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WATERTOWN ARSENAL LABORATORIES

A TECHNIQUE FOR THE MICROSTRUCTURAL EXAMINATION OF POLYCRYSTALLINE GRAPHITES

TECHNICAL REPORT NO. WAL TR 132.5/1

BY

A. TARPINIAN

G. E. GAZZA

FEBRUARY 1959

O.O. PROJECT: TB4-004, HEAT RESISTANT MATERIALS
D/A PROJECT: 593-32-004

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Metallography, Graphite

A TECHNIQUE FOR THE MICROSTRUCTURAL EXAMINATION OF POLYCRYSTALLINE GRAPHITES

Technical Report No. WAL 132.5/1

By

A. Tarpinian

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TITLE

A TECHNIQUE FOR THE MICROSTRUCTURAL EXAMINATION OF POLYCRYSTALLINE GRAPHITES

ABSTRACT

The application of a metallographic technique for the microstructural study of polycrystalline graphite bodies is presented. The porous nature of these bodies presents a problem during the grinding and polishing operations used in their preparation for microstructural examination. These operations tend to break-down the pore walls, causing destruction and smearing of the original structures. In order to prevent this smearing a technique for impregnating the porous structure with a resin that hardens at room temperature was used to reinforce the graphite specimens. This operation showed significant improvement in polishing efficiency and preservation of microstructure. Although some structure was visible upon microscopic examination after polishing, finer detail was brought out by etching with a solution of potassium dichromate in concentrated phosphoric acid. This etchant reacted quite readily with the graphite surface, yet at a rate that was easily controlled.

A variety of photomicrographs, obtained using the techniques described, is presented and discussed. Since sufficient data on specimen materials were not available limited interpretations of the observed structures are made. Structural interpretation will be attempted after further work with specimens of known composition and fabrication.

A. Tarpinian
A. TARPINIAN
Physical Chemist

G. E. Gazza
G. E. GAZZA
Physical Metallurgist

APPROVED:

J. F. Sullivan
J. F. SULLIVAN
Director
Watertown Arsenal Laboratories

INTRODUCTION

The need for advancing graphite technology beyond the limited areas in which it had existed in prior years has been prompted by interest in this material for use as component parts of missiles and atomic reactors.

The high sublimation point, increase of strength with temperature (up to approximately 2500°C), good thermal shock resistance, low density, good machinability, together with low cost and ready availability, make graphite very attractive for use in those components of rocket engines that require resistance to very high temperatures. These advantages of graphite are, to some extent, counterbalanced by its non-uniform properties, low strength, and poor oxidation resistance.

At present, an increasing amount of effort is being expended in the development of synthetic graphites having improved oxidation resistance, higher strength, and homogeneity of properties.

An understanding of the effects of microstructure on the properties of carbon and graphite bodies is essential for the proper selection and control of the processing variables involved during fabrication. In contrast to metals the effects of microstructure on the properties of graphite materials have not been investigated sufficiently to demonstrate which features are most significant. As in the case of metals, knowledge of the microstructure would promote rapid progress in a better understanding of the material.

Background

Artificial graphites are a conglomerate of graphite particles joined together by a bridge of binder graphite. They are quite often non-uniform, and these variations have a pronounced effect on many properties. The largest effects are due to the porosity which is always present and cannot be avoided since the original coke particles used in the mix are held together with an organic binder, which gives off volatiles during the subsequent carbonizing heat treatment of the binder. Knowledge of structural details such as particle and interparticle porosity, pore and particle shapes, particle orientations and size distributions, and structural relationships between binder and particle, along with measureable properties such as strength, oxidation rate, thermal conductivity, thermal expansion, crystal structure, electrical conductivity, and texture will prove to be invaluable in the proper control of processing variables during the synthesis of graphites.

Most mechanical failures have been observed to take place through these binder bridges between particles. It is speculated that this preferential fracture through the binder is caused by a high concentration of stresses at the bridges due to the differences in thermal contraction between binder graphite and particle graphite.[1] A study of the structural characteristics of these binder bridges on a variety of graphites would conceivably lead to a better understanding of the effect of different types of binders on the structural nature of these bridges. Particle size, shape, distribution, and orientations affect the location and number of these bridges, which in turn affect the anisotropic behavior of the physical and chemical properties of the graphite bodies. Thermal and electrical conductivity is also affected by particle size, shape, distribution, and orientation, along with the size of the binder bridges. Since the thermal conductivity of graphite single crystals is greater parallel to the graphite (002) planes[2] and the crystallite alignment within the particles is such that the (002) planes are more or less parallel to the long axis of the particle,[3] differences in thermal conductivity will be found in polycrystalline graphites having different particle orientation and binder content.

The effect of surface area and binder content is very significant in the oxidation kinetics of polycrystalline graphites. It has been shown that the binder graphite oxidizes at a more rapid rate than the well-crystallized particles that it joins and that the surface area increases considerably at the beginning of oxidation due to the rapid oxidation of the binder material, causing a high oxidation rate during the removal of the more active sites provided by the binder graphite.[4]

It can be seen from the foregoing discussion that it would be advantageous to develop a method for observing the microstructures of polycrystalline graphite bodies. The primary purpose of this paper is to present preliminary studies made toward developing a satisfactory technique for the preparation and treatment of polycrystalline graphites for their subsequent microstructural examination.

