

## Electron Microscopic Observation of Carbon Brushes. I.

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(Received June 16, 1952)

It was found in the previous paper concerning the X-ray examination of several commercial carbon brushes that the degrees of graphitization, i. e. the completeness of crystallization and the crystal size and form of graphite crystallites of brushes could be distinguished from each other by the X-ray method.<sup>(1)</sup> The electron microscopic examination of carbon brushes has been made in the present study to observe the microstructure of brushes, such as the mode of aggregation, the form and size of carbon particles.

Carbon brushes observed were the laboratory products of known composition and treatments.

### Experimental

#### Samples:

Experimental carbon brushes of Nippon Carbon Co.:

Raw materials and intermediate product:

A: Carbon black (Nippon Oil Co.), Ash 0.11%, Density 1.730.

B: Pitch coke (Great Lake), Ash 0.8%, Density 2.080.

C: [(A 50%, B 50%): Tar=10:6] graphitized at 2500°C.,

Carbon brush blocks:

D: [Powder of C):(Tar 30%, Pitch 70%)=100:45] graphitized at 2500°C.,

E: [(B graphitized at 2500°C. 50%, Domestic coal tar pitch coke (ash 1.0%, density 1.800) graphitized at 2500°C. 50%):(Tar 30%, Pitch 70%)=100:45] graphitized at 2500°C.,

F: [(Coal tar pitch coke graphitized at 2500°C.):(Tar 30%, Pitch 70%)=100:45] graphitized at 2500°C.,

G: [(B graphitized at 2500°C.):(Tar 30%, Pitch 70%)=100:45] graphitized at 2500°C.,

H: [(A 30%, B 70%):Tar=10:6 graphitized at 2500°C.):(Tar 30%, Pitch 70%)=100:45] graphitized at 2500°C.,

I: [(A 50%, B 50%):(Tar 80%, Pitch 20%)=100:45] graphitized at 2500°C.,

J: [(A 50%, B 50%):(Tar 50%, Pitch 50%)=100:45] graphitized at 2500°C.,

K: Carbon brush block showing glancing specks.

Raw materials, intermediate product and some of the brush blocks were powdered and dispersed in a dilute aqueous solution of alcohol by super-sonic

vibration. Surface replicas of carbon brushes were prepared to observe the microstructure of brushes. The block of the carbon brush sample was flattened by rubbing on a ground glass plate, with a little amount of No. 800 Alundum and water. Carbon fragments which adhered on the surface of the block were rubbed away by a piece of wet cotton. Ten % solution of polyvinyl alcohol or nitrocellulose was placed on the surface. A film which formed on the surface was stripped, so as to remove carbon fragments attached to the surface of the block. It was necessary to repeat the procedure two or three times in order to obtain a clear replica with only a small amount of adhered carbon particles. Positive aluminum replicas of the cleaned surface were prepared by the well-known methyl metacrylate-aluminum method. The thickness of aluminum replica film of about 25~30 m $\mu$  was suitable to observe carbon brush surfaces.

#### Results:

A: The particle size of A was 0.1~0.05  $\mu$  or smaller (Fig. 1.).

B: The powdered B consisted of irregular flakes (Fig. 2.).

C: C consisted of two different parts, i. e. parts of aggregates of minute carbon particles of approximately the same size as A and parts of pitch coke origin resembling B (Fig. 3.). The appearance of minute carbon particles of the former parts was slightly altered from the original black. They must be single crystals of graphite as were observed in the graphitized carbon blacks. The hexagonal face angle could not be seen, as the particles were too small to resolve the angle with our instrument.

D: The observation of surface replicas of D showed that the parts of pitch coke origin were aggregates of flat and irregular crystals and the parts of carbon black origin had small projects of approximately the same size as the original black (Fig. 4. and 5.).

E: Flat and irregular crystal surface of the size 0.5~0.6  $\mu$  predominated and crystal surfaces of several microns were observed in some parts of the replicas of E. Thin irregular crystals of graphite were found attached on the replica (Fig. 6.). The layer structure of graphite crystal can be seen on the large crystal surface shown in Fig. 7. A sectional view of layers of graphite crystals were reproduced in the replica and are seen near the large crystal surface on the figure. Smaller crystal surfaces are seen in other parts of the replica. Minute particles of ca. 0.01  $\mu$

(1) T. Noda and S. Sato, This Bulletin 25, 195 (1952).

