FOREWORD

This work was conducted by the National Carbon Company, a Division of Union Carbide Corporation, under USAF Contract No. AF 33 (616)-6915. This contract was initiated under Project No. 7350 "Refractory Inorganic Non-Metallic Materials," Task No. 735002 "Refractory Inorganic Non-Metallic Materials: Graphitic;" Project No. 7381 "Materials Application," Task No. 738102 "Materials Processes;" and Project No. 7-817 "Process Development for Graphite Materials." The work was administrated under the direction of the Directorate of Materials and Processes, Deputy for Technology, Aeronautical Systems Division, with Captain R. H. Wilson, L. J. Conlon and W. P. Conrardy acting as Project Engineers.

Work under this contract has been in progress since May 1, 1960. The work covered in this report was conducted at the Advanced Materials Laboratory of National Carbon Company, located at Lawrenceburg, Tennessee, under the direction of Mr. R. M. Bushong, Director of the Advanced Materials Project and of Mr. R. C. Stroup, Manager of the Advanced Materials Laboratory. This report covers work conducted from May 1, 1960 through November 30, 1962.

This is the twenty-second of a series of volumes of WADD Technical Report 61-72 prepared to describe various phases of the work. Other volumes of this series are:

Volume I - Observations by Electron Microscopy of Dislocations in Graphite, by R. Sprague.

Volume II - Applications of Anisotropic Elastic Continuum Theory to Dislocations in Graphite, by G. B. Spence.

Volume III - Decoration of Dislocations and Low Angle Grain Boundaries in Graphite Single Crystal, by R. Bacon and R. Sprague.

Volume IV - Adaptation of Radiographic Principles to the Quality Control of Graphite, by R. W. Wallouch.

Volume V - Analysis of Creep and Recovery Curves for ATJ Graphite, by E. J. Seldin and R. N. Draper.

Volume VI - Creep of Carbons and Graphites in Flexure at High Temperatures, by E. J. Seldin.

Volume VII - High Density Recrystallized Graphite by Hot-Forming, by E. A. Neel, A. A. Kellar and K. J. Zeitsch.

Volume VIII	Electron Spin Resonance in Polycrystalline Graphite, by L. Singer and G. Wagoner.
Volume IX	Fabrication and Properties of Carbonized Cloth Composites, by W. C. Beasley and E. L. Piper.
Volume X	Thermal Reactivity of Aromatic Hydrocarbons, by I. C. Lewis and T. Edstrom.
Volume XI	Characterization of Binders Used in Fabrication of Graphite Bodies, by E. de Ruiter, A. Halleux, V. Sandor, and H. Tschamler.
Volume XII	Development of an Improved Large Diameter Fine-Grain Graphite for Aerospace Applications, by C. W. Waters and E. L. Piper.
Volume XIII	Development of a Fine-Grain Isotropic Graphite for Structural and Substrate Applications, by R. A. Howard and E. L. Piper.
Volume XIV	Study of High Temperature Tensile Properties of ZTA Grade Graphite, by R. M. Hale and W. M. Fassell.
Volume XV	Alumina-Condensed Furfuryl Alcohol Resins, by C. W. Boquist, E. R. Nielsen, H. J. O'Neil and R. E. Putcher-Armour Research Foundation.
Volume XVI	An Electron Spin Resonance Study of Thermal Decomposition Reactions of Organic Compounds, by L. S. Singer and I. C. Lewis.
Volume XVII	Radiography of Carbon and Graphite, by T. C. Furnas, Jr. and M. R. Rosumny.
Volume XVIII	High Temperature Tensile Creep of Graphite, by E. J. Seldin.
Volume XIX	Thermal Stresses in Anisotropic Hollow Cylinders, by Tu-Lung Weng.
Volume XX	The Electric and Magnetic Properties of Pyrolytic Graphite, by G. Wagoner and B. H. Eckstein.
Volume XXI	An Electron Spin Resonance Study of Thermal Decomposition Reactions of Organic Compounds, by L. S. Singer and I. C. Lewis.

ABSTRACT

Photomicrographic techniques have been used to examine the microstructure of the many grades of formed carbon and graphite developed under Contract No. AF 33(616)-6915. These techniques have aided in the study of these grades and in predicting or understanding their behavior in various applications. Photomicrography has allowed identification of various materials such as cokes, graphites, pitch, etc., within the formed article and has permitted a study of the distribution of these materials. The effect of impregnation of the graphite with pitches and resins can be more clearly defined by careful photomicrographic examination. Standard metallographic equipment can be used for such studies but preparation of the carbon and graphite requires special techniques. Mounting methods to prevent smearing or chipping of the samples during preparation and to obtain very highly polished surfaces are discussed in this report. Also described are various techniques of lighting to aid in revealing and interpreting microstructure, and the different types of photographic film, plates, developers and printing papers required for showing pertinent details in the final photomicrograph.

This report has been reviewed and is approved.

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1. INTRODUCTION

The extensive use of carbon and graphite in diverse industrial and military applications is due to many desirable physical and chemical properties over a wide range of environmental conditions. Careful selection of raw materials and manufacturing processes permit the designing of carbon and graphites having properties in ranges desired for specific applications. A knowledge of the microstructure of the formed article is very beneficial in predicting and understanding the behavior of graphites under various environmental conditions and also helps in the development of new grades. Useful information concerning microstructure may be deduced from particle size measurements on raw materials, particularly filler materials, and from porosimetry measurements on the completed product. More direct and detailed information can be obtained by optical microscopy. With sufficient experience it is possible to identify types of raw materials, particle size and shape, and the effects of manufacturing processes on microstructure. Because carbon and graphite are soft and porous materials, they require different methods of preparation than do the metals and ceramics. The purpose of this report is to describe the special mounting procedures as well as various lighting and photographic techniques used during the contract effort for effective optical microscopic and photomicroscopic examination of carbon and graphite. Other studies concerned with specific metallographic techniques have been published.(1,2,3,4,5)

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2. SAMPLE PREPARATION

A flat specimen, free from surface scratches, is necessary for good metallographic examination and subsequent photomicrographs of good definition. If the specimen is not properly prepared, its superficial structure may appear entirely different from that which is truly present. Microscopic examination of a poorly prepared specimen can obviously lead to erroneous conclusions. Since most carbonaceous materials of structural interest are quite soft and porous, they must be impregnated with a material capable of preventing the tearing of particles from the surface during grinding and polishing, otherwise the surface will not only be pitted but loose particles will smear and distort the underlying structure.

2.1. Sampling and Mounting

A representative sample of the material to be examined, cut in the form of a one-half to three-fourths-inch cube, or a cylinder of similar dimensions, is a convenient specimen size. The specimen is placed, with the surface to be examined face down, in a 25-mm diameter by 95-mm long flat-bottomed glass vial and covered with monomeric styrene to a depth of approximately one inch. The vial is placed in a dessicator and maintained under a vacuum of 15 - 30 mm of mercury for one hour, or until air bubbles no longer escape from the specimen. Under vacuum, the styrene fills the voids vacated by the air. The styrene is polymerized by placing the vial in a 105° - 130°C oven for a period of three days. Above 130°C there is danger of bubble formation in the partially polymerized styrene and these, like any unfilled pores, may appear on the surface to be examined making the sample almost impossible to polish due to carry-over of polishing compound from one step to the next. Three days at 105° - 130°C insures complete polymerization so that the styrene will not smear during grinding and polishing. The styrene is sufficiently transparent after polishing to allow limited visual penetration into pores and voids.

Figures 1 and 2 illustrate the importance of styrene impregnation. Both photomicrographs are of the same material, but only the sample in Figure 1 was impregnated. In Figure 1 the binder coke (areas of porous or spongy material) is clearly visible but this coke has been removed while grinding and polishing the unimpregnated sample (Figure 2), leaving dark voids in its place.