POLISHING AND ETCHING STUDIES

Impregnation

The porous and brittle nature of graphite bodies presents a problem during the grinding and polishing operations used in their preparation for microstructural examination. These operations tend to break down the pore walls, causing destruction and smearing of the original structure. In order to reduce this break-down to a minimum a technique of impregnating with resins, used quite often for reinforcing powdered metal compacts prior to polishing, was used. In these studies, all specimens were impregnated with a resin mixture used by Martin and Shea[5] in their work with carbon microstructures. Etching techniques were not included in the referenced work. The impregnating resin is composed of the following components: 3 parts by weight Singapore dammar resin, 2 parts by weight high grade orange flake shellac, and 1 part by weight true Venice turpentine. The function of the resin impregnation is to strengthen the material internally by filling the open pores and thereby prevent pore wall break-down and surface flow during the polishing operation. Low viscosity, acid resistant epoxy resins are also being considered. Although none have been tried to date, a lower viscosity epoxy resin may have more penetrating ability and thereby further increase the surface strength adding to polishing efficiency and preservation of microstructure. An epoxy resin with little or no volatiles, on curing, would help eliminate any displacement of resin in the pores.

Since all materials were too dense to allow significant penetration of resin by immersion alone, the following procedure for resin impregnation was used: the resin is melted and stirred, to produce a homogeneous mixture, in an iron pot lined with aluminum foil (the foil insures easy removal of the expended resin). After the resin mixture is prepared it is allowed to cool and solidify. The specimens are then placed on the solid resin, and the pot is evacuated to approximately 200 microns of mercury. Upon achieving this pressure, the pot is heated to remelt the resin thus allowing the specimen to become submerged. At this point, the vacuum is released allowing the atmosphere into the system, thereby forcing the melted resin into the pores of the specimens.

Polishing

After the resin has hardened, the excess covering the specimen is ground off. After impregnation, the specimen is polished using the following paper and wheel abrasives in the order listed: #400 paper (wet), #600 paper (wet), 1-0 through 4-0 papers (dry), chromic oxide abrasive on rayon acetate cloth wheel (550 rpm) and Gamal on microcloth wheel (hand polished). This procedure produces a smooth, scratch-free surface. The improvement afforded by impregnation prior to polishing is well demonstrated in Figures 1 and 2. Figure 1 is a photomicrograph of a specimen polished without impregnation, and Figure 2 is a specimen polished after impregnation. Note the lack of definition in the unimpregnated specimen as compared to the impregnated one. It may be noted here that pores showing the presence of impregnant (Figure 2) are open pores. The converse is not absolutely true, however, since some impregnant may have been removed during polishing.

Figure 3 is a photomicrograph of a grade of graphite used in making jet vanes for the German V-2 rocket during World War II. The large particle size is typical of this type of graphite. Two particles joining each other at an angle are pictured here, and although the typical lamellar structure of the graphite particles is observed much of the finer detail is hidden. At this stage no distinction can be made between the original coke particles and the binder bridges resulting from the pyrolysis of the organic binder.

Etching

It is logical to assume that structural differences should exist between binder graphite and particle graphite. Although they are made up of the same element it is a well established fact that carbons do differ from one another structurally, and that these differences depend on the conditions of pyrolysis as well as the type of organic compound from which they are derived. As previously mentioned, artificial graphites are a mixture of anisotropic graphite particles bound together into a solid piece by graphite bridges formed by the original organic binder between particles. In the process of forming these bodies prior to baking, while the binder is still fluid, the coke particles tend to align themselves more or less parallel or perpendicular to the forming force depending on whether they are being extruded. or molded. The degree of this alignment depends on the size of the coke particle, along with the viscosity and amount of binder. Upon heat treatment of the *green* mix, carbonization of the binder takes place resulting in the deposition of carbon and the evolution of volatiles, creating pores and carbon bridges between particles. One can speculate here and say that there is no reason for assuming that the binder will graphitize into crystals with any preferential crystallite orientation. Thus, after heat treatment to graphitizing temperatures, there exists a situation where highly anisotropic multicrystalline graphite particles, aligned to a degree parallel to a particular direction, are surrounded and bound together with binder graphite having a random orientation of crystallites. It is this speculated difference between the two types of graphite that the authors felt would allow the use of etching agents to contrast the binder and particle microstructures. Difference in reaction rates with the etching agent would cause the selective etching, which is essential for contrast.

Since carbon is one of the more inert of materials with respect to chemical reaction with other elements and compounds, the choice of chemical reaction for etching is limited to three types: oxidation, formation of lamellar compounds, and carbide formation at high temperatues. Of the three classes of reaction, oxidation seems to be the most feasible for use in etching since the reaction product is gaseous (CO_2) and does not require removal or solution away from the underlying structure. Since wet oxidation is more easily controlled than gaseous oxidation, efforts to determine the proper reagents and conditions were directed toward preliminary studies of such chemical oxidants as potassium dichromate-phosphoric acid mixtures,[6] fuming perchloric acid, hydrogen peroxide, nital, and perchloric acid-ammonium metavanadate mixtures.[7] The potassium dichromate-phosphoric acid mixture (20 gm potassium dichromate in 100 cc of conc. phosphoric acid) showed the most promise in its ability to oxidize the graphite surfaces readily, yet at a rate which is easily controlled. The velocity of the reaction is quite slow at room temperature but is accelerated by moderate heating.

