360	2.20	0.78

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Table 4. Graphitization at 2600°C.

Material	Specific electrical resistance $\rho \times 10^4$ ohms/cm	Density	Ash content in percent	Ignition point in °C	Appearance and hardness
Petroleum coke	87.4	2.171	0.07	659	Hard, of silvery grey color.
Foundry coke	97.5	2.100	0.16	615	Not so hard as before graphitization. Color silvery grey
Tar coke	102.6	2.12	0.59	645	The same as in the case of petroleum coke.
Anthracite No. 1	316	2.213	1.80	650	The pieces split into plates. The latter swelled perpendicularly to the layers and became very soft.

For comparison with the data listed in this table, one should note that the specific electrical resistance of the powdered material of a Siemens-Planina graphite electrode (75+125 mesh), when measured under the same conditions, was found to be 0.0082 ohms per centimeter, i. e. practically the same as that of graphitized petroleum coke.

Table 5. Graphitization of A. K. Anthracite.

Temperature in °C	Specific electrical resistance $\rho \times 10^4$ ohms/cm	Density	Ash content in percent	Temperature of ignition in °C in the air for grains having dimensions of 0.5-1.0 mm	Appearance and mechanical properties.
800	9500	1.643	4.21	475	Dense dark pieces
1200	1095	1.848	4.23	607	Pieces developed surface cracks
2000	615	1.883	3.9	625	Pieces cracked into individual separate plates of a silvery grey color.
2300	463	2.166	1.4	625	
2600	228	2.226	1.09	580	Plates swelled and became soft.

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Table 6. Determination of the Crystal Size by the X-ray Method.

Material	Temperature (in °C) and duration of heat treatment.	Dimensions of crystals in Angstroms	
		Along the a and b axes	Along the c axis
Petroleum coke	800 - 1 hour	19 ± 2.0	16 ± 1.5
" "	2500 " "	» 180	» 180
A. K. Anthracite	800 [̄ 7]	18.3 ± 2	11 ± 1.0
" " "	1200 " "	32 ± 5	16.0 ± 1.3
" " "	2300 " "	-118.0	77 ± 12
" " "	2600 " "	» 180	» 180

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Table 7. Effect of Heating on the Orientation of Crystals.

No. of Experiment.	Kind of sample.	at Tip Inside Angle/of Corns Formed by Hexagonal Crystal Axes.
1	A. K. Anthracite heated for 1 hour at 800°	48°
2	Ditto, heated for 6 hrs. at 1200°	48°
3	Ditto, heated for 1 hour at 2300°	36°
4	Strips of swollen anthracite after heating at 2600°	56°

1-  
63



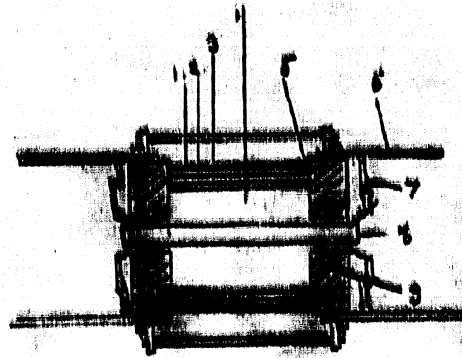


Figure 1. Laboratory Resistance Furnace.

1 - asbestos; 2 - chamotte; 3 - asbestos; 4 - carbon black and charcoal;  
5 - jacket; 6 - current lead; 7 - cooler; 8 - carbon tube; 9 - carbon  
blocks.

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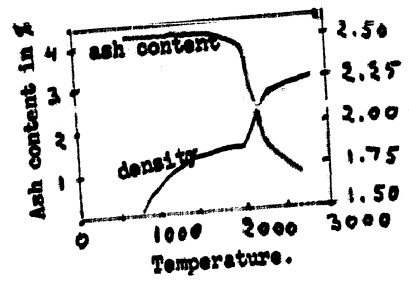


Figure 9. Changes in Ash Content and Density of A. K. Anthracite During Graphitization.

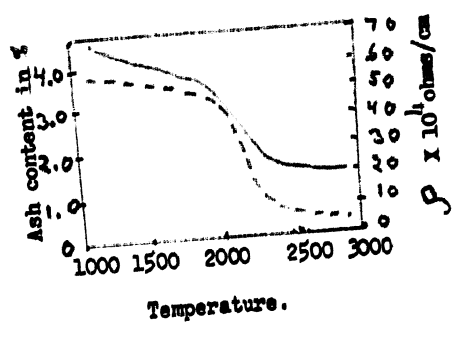


Figure 10. Changes in Ash Content and Specific Electrical Resistance of Petroleum Coke Electrodes During Graphitization.

— specific electrical resistance  
 - - - ash content

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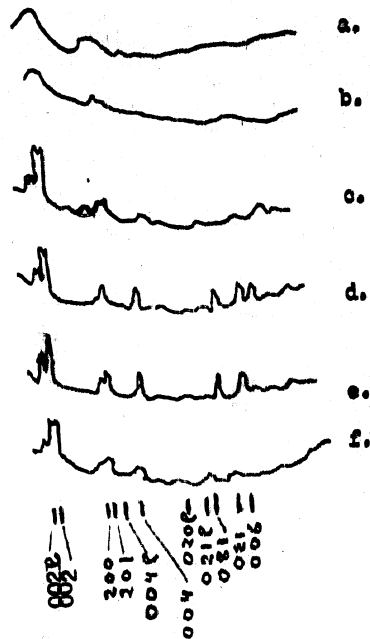


Figure 11. Photometric Curves of X-ray Diagrams Taken From the Following Samples of Anthracite and Graphite:

- a) anthracite heated for 1 hour at 800°;
- b) anthracite heated for 1 hour at 1200°;
- c) anthracite heated for 1 hour at 2300°;
- d) anthracite heated at 2600°;
- e) petroleum coke heated for 1 hour at 2550°;
- f) Acheson graphite.

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*are omitted but*  
[The figures listed below can be seen in the original text in the Library of Congress]

- Figure 2. Scales of Graphite Prepared From Anthracite (+125 mesh). Magnification 1:50.
  - Figure 3. Scales of Ceylon Graphite (+125 mesh). Magnification 1:50.
  - Figure 4. Swollen Anthracite.
  - Figure 5. Swollen Anthracite.
  - Figure 6. Surface of Anthracite Layer After Graphitization at 2300°. Inflations Are Visible on the Surface. Magnification 1:12.
  - Figure 7. Anthracite Plate Blown Up Into a Sphere. Magnification 1:12.
  - Figure 8. Surface of Anthracite Which Had Been Graphitized at 2600°. Magnification 1:50.
- 
- Figure 12. X-ray Diagrams Showing Changes in the Orientation of Crystals During the Graphitization of Anthracite.
    - 1) Heated for 1 hour at 800°;
    - 2) heated for 6 hours at 1200°;
    - 3) heated for 1 hour at 2300°;
    - 4) heated at 2600°.

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