The importance of the styrene impregnation is further emphasized at higher magnifications. Figure 3 is a photomicrograph taken at 250 X magnification of a graphite sample which was impregnated with styrene. The microstructure of the individual graphite particles is shown in fine detail. Figure 4 shows the same type of graphite at the same magnification as in Figure 3 but without the aid of styrene impregnation. It is very

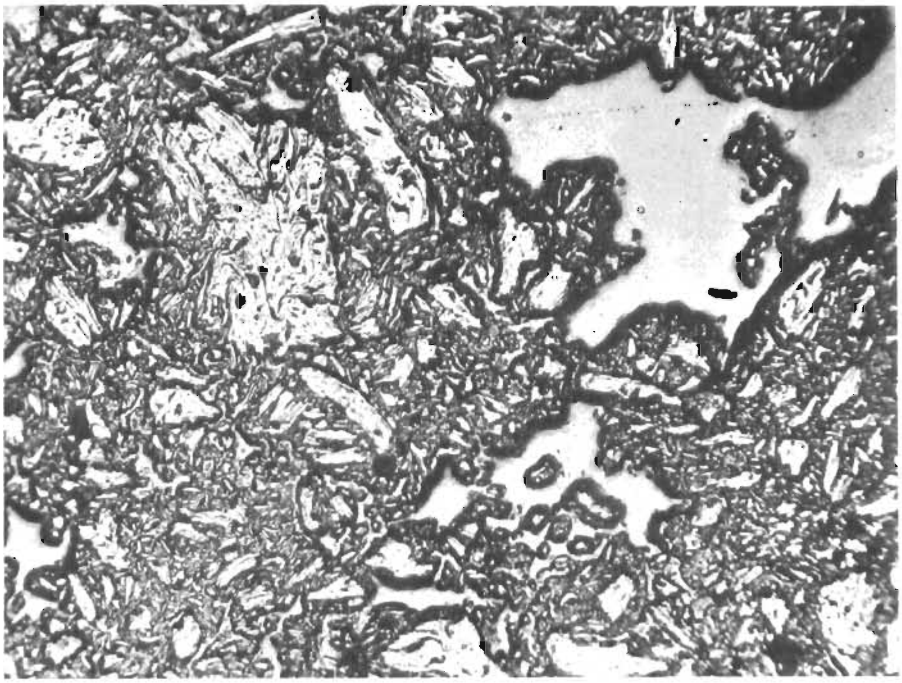

Figure 1. Coke-Pitch Material With Styrene Impregnation, 100 X.

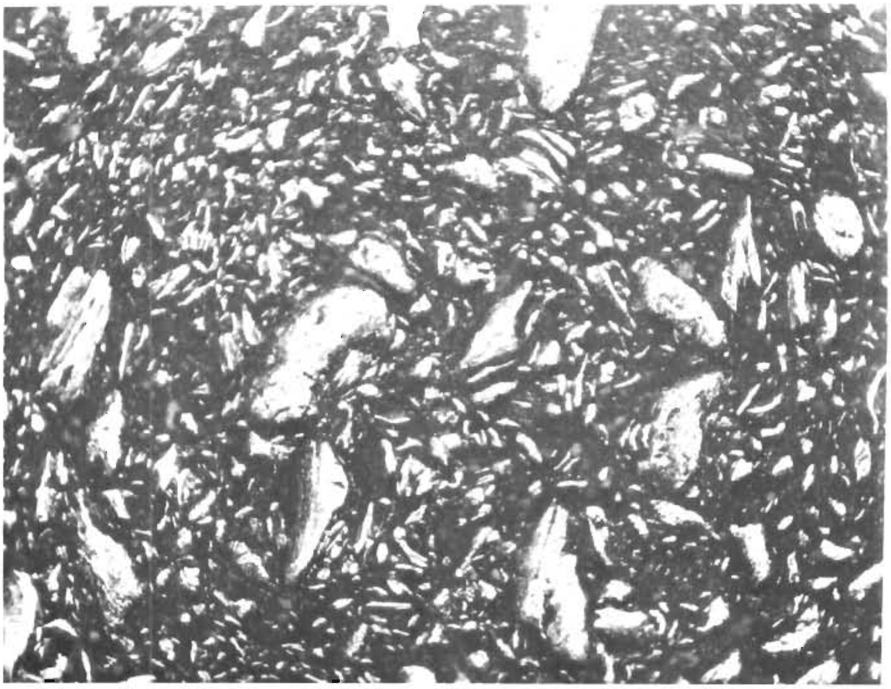

Figure 2. Coke-Pitch Material Without Styrene Impregnation, 100 X.

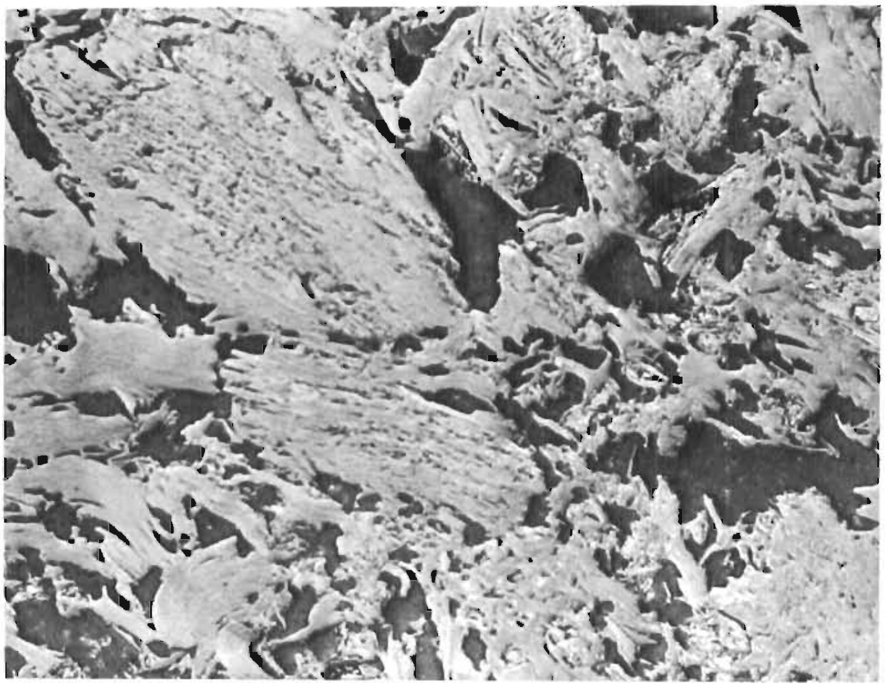

Figure 3. Graphite Sample With Styrene Impregnation, 250 X.

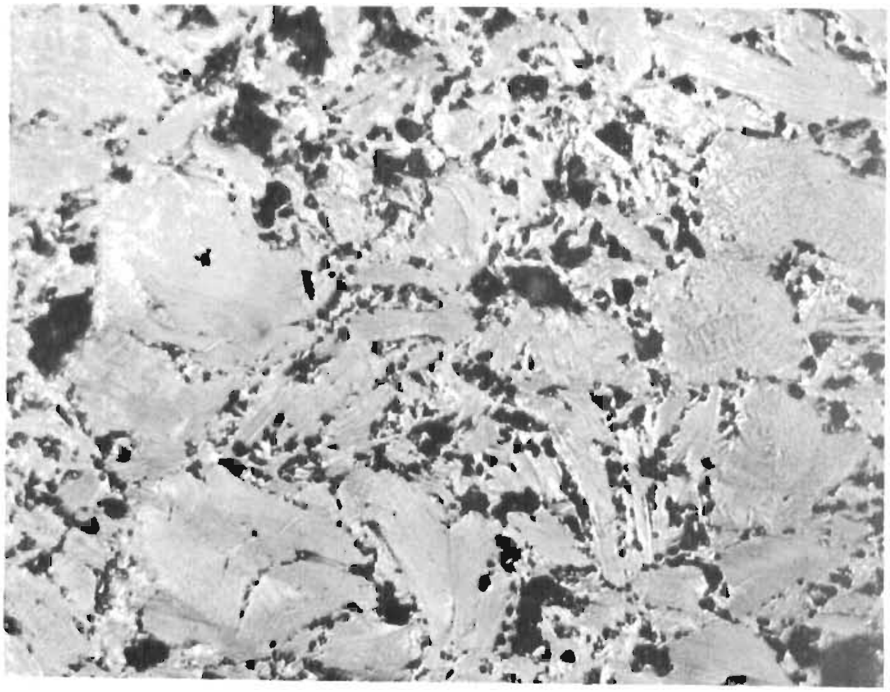

Figure 4. Graphite Sample Without Styrene Impregnation, 250 X.

evident that the microstructural details have been obscured by glazing or smearing of the surface during polishing.