After polishing, the specimens were dipped into the hot (80-100°C) etchant (potassium dichromate-phosphoric acid) for 15-second intervals. After each interval, the specimen was metallographically examined in order to determine an optimum etching time. This time was found to be dependent on the grade of graphite being examined, as some specimens would etch much faster than others. In general, the majority of specimens examined were found to be suitably etched in an approximate time range of 30 to 60 seconds. Due to the porous nature of the material, the specimens had to be throughly washed after etching in order to remove all of the etchant from the pores. To eliminate staining effects alcohol was not used to facilitate drying as is the general practice with metallic materials.

During the process of etching, evidence of resin removal from the pores is apparent. This results in the weakening and contamination of the etchant solution after 5 or 6 specimens have been etched. As a result, fresh etchant must be used. Repolishing is not recommended after etching since most of the reinforcing resin impregnant is removed. Over-etching of the specimen may produce lamellar-type compounds, which distort and expand the graphite lattice creating areas of high stress concentration. The internal stresses within the graphite act on these areas, and large cracks within the specimen are produced. Etching rates vary considerably among different grades of graphites. A possible explanation for this may be found in the studies made by Spalaris (see Reference 4). In his studies on gaseous oxidation of graphites, he found that the binder carbon reacts more readily than the particle carbon. This may be due to the creation of higher energy areas in the binder caused by differences in crystallite alignment and/or stresses produced by differences in thermal contraction between binder and particle carbon. Bearing this fact in mind a possible explanation of the difference in etching characteristics of different commercial graphites may be the difference in the amount of binder used (active areas) in their fabrication.

DISCUSSION OF RESULTS

At present, the authors have no information about the amount of binder used in the different grades studied. Figure 4 shows quite clearly that the binder graphite, or what is assumed to be the binder graphite, does in fact react with the etchant at a faster rate than the particle graphite. It is apparent that quite a bit of attack or over-etching has taken place in the area between particles and very little etching within the particles. Figure 5 is a photomicrograph of a selected binder area. Etching was controlled so that only the binder was etched leaving the particles around it unetched. Note the typical flow characteristics which are very often observed on a macroscale after pitch has been coked.

It may be seen that differences in the etching reaction within particles also exist due to the variation of crystallite orientation within the particle. Apparently, when the edges of the crystallite layers are parallel to the surface to be etched, the reaction reveals a lamellar structure such as seen in Figure 6. This may be due to the reaction of the more active edge atoms in the layer lattice of the graphite.[8] As a result, those crystallites that are so positioned within the particles will tend to etch faster and reveal their lamellar structure. It should be mentioned here that in making an analogy of polycrystalline graphite structures with polycrystalline metallic structures, one must consider the binder bridges between particles as another phase and the crystallites within particles as individual grains.

On the unetched specimens, some structure is visible (see Figure 3), but this is only the larger cracks and pores made visible by the erosive action of the abrasive particle during the polishing operation. The smaller discontinuities may only be observed after the specimen has been etched (as shown in Figure 7). Sometimes, selective erosion of the abrasive particles on the weakest surface areas may be seen (as shown in Figure 8). The graphite particle, under polarized light, appears to consist of a number of packets before etching. The packet boundaries are actually only portions of the lamellar structure that is brought out after etching.

From the differences in etching rate between particle and binder it may be concluded that etching for microstructural examination of both binder and particle, using the potassium dichromate-phosphoric acid etchant, is not feasible. One must etch for the particular phase of interest.

Figure 9 is a photomicrograph of a laboratory graphite made by the authors. The specimen was formulated by mixing 70 wt % petroleum coke with 30 wt % phenolic plastic.* The petroleum coke had the following particle size distribution as determined by U. S. Standard Sieves: 7 wt % (-100 + 200 mesh), 33 wt % (-200 + 325 mesh), and 60 wt % (-325 mesh). After mixing, specimens were formed by molding cold. After forming and baking, the specimen was graphitized at 2800°C. The distribution of porosity appears to be fairly homogeneous. The values obtained from particle size measurements from the photomicrographs were in approximate agreement with the particle size distribution of coke used in the fabrication of the specimen. These results indicate, to some extent, the reliability of the polishing and etching technique.