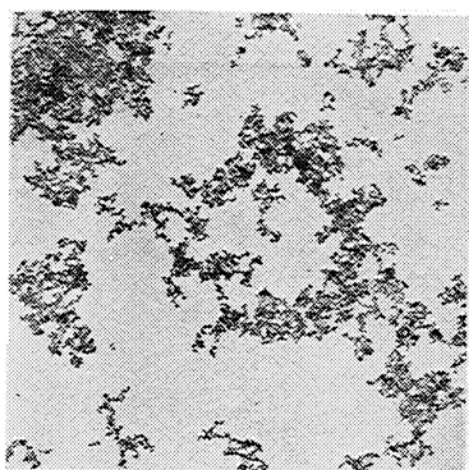


Fig. 1.—A (Nippon Oil Co. black).  
×10,000

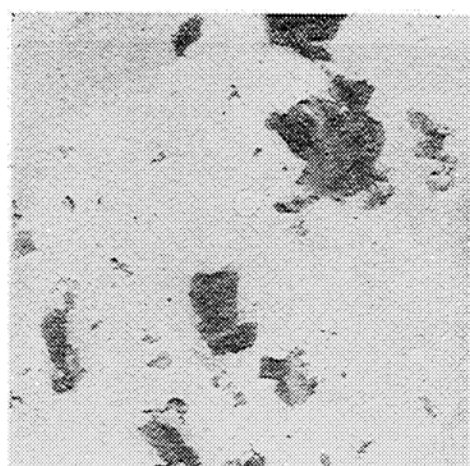


Fig. 2.—B (Great Lake pitch coke).  
×10,000

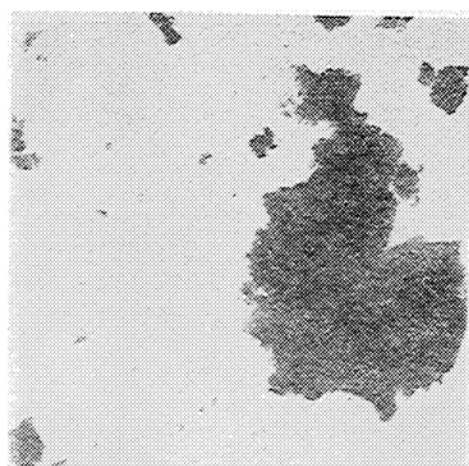


Fig. 3.—C.  
×10,000

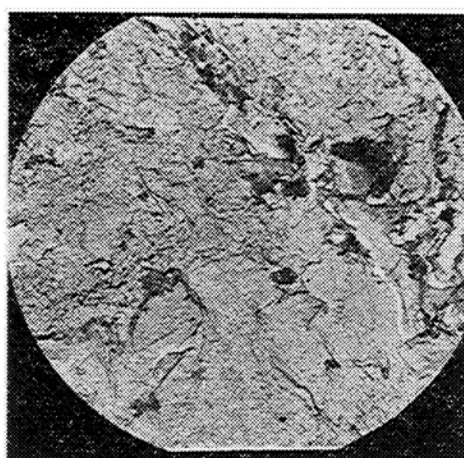


Fig. 4.—D.  
×4,200

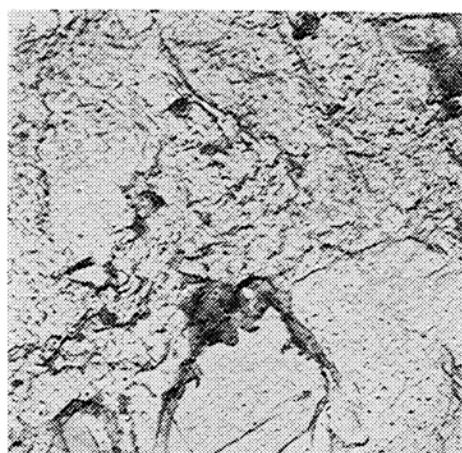
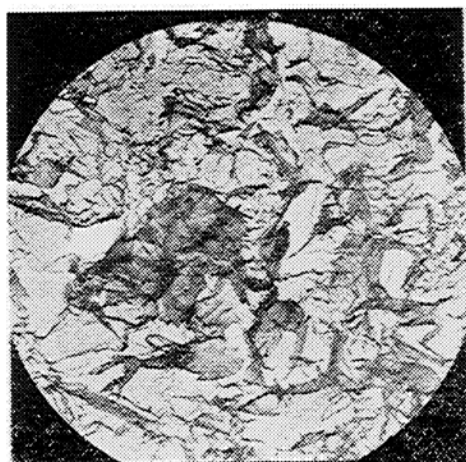
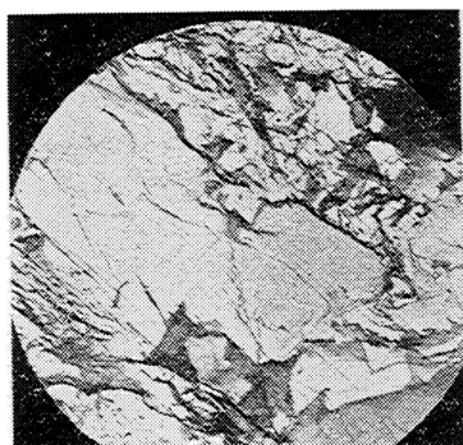
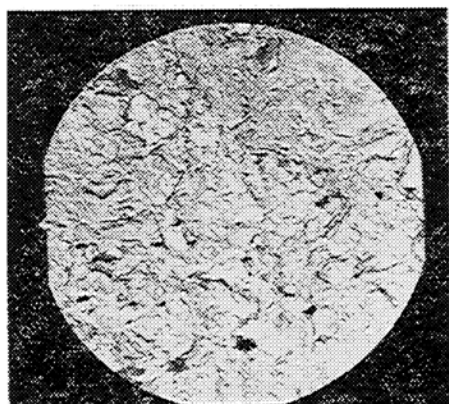
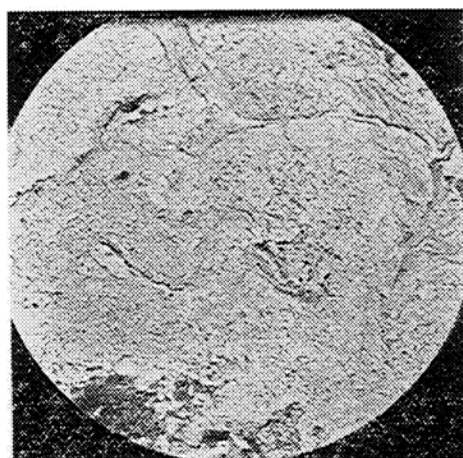
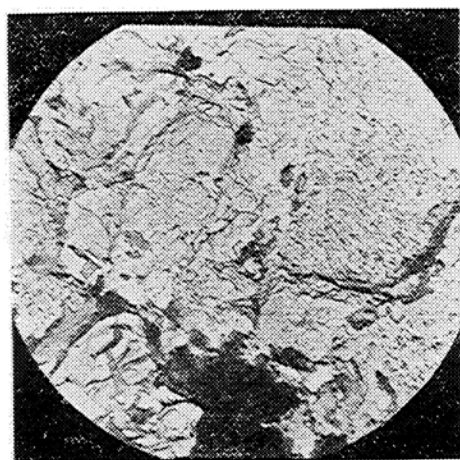


Fig. 5.—D. A part of Fig. 4.  
×10,000

Fig. 6.—E.  $\times 4,200$ Fig. 7.—E.  $\times 4,200$ Fig. 8.—F.  $\times 2,000$ Fig. 9.—I.  $\times 4,200$ Fig. 10.—I.  $\times 4,200$

were found besides flaky graphite crystals in the directly observed powdered specimens of E.

F: Main parts of crystals of F were more irregular and smaller than those of E (Fig. 8).

G: Well developed crystal surfaces were seen in larger parts of G.

H: The parts of carbon black origin and of pitch coke origin were clearly seen from an electron micrograph of the powdered specimens of H. The appearance of the powders was the same as those of C. Particles of approximately the same size as the original carbon black A linked together to aggregates.

I: A part of carbon black origin of I is shown in Fig. 9. Boundaries of particles of aggregated carbon blacks are seen. A part of pitch coke origin is shown besides a part of carbon black origin in Fig. 10.

J: The appearance of J was the same as I.

K: Well developed crystal surfaces as F were seen in replicas of K.

### Summary and Discussion

Pitch coke type brushes were found to consist of graphite crystallites having irregular forms and flat surfaces. Sizes of the graphite crystallites were very variable. Well graphitized crystals have relatively flat and smooth surfaces. Less graphitized crystals have rough

surfaces. Well-developed flat crystals were split into thin flakes and attached on replica films. Parts of small and badly developed crystallites were seen side by side with the well-developed ones.

Carbon black type brushes were found to contain aggregated parts of minute carbon particles and parts of pitch coke origin. The size of the minute particles was approximately the same as that of the original carbon black particles. The minute particles were seen as minute projections or pits on replicas, while they were observed as being linked together to aggregates in directly observed specimens.

The authors wish to express their hearty thanks to Prof. Yoneichiro Sakaki of Nagoya University for his kind advice for the manipulation of electron microscope. This research was supported by the Grant in Aid for Developmental Scientific Research of the Ministry of Education and was made under authority of Committee No. 117 (Carbon Materials for Electrical Machines) of the Japan Society for the Promotion of Scientific Research.

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