Polymerized styrene is difficult to polish without chipping and the resulting chips frequently scratch the specimen surface. Molding the styrene-impregnated specimen in Lucite will prevent these fractures. Molding is accomplished at a pressure of about 4000 lbs./in.^{2} and temperatures of 120 - 150°C using a No. 1330 Buehler speed press designed specifically for mounting metallographic specimens in plastic. When properly molded the Lucite is transparent, which suggests a simple method of permanently identifying specimens. An identification number pasted on the rear surface of the polystyrene, prior to final molding in Lucite, can neither be destroyed nor lost. This method is effective only if the molded Lucite is clear. Molding at too high a temperature or removing the specimen from the mold above 75°C results in the Lucite becoming opaque (see Figure 5).

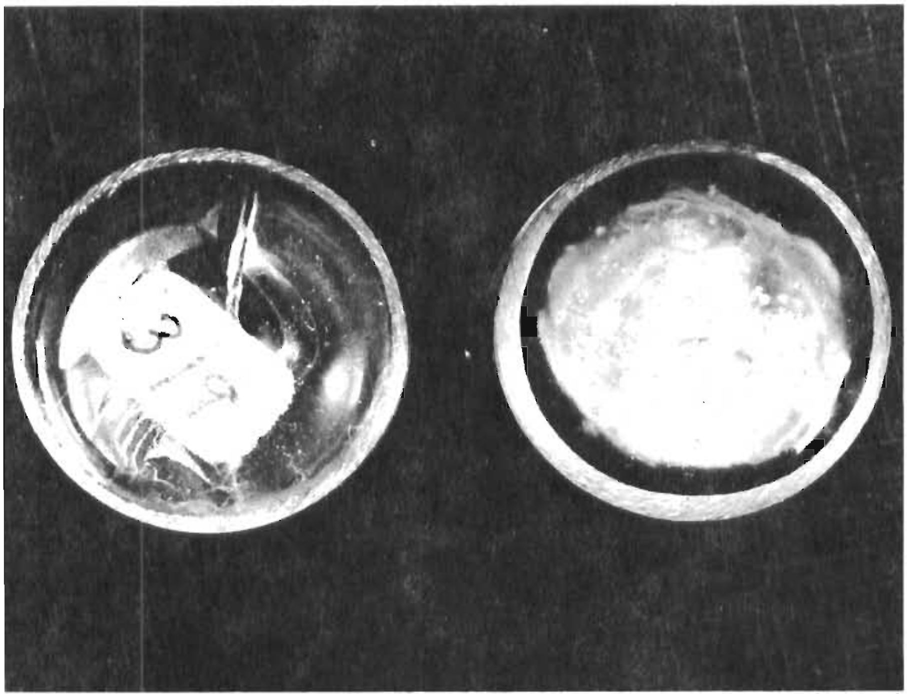

Figure 5. Polystyrene-Mounted Specimen Properly Molded (Left) and Improperly Molded (Right) in Lucite, 2 X.

The described mounting procedure is satisfactory for practically all carbonaceous materials including particles, fibers, cloth, molded and extruded pieces, etc., although there are isolated cases in which the method is either not feasible or is not desirable. Formed but unbaked samples of bulk carbon or graphite, containing a low melting point pitch, are an example of the former. Such specimens may be impregnated with monomeric styrene and mounted in Lucite providing the reaction temperature is reduced and the time is increased. Specimens that bubble at low temperatures may require special mounting materials. Resins, which can be made to polymerize at or near room temperature with the addition of a chemical catalyst, are available and may be used for mounting. The resin-catalyst mixture is cast around the specimen using a glass vial as a mold without the aid of pressure and samples are ready for grinding in thirty minutes. The quick-mounting material may be used in place of styrene and Lucite on all specimen types in the interest of expediency but is recommended for visual examination only. When photomicrographs are desired, the more laborious styrene-Lucite technique is recommended.

2.2. Grinding

Tool marks and zones of particle deformation are produced when the specimen is cut from the larger piece of material and these must be removed by many grinding and polishing stages before the true microstructure is revealed. Each step of grinding and polishing produces its own deformed layer and, therefore, each step employs a finer abrasive to remove the previous layer. Successively thinner layers of deformation are produced in this way until the final polishing step leaves a deformed layer so thin that it will not appreciably obscure the true microstructure.

Grinding is carried out on three diamond wheels which have a permanently-mounted abrasive and can be used repeatedly with a minimum of care and maintenance. The diamond wheels are especially useful for the preparation of carbonaceous samples for metallographic examination because of the variation of hardness and abrasiveness in different grades of material. Diamond sizes on these three wheels are 100 grit for coarse, 320 grit for intermediate and 500 grit for fine grinding.

Figure 6 illustrates the unground surface of a typical graphite specimen after it has been permanently mounted. The graphite is completely covered by a thin layer of Lucite making the specimen difficult to distinguish from the surrounding medium. Coarse grinding removes the plastic coating from the surface of the specimen, as illustrated in Figure 7. The mount edges are beveled on the wheel prior to coarse grinding to prevent gouging the adhesive which holds the diamonds to the wheel. Rough grinding is completed when all unwanted plastic materials are removed and the specimen surface is flat. Before proceeding from one grinding step to another, it is necessary to thoroughly wash the sample surface to remove any foreign material or loose particles.

The intermediate grinding stage removes the coarse scratches introduced during rough grinding and Figure 8 shows the sample surface after completion of the intermediate stage. Practically all of the coarse scratches have been removed and replaced by finer scratches. The sample surface after fine grinding is illustrated in Figure 9. The scratches introduced from the intermediate grinding step have been removed and replaced by still finer scratches.

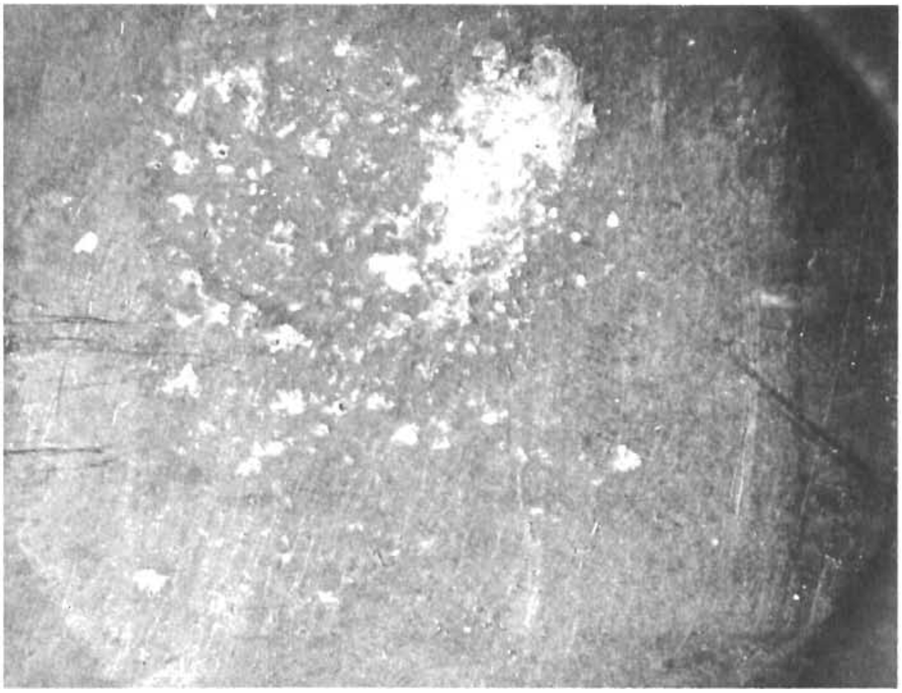

Figure 6. Mounted Graphite Specimen Before Grinding, 7X.