Porosity, on which many of the physical properties are dependent, may be seen to exist as microporosity and macroporosity. The microporosity may be seen in the lamellar structure of the particles. Macroporosity which is formed during the volatilization of gases in the baking stage is found in the binder areas and has a wide range of size distribution. This porosity is not always uniform throughout the material. Quite frequently, voids follow the contour of large particles as seen in Figures 8 and 10. This probably occurs as a result of thermal contraction during cooling. This type of macrovoid would be a source of higher stress concentration than that formed during the volatilization of gases, and would be more detrimental to the strength of the material. It is known that particle shapes are dependent on the grinding and milling operation performed on them, whether a hard coke or a soft coke was used and the state they are in while they are being ground or milled. In accordance with E. A. Kmetko[9] it may be stated that soft cokes break up in grinding into platlets. The microcrystals in the platlets are oriented with their basal planes more or less parallel to the lamellar planes. Hard cokes break up into jagged, roughly equiaxed particles, characteristic of amorphous substances. It can be seen from the photomicrographs that the variables involved have produced a variety of particle sizes and shapes. Figures 11 through 18 are photomicrographs of graphites prepared using the techniques described. The purpose of presenting these is only to illustrate a variety of structures found between different grades and also within the same grade. Appropriate magnifications were chosen that would best illustrate the particular type of structure being presented.

**BIS 2600, made by Bakelite Co., Division of Union Carbide Corp.*

Under consideration is the etching of polished graphite by ion bombardment (cathodic etching). Topographical differences that would develop on the graphite surface would denote differences in hardness due to orientation and internal stresses. Unlike chemical etchants, a clean, stain-free surface would be produced. Since ion bombardment etches only by differences in hardness on the surface of a specimen whereas chemicals etch by the chemical reactivity of each phase, the ion bombardment may not etch as selectively on graphite as do chemical etchants.

CONCLUSIONS

The purpose of this investigation was to determine whether a suitable technique for studying polycrystalline graphite microstructures could be developed. The interpretation of these structures was not the prime objective. Interpretations will be more easily made using specimens of known particle size distribution, type of binder, type of coke, and binder-to-coke ratio. The importance of microstructural examination cannot be stressed enough, especially in relation to carbon and graphite technology where the methods of investigation seem to be limited mostly to the deductive type where measured physical properties are compared with processing variables. This technique can be slow and fruitless because of the multitude of variables involved.

The authors feel that the work presented here is a sound beginning toward the application of a tool that has proven to be instrumental in advancing the technology of other materials.

ACKNOWLEDGMENT

The authors would like to acknowledge the assistance of Mr. A. P. Levitt, Chief, High Temperature Branch, Watertown Arsenal Laboratories, under whose direction the project was carried out.

FIGURE 1

COMMERCIAL GRAPHITE (ELECTRODE GRADE) - SAMPLE A
POLISHED WITHOUT IMPREGNATION (UNETCHED)
MAGNIFICATION - 100X

FIGURE 2

COMMERCIAL GRAPHITE (ELECTRODE GRADE) - SAMPLE A
IMPREGNATED AND POLISHED (UNETCHED)
MAGNIFICATION - 100X

Resin filled pore indicated by arrow. Empty pore indicated by circle.

WTN.630 - 16,648 A

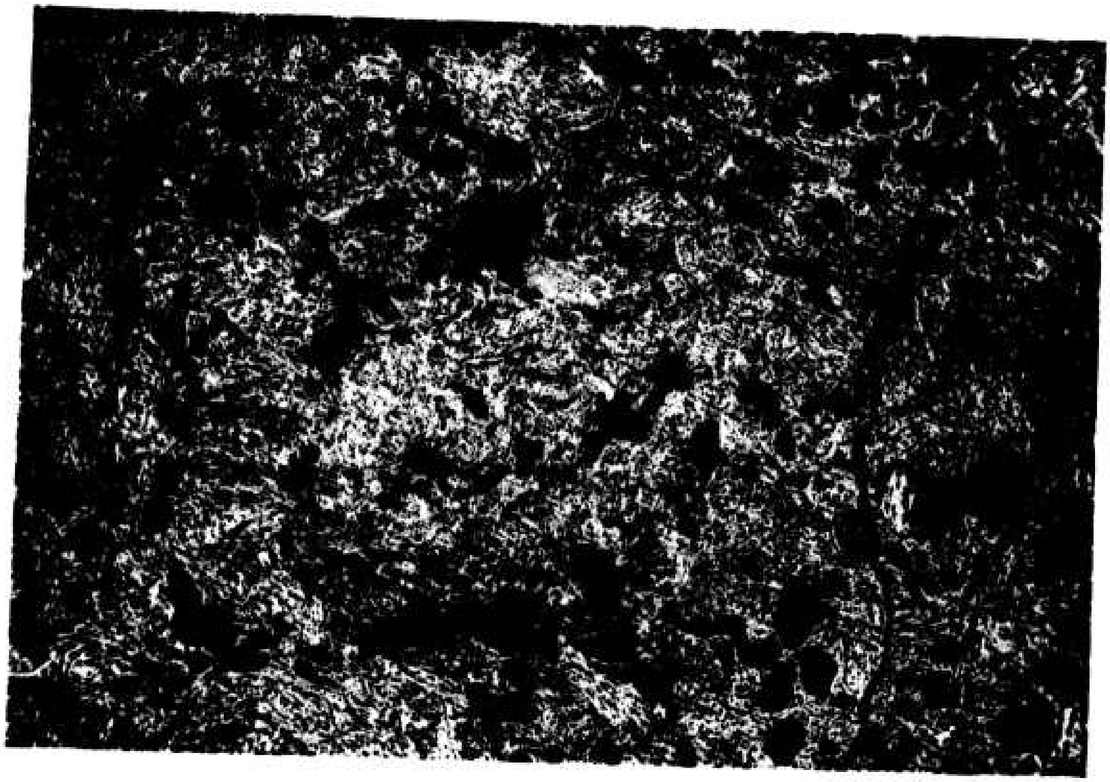

FIGURE 1

Wtn. 639-16,648

FIGURE 2

FIGURE 3

GERMAN V-2 GRAPHITE - SAMPLE B
IMPREGNATED AND POLISHED (UNETCHED)
MAGNIFICATION - 100X

FIGURE 4

COMMERCIAL GRAPHITE (ELECTRODE GRADE) - SAMPLE C
IMPREGNATED AND POLISHED
ETCHED WITH PHOSPHORIC ACID - POTASSIUM DICHROMATE SOLUTION
MAGNIFICATION - 1000X

Arrow indicates typical etched-out binder area.

WTN.630 - 16,647 A

FIGURE 3

Wtn. 639-16,647

FIGURE 4

FIGURE 5

GRAPHITE MADE BY THE AUTHORS
TYPICAL BINDER AREA
IMPREGNATED AND POLISHED
ETCHED WITH PHOSPHORIC ACID - POTASSIUM DICHROMATE SOLUTION
MAGNIFICATION - 1000X

FIGURE 6

GERMAN JET VANE GRAPHITE - SAMPLE D
IMPREGNATED AND POLISHED
ETCHED WITH PHOSPHORIC ACID - POTASSIUM DICHROMATE SOLUTION
MAGNIFICATION - 1000X

Note difference in etching characteristics between particles.
Particle on left exhibiting typical lamellar structure of graphite.

WTN.639 - 16,646 A

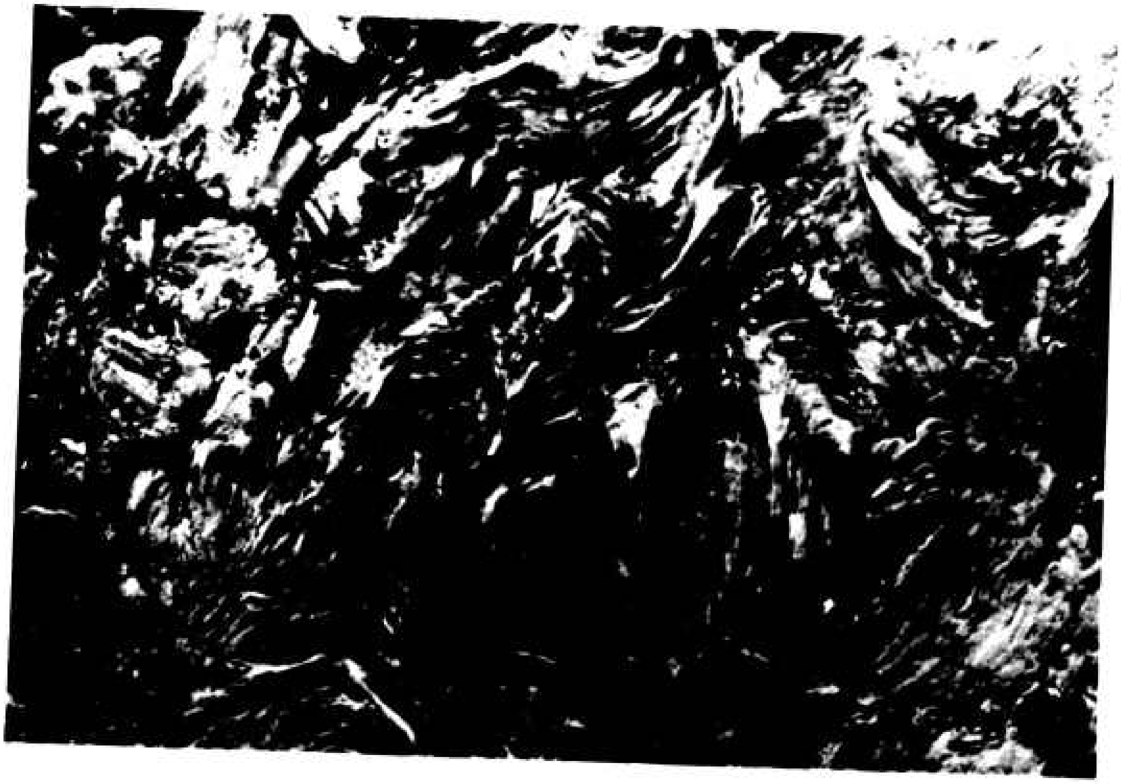

FIGURE 5

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FIGURE 6

FIGURE 7

GERMAN JET VANE GRAPHITE - SAMPLE E
IMPREGNATED AND POLISHED
ETCHED WITH PHOSPHORIC ACID - POTASSIUM DICHROMATE SOLUTION
MAGNIFICATION - 100X

FIGURE 8

GERMAN JET VANE GRAPHITE - SAMPLE E
IMPREGNATED AND POLISHED
UNETCHED - POLARIZED LIGHT
MAGNIFICATION - 750X

Note void following contour of the large particle at the left. Packet formation is shown by light and dark areas within the particle that follow the curvature of the particle.