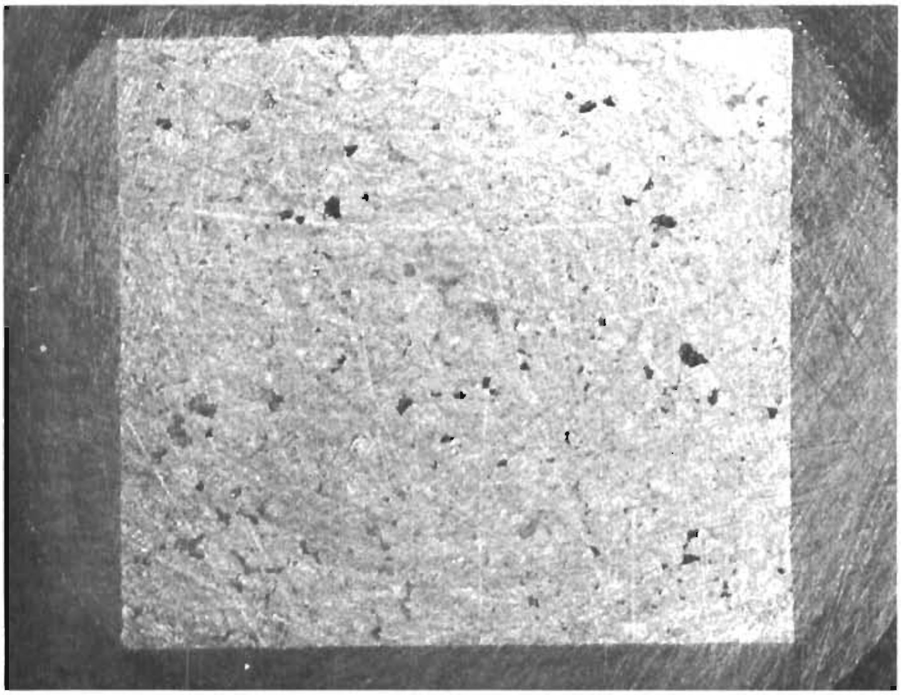

Figure 7. Graphite Specimen Surface After Grinding on 100-Grit Diamond Wheel, 7X.

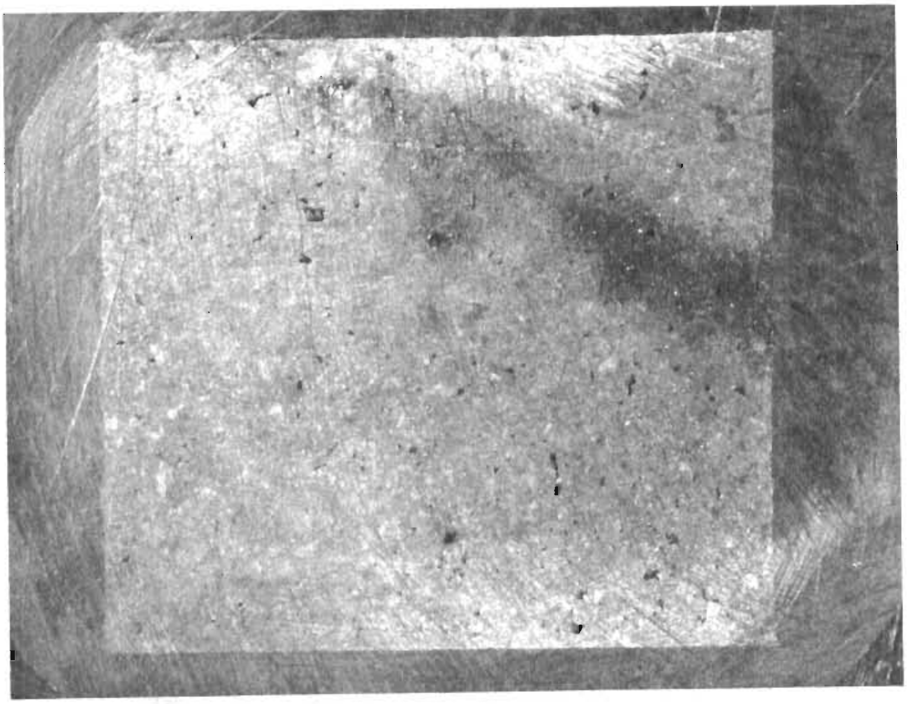

Figure 8. Graphite Specimen Surface After Grinding on 320-Grit Diamond Wheel, 7 X.

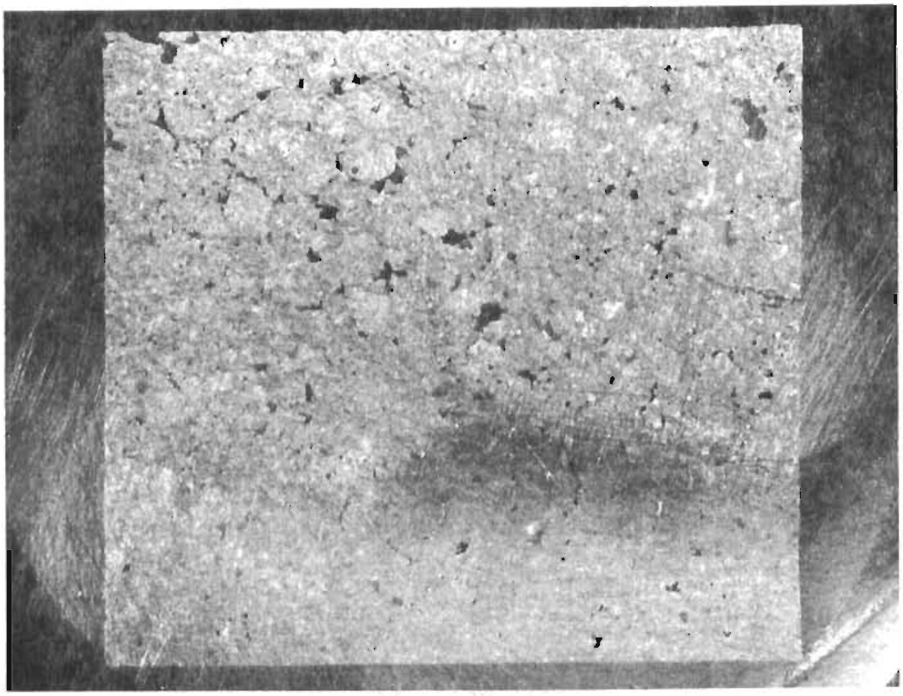

Figure 9. Graphite Specimen Surface After Grinding on 500-Grit Diamond Wheel, 7 X.

2.3. Polishing

The purpose of polishing is to remove scratches which are left after the last grinding step and to give the specimen a glossy surface free from these induced imperfections. Polishing involves two primary stages - rough polishing and fine polishing.

Rough polishing is accomplished using an aqueous suspension of No. 600 silicon carbide abrasive on a Buehler microcloth (No. 40-7028) rotating at 170 rpm. The cloth must be kept properly wetted with the suspension throughout the polishing operation. If the cloth is too dry, the specimen may become relief polished, while if too wet, the abrasive action of the silicon carbide is reduced and polishing time is increased.

Figure 10 shows a specimen surface after completion of rough polishing. The surface has a uniform dull luster and contains very fine scratches from the silicon carbide abrasive. The success of this polishing operation depends upon the care taken in the preceding grinding operations. If deep scratches are not removed in the fine grinding operations it will be impossible to obtain a satisfactorily polished specimen.

The specimen now must be thoroughly washed under a water jet since a clean specimen is extremely important before continuing to the next polishing stage. Deep scratches will result if all the silicon carbide is not removed, and will necessitate returning the sample to the rough polishing wheel.

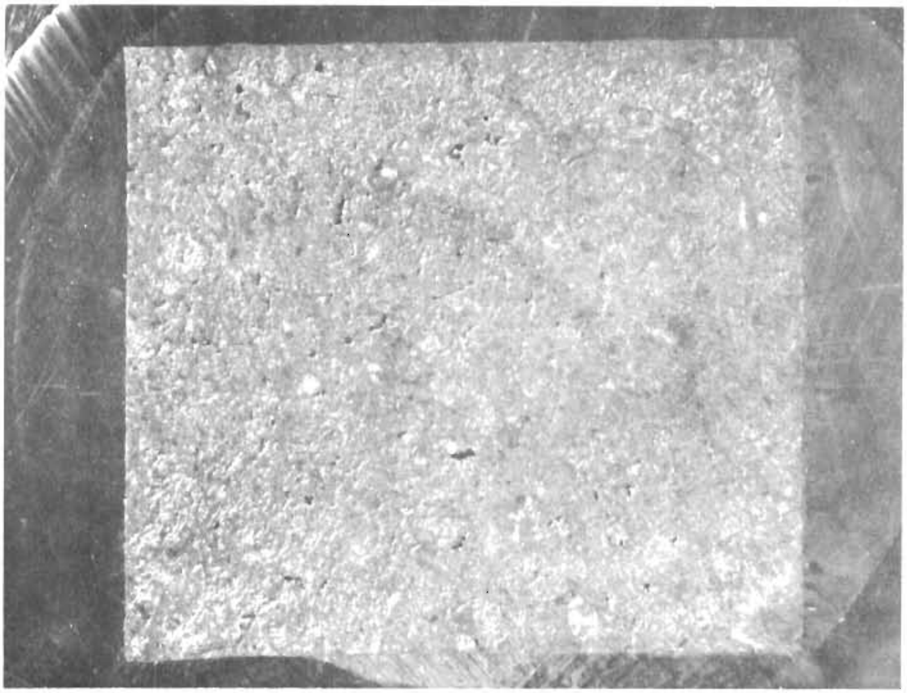

Figure 10. Graphite Specimen Surface After Polishing With 600-Grit Silicon Carbide Abrasive, 7X.

Final polishing requires two separate stages. The first of these uses an aqueous suspension of Linde Grade A alumina powder (0.3 micron particle size) as the abrasive. Polishing time is kept to a minimum so that the distortion layer will be very thin. Usually three to five minutes polishing will suffice for this step. At this point the specimen has a polished luster and the structure is more visible, as illustrated in Figure 11. Again the mount is thoroughly washed before moving to the final stage.

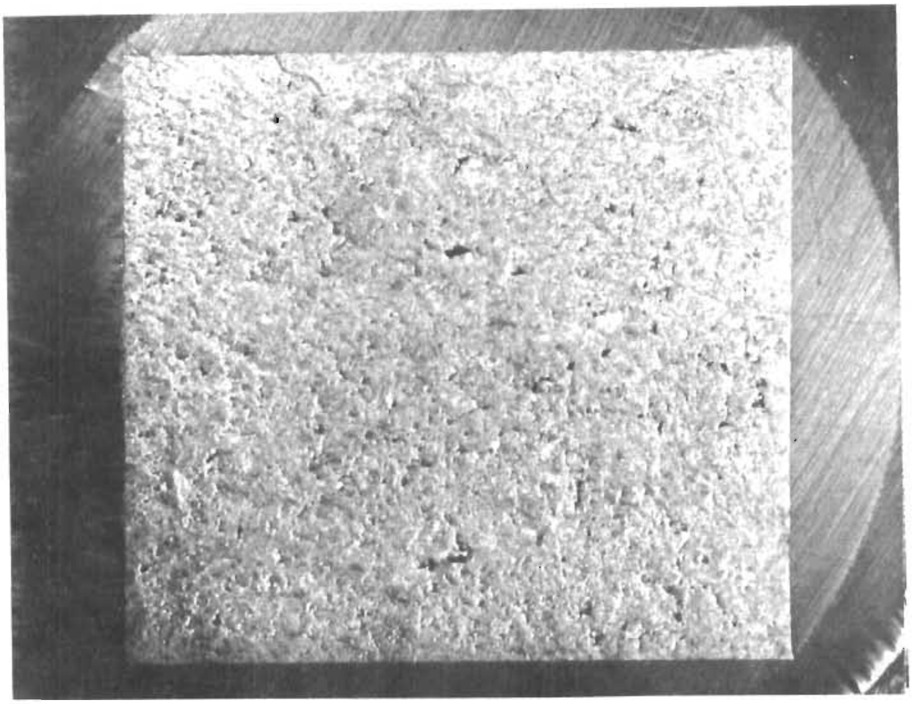

Figure 11. Graphite Specimen Surface After Polishing With 0.3 Micron Alumina Abrasive, 7X.

The last step consists of three to five minutes polishing with Linde Grade B alumina which is 0.05 microns in size. After completion of this step, the specimen surface should have a high polish and be free from scratches.

Figure 12 shows a typical surface for a graphite specimen which has been completely polished. Final polishing with Grade B alumina has removed microscopic scratches which would prove bothersome at magnifications above 100X. The specimen surface is again washed in running water, dipped in distilled water and dried in air jet. Failure to wash the specimen thoroughly will leave a thin film of alumina on the surface which will greatly hamper visual and photographic examination. Care must be exercised to protect the specimen from being stained by fingerprints or scratched during handling and storage.

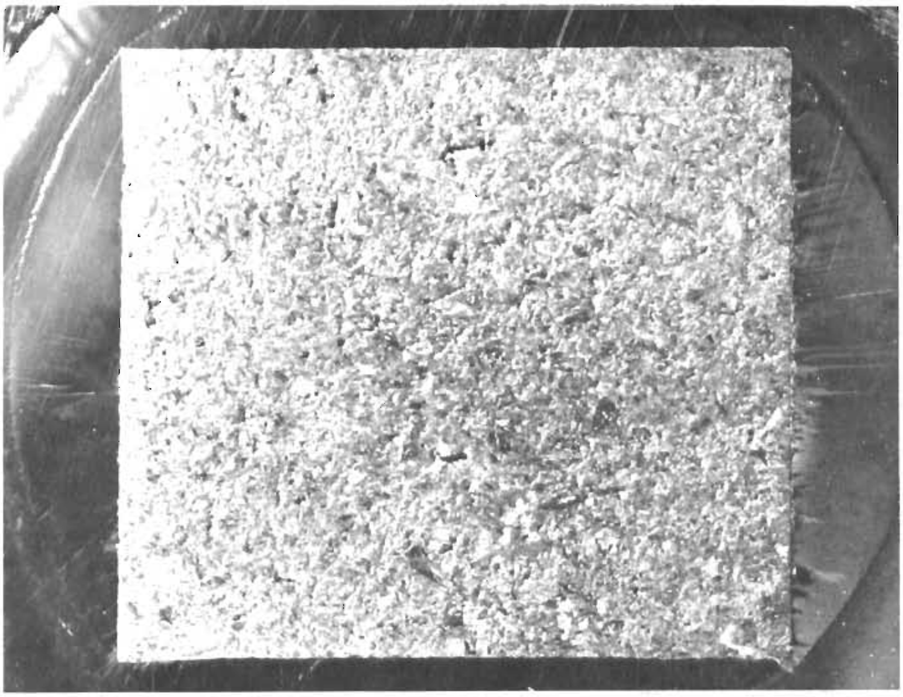

Figure 12. Graphite Specimen Surface After Final Polishing with 0.05 Micron Alumina Abrasive, 7 X.