WTN.639 - 16,645 A

FIGURE 7

Wtn. 639-16,645

FIGURE 8

FIGURE 9

GRAPHITE MADE BY THE AUTHORS
IMPREGNATED AND POLISHED
ETCHED WITH PHOSPHORIC ACID - POTASSIUM DICHROMATE SOLUTION
MAGNIFICATION - 100X

FIGURE 10

GERMAN JET VANE GRAPHITE - SAMPLE D
IMPREGNATED AND POLISHED
ETCHED WITH PHOSPHORIC ACID - POTASSIUM DICHROMATE SOLUTION
MAGNIFICATION - 100X

WTN.639 - 16,644 A

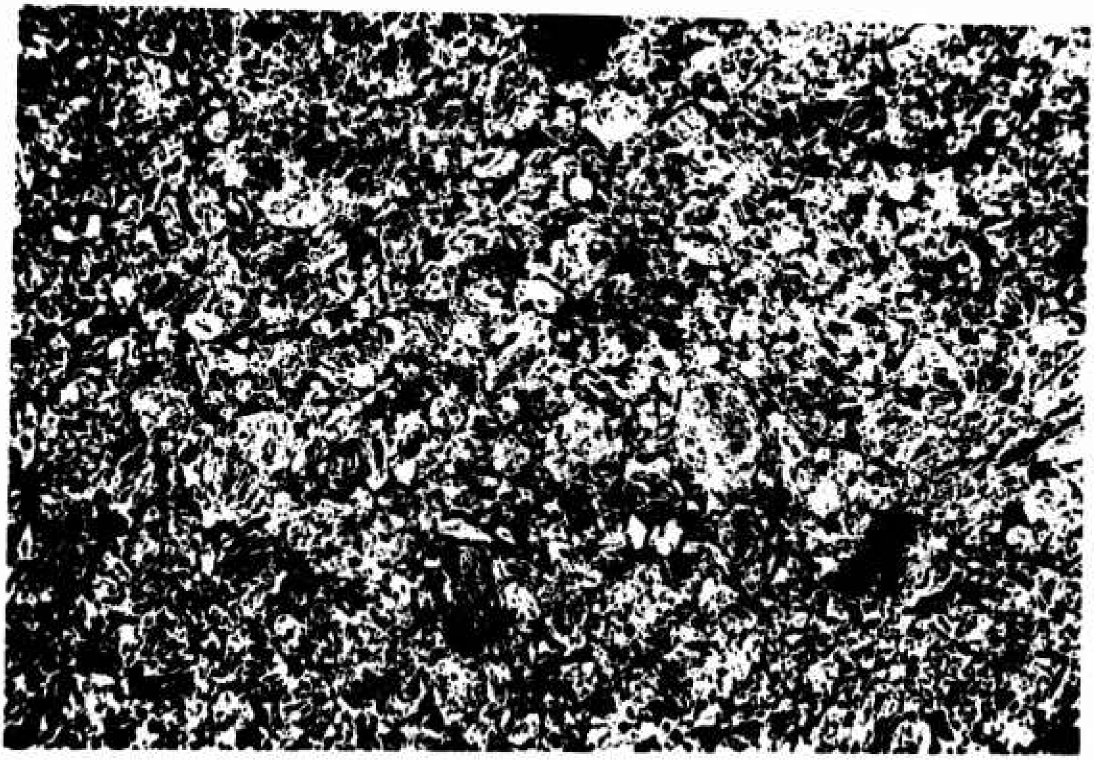

FIGURE 9

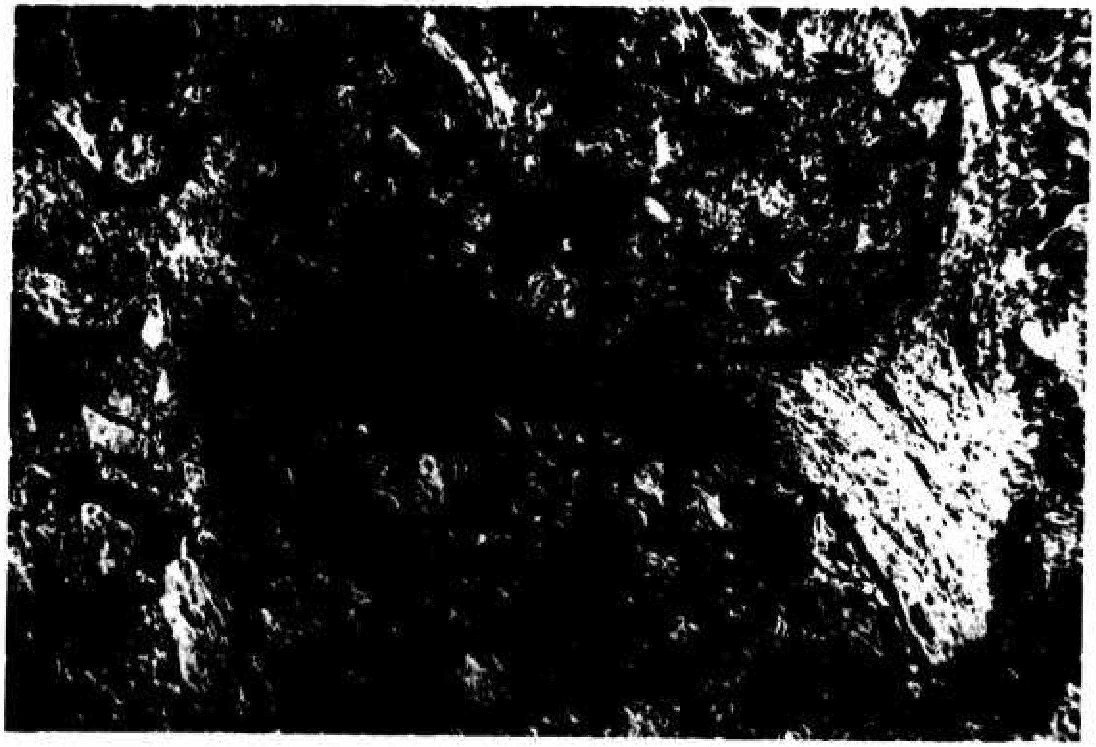

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FIGURE 10

FIGURE 11

GERMAN JET VANE GRAPHITE - SAMPLE D
IMPREGNATED AND POLISHED
ETCHED WITH POTASSIUM DICHROMATE - PHOSPHORIC ACID SOLUTION
MAGNIFICATION - 100X

FIGURE 12

GERMAN JET VANE GRAPHITE - SAMPLE D
IMPREGNATED AND POLISHED
ETCHED WITH PHOSPHORIC ACID - POTASSIUM DICHROMATE SOLUTION
MAGNIFICATION - 100X

WTN.639 - 16,643 A

FIGURE 11

Wtn. 639-16,643

FIGURE 12

FIGURE 13

GERMAN JET VANE GRAPHITE - SAMPLE E
IMPREGNATED AND POLISHED
ETCHED WITH PHOSPHORIC ACID - POTASSIUM DICHROMATE SOLUTION