In cases where both a soft and a hard phase are present in a graphite material, extreme care must be taken not to relief polish the specimen. Relief polishing is caused by a more rapid removal of the softer components of the sample. Cloths which have little or no nap (such as nylon) can often be used in place of the heavy nap microcloth to reduce the effects of relief polishing. If it is necessary to use the microcloth for dual-phase materials, polishing time should be kept to an absolute minimum. Figure 13 is a photomicrograph of graphite flour particles showing the effects of relief polishing. In this instance, the styrene was polished away more rapidly than the harder graphite particles resulting in a shadowy halo around most of the flour material. The shadows mask the particle perimeters making detailed study difficult. Figure 14 is the same specimen of graphite flour as shown in Figure 13 after regrinding and polishing in a manner so as to avoid relief polishing. The sharper, more detailed photomicrograph of Figure 14 is the result of shadows around the flour particles being nearly eliminated by the careful preparation of the specimen.

Extensive relief polishing may make microscopic focusing difficult or impossible by exceeding the depth of focus of the microscope or metallograph. Figures 15 and 16 are photomicrographs of a ceramic coating on graphite-base substrate material. In Figure 15 the substrate is in focus but the coating is blurred, while in Figure 16 the coating is in focus but the substrate is blurred. The relief polishing in this case has made it impossible to obtain a sharp photomicrograph of both phases and the clarity of one must be sacrificed to reveal the detail of the other. The use of a vibratory polisher, with appropriate abrasives, will reduce considerably the relief polishing effect when ceramics are a part of the carbon and graphite sample.

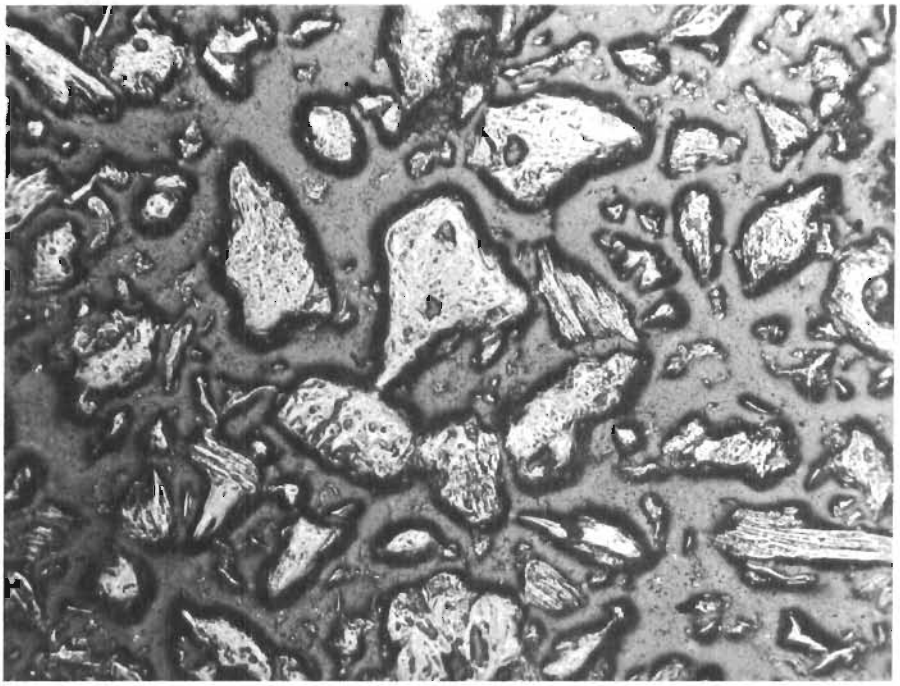

Figure 13. Graphite Flour Showing Relief Polishing Effects, 100 X.

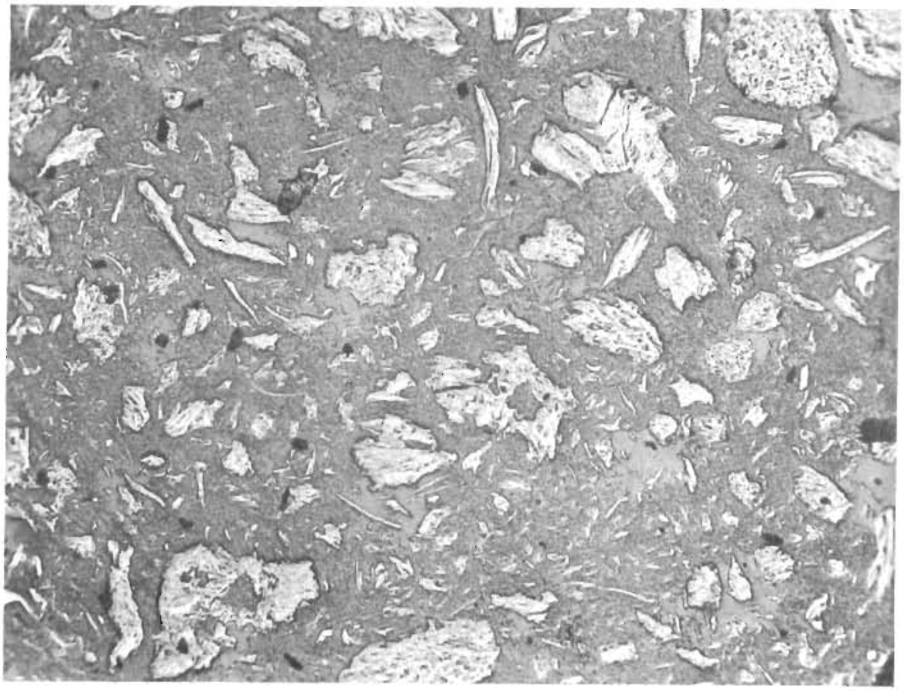

Figure 14. Graphite Flour in the Absence of Relief Polishing, 100 X.

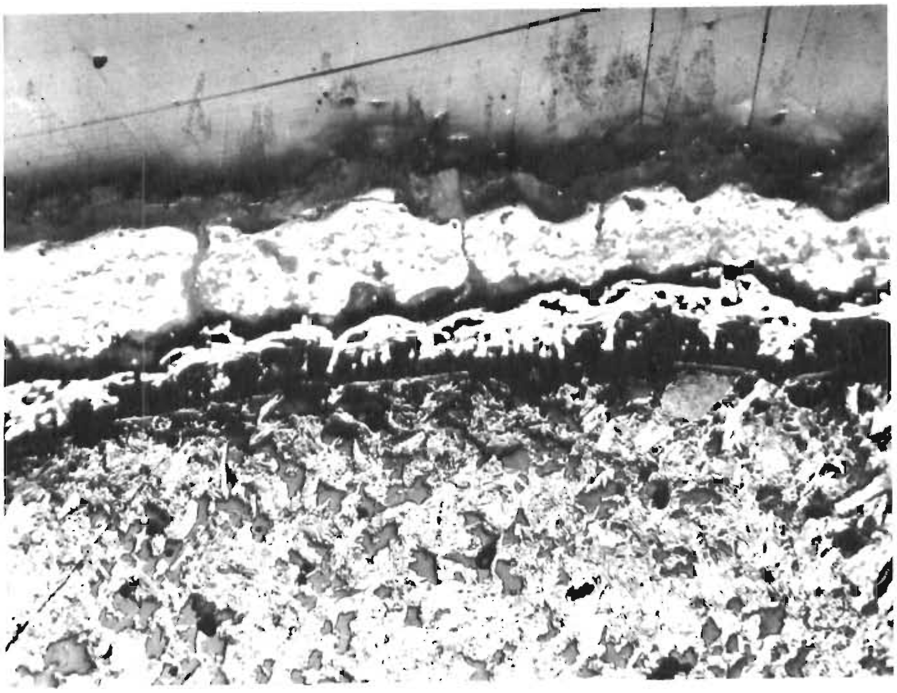

Figure 15. Relief-Polished Ceramic Coating on Graphite Substrate, Substrate in Focus, 100 X.

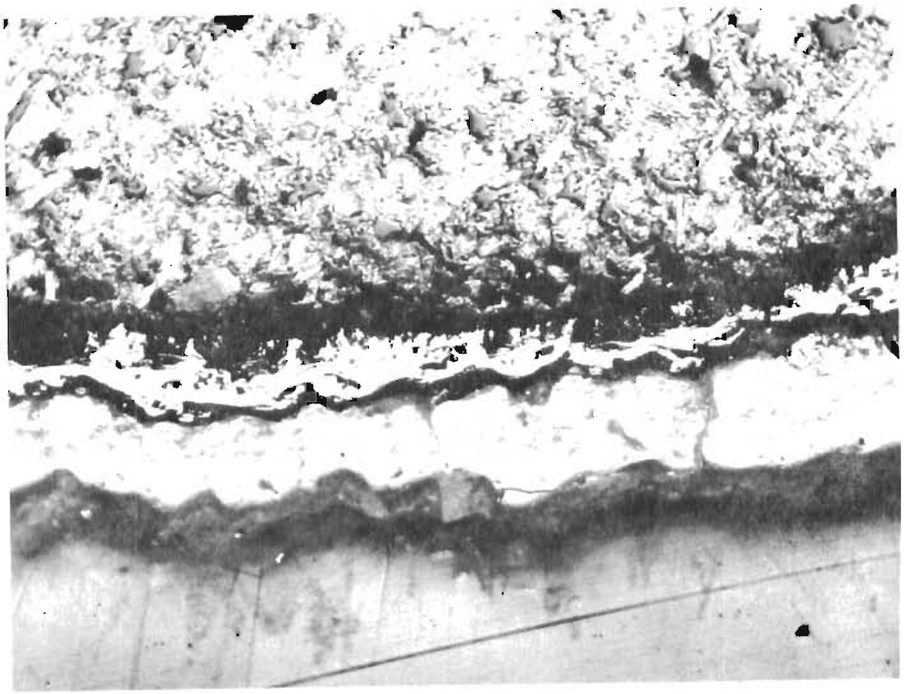

Figure 16. Relief-Polished Ceramic Coating on Graphite Substrate, Coating in Focus, 100 X.

Specimen preparation methods described thus far have been used to obtain excellent results for carbon and graphite materials. However, this does not imply that they are the only techniques applicable to these materials. Preparative procedures should be flexible enough to accommodate the wide range of hardness and abrasiveness encountered in carbonaceous materials.

3. PHOTOMICROGRAPHY

A properly prepared specimen is absolutely necessary for high-quality photomicrographs. Knowledge of the photographic apparatus and techniques of photography are also necessary to obtain a truly representative photograph of the subject material.

3.1. Objectives and Eyepieces

A photomicrograph representative of a given specimen can usually be made at 100 X magnification. If a specific area of a specimen is of particular interest, much higher magnifications may be necessary. Achromatic objectives are used in making photomicrographs for magnifications up to 1000 X. For magnifications above 1000 X, oil-immersion apochromatic objectives are preferred.

Huygenian eyepieces are most often used in conjunction with achromatic objectives for both visual and photomicrographic work. These eyepieces do not produce an image which is completely free from distortion but for most applications the loss is negligible. If a flatter field is necessary for photomicrography after visual examination of a specimen, an amplifier eyepiece may be used instead of the usual eyepiece. The amplifier is designed to partially correct for the curvature of field present in microscope objectives. Curvature of field produces a difference in focus from the center to the periphery of the image field. The amplifier amends this curvature, producing a flatter field with more of the image in focus. The field of view of amplifier lenses is so small that they cannot effectively be used for visual examination.

3.2. Illumination

Metallographs and metallurgical microscopes are equipped with means of supplying several types of illumination from a single light source. The microscopist may choose among three types of light source; the ribbon filament, the zirconium arc, and the carbon arc. The latter, chosen for this work, is the brightest of the three sources and is extremely useful in obtaining reasonable photomicrographic exposure times, particularly with polarized light and sensitive tint illumination.

3.2.1. Bright-Field

Bright-field illumination with suitable filters is preferred for most photomicrographic work and renders a dark image on a lighter background as shown in Figures 17 and 18. Achromatic objectives are spherically corrected in the yellow-green region of the spectrum,(c) hence a yellow and green filter combination (Figure 17) gives the best image definition. The yellow filter cuts out blue and violet light, and the red light is eliminated by the green filter. Resolving power and definition is slightly decreased if the filters are used separately. A green and yellow filter combination can also be used with apochromatic objectives which are spherically corrected for the green and blue color regions. Blue filters can be used with apochromatic objectives because of the higher color correction present in these objectives but should not be used with achromatic objectives because correction for spherical aberration does not extend to the red or violet regions. Figure 18 illustrates a specimen photographed with an achromatic objective and a blue filter. Much of the detail of Figure 17 is lost in Figure 18 because of the fuzzy and indistinct image produced with a blue filter.

Oblique illumination is frequently used to highlight a specimen which has little surface relief. This type of lighting creates shadows which accentuate areas of depression and relief. Figure 19 illustrates the appearance of a specimen in direct axial illumination as opposed to its appearance in the oblique lighting of Figure 20. The differences in relief and in structural detail are much more obvious with the oblique lighting. Oblique illumination is not recommended for specimens composed entirely of particles because shadows are produced around the particle and the surface detail is obscured.

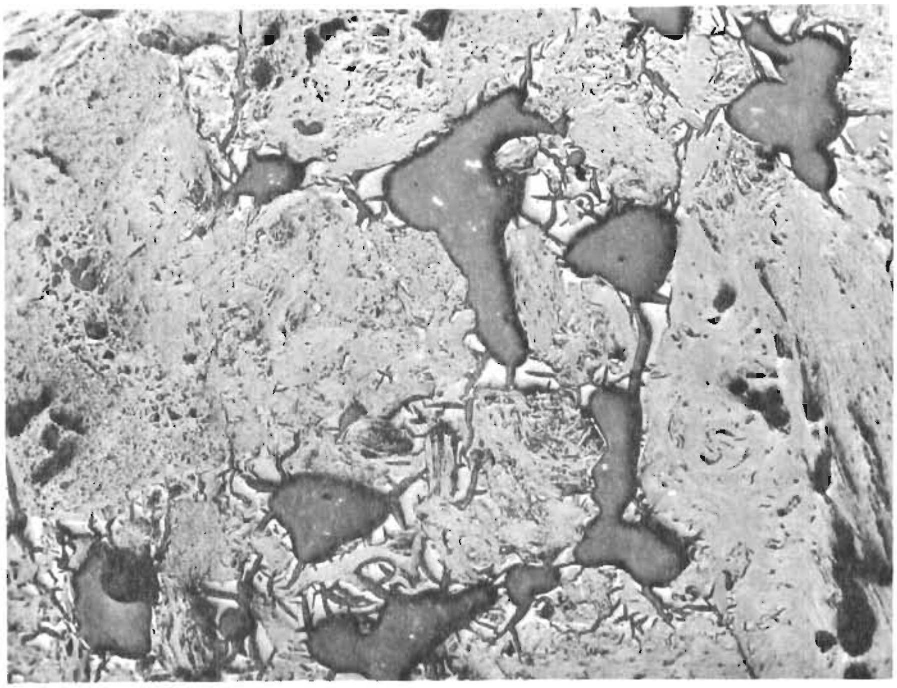

Figure 17. Impregnated Graphite, Bright-Field Illumination, Yellow and Green Filters, 100 X.