MAGNIFICATION - 100X

FIGURE 14

GERMAN JET VANE GRAPHITE - SAMPLE F
IMPREGNATED AND POLISHED
ETCHED WITH POTASSIUM DICHROMATE - PHOSPHORIC ACID SOLUTION
MAGNIFICATION - 100X

WTN.639 - 16,642 A

FIGURE 13

Wtn. 639-16,642

FIGURE 14

FIGURE 15

COMMERCIAL GRAPHITE (ELECTRODE GRADE) - SAMPLE G
IMPREGNATED AND POLISHED
ETCHED WITH POTASSIUM DICHROMATE - PHOSPHORIC ACID SOLUTION
MAGNIFICATION - 200X

FIGURE 16

COMMERCIAL GRAPHITE (ELECTRODE GRADE) - SAMPLE H
IMPREGNATED AND POLISHED
ETCHED WITH POTASSIUM DICHROMATE - PHOSPHORIC ACID SOLUTION
MAGNIFICATION - 300X

FIGURE 15

Wtn. 639-16,641

FIGURE 16

FIGURE 17

COMMERCIAL GRAPHITE (ELECTRODE GRADE) - SAMPLE I
IMPREGNATED AND POLISHED
ETCHED WITH POTASSIUM DICHROMATE - PHOSPHORIC ACID SOLUTION
MAGNIFICATION - 200X

FIGURE 18

COMMERCIAL GRAPHITE (ELECTRODE GRADE) - SAMPLE J
IMPREGNATED AND POLISHED
ETCHED WITH POTASSIUM DICHROMATE - PHOSPHORIC ACID SOLUTION
MAGNIFICATION - 500X

Note discontinuity of lamellar structure indicated by arrow.

WTN.639 - 16,640 A

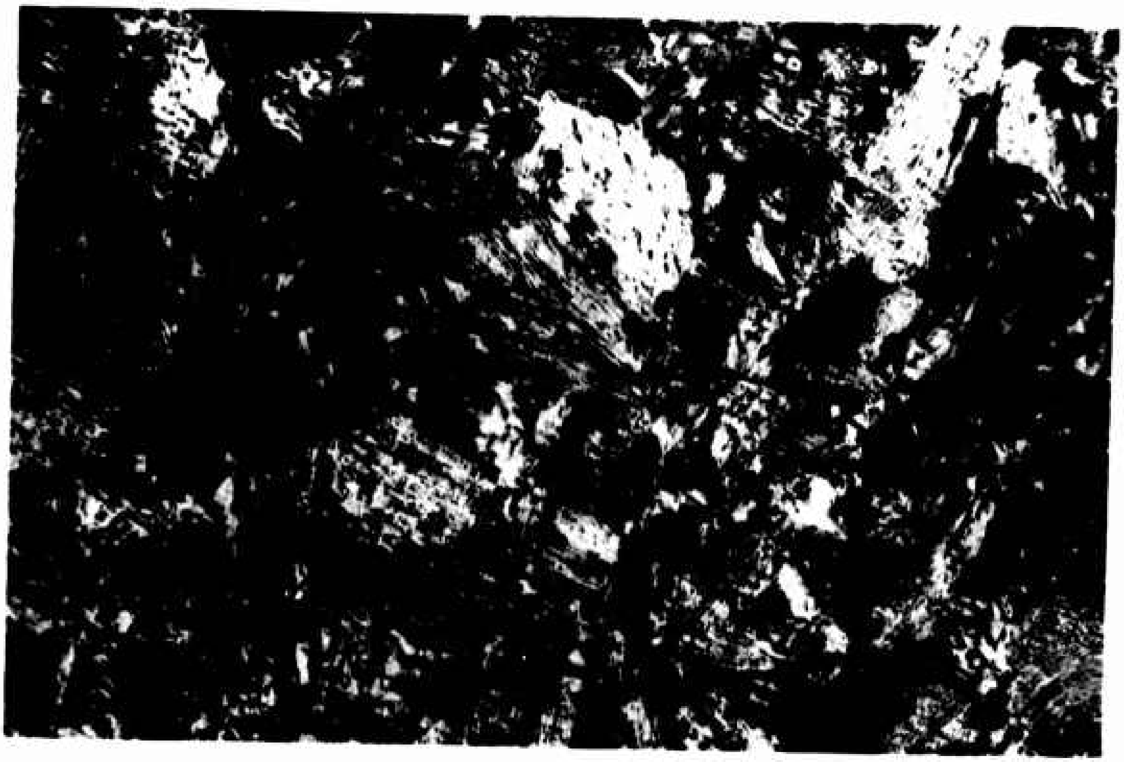
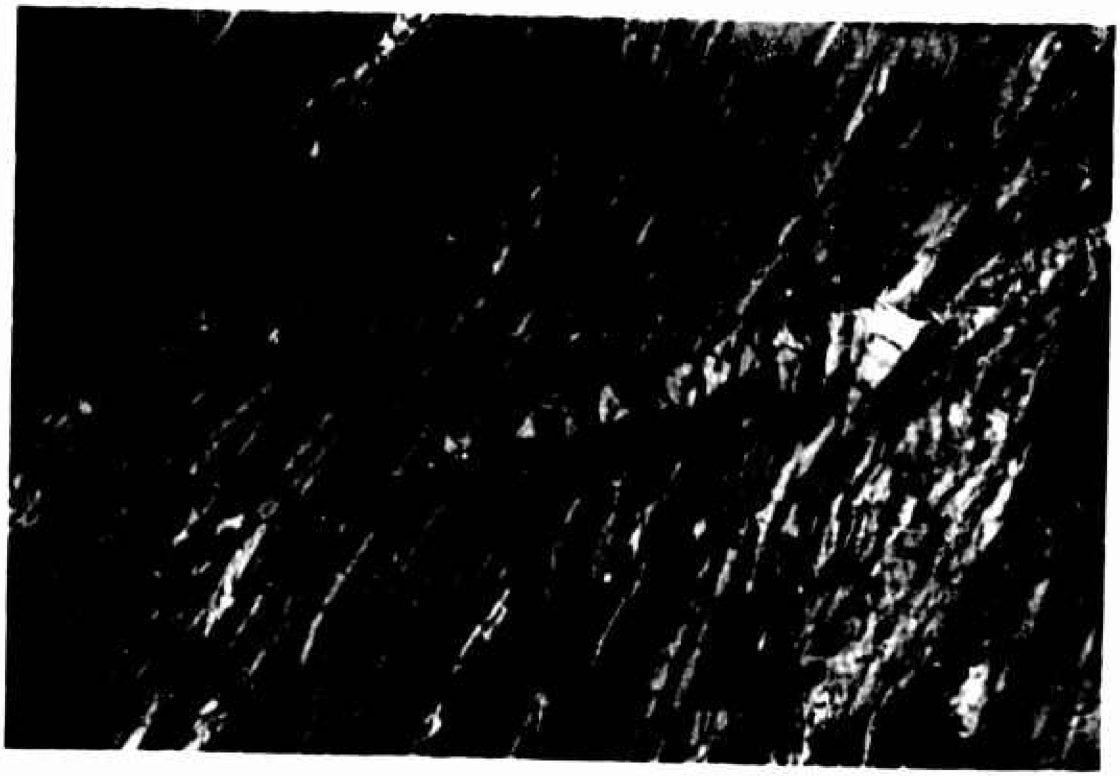


FIGURE 17



Wtn. 639-16,640

FIGURE 18

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AD_____________ Accession No._____________
Watertown Arsenal Laboratories, Watertown 72, Mass.
A TECHNIQUE FOR THE MICROSTRUCTURAL EXAMINATION OF POLYCRYSTALLINE GRAPHITES - A. Tarpinian and G. E. Gazza

Report No. WAL TR 132.5/1, Feb 1959, 9 pp - illus, DA Proj 593-32-004, OO Proj TB4-004, Unclassified Report

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