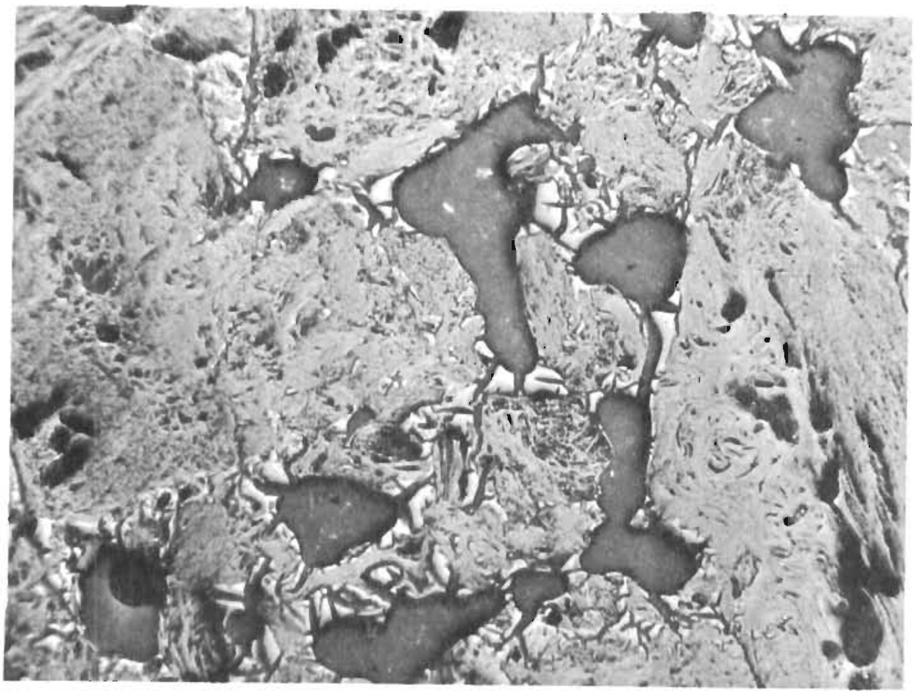

Figure 18. Impregnated Graphite, Bright-Field Illumination, Blue Filter, 100 X.

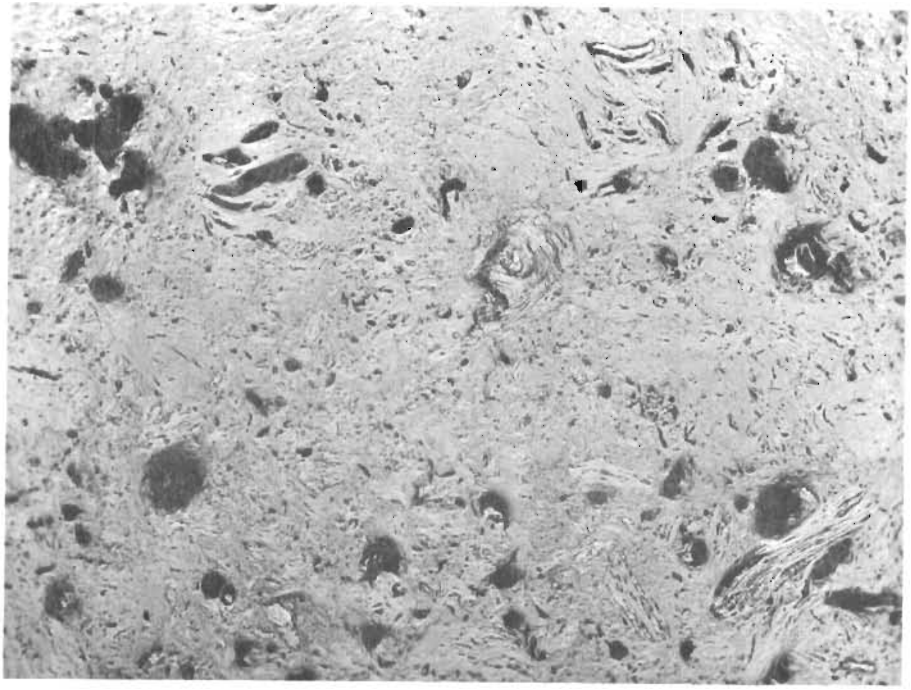

Figure 19. Specimen Illuminated by Means of Direct Axial Illumination, 100 X.

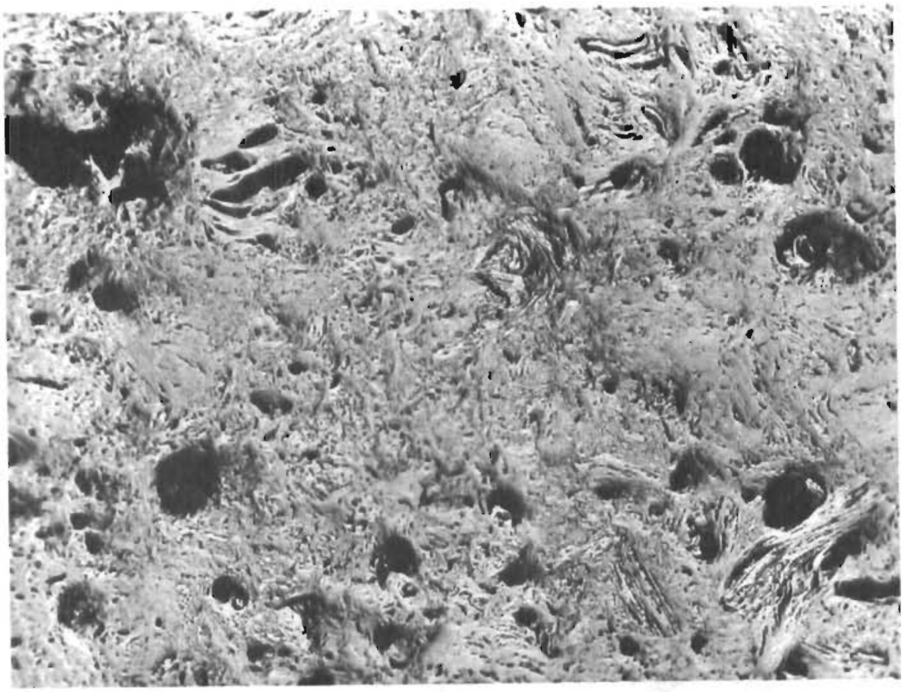

Figure 20. Specimen Illuminated by Means of Oblique Illumination, 100 X.

3.2.2. Polarized Light and Sensitive Tint

Polarized light or sensitive tint illumination can be used to reveal microstructures not readily apparent with filtered bright-field illumination. Completely polarized light darkens the styrene and impregnant, revealing only the graphite particles, as illustrated in Figure 21. Here the polarized light has been used to advantage in showing both the structure and distribution of the graphite particles. In other cases polarized light may tend to destroy detail as illustrated in Figure 22, which is the same specimen area shown in Figures 23 and 24. Such drastic changes in the appearance of a specimen illustrate the care needed in choosing the proper illumination when making micrographs of carbon and graphite materials.

Sensitive tint illumination is essential in color photomicrography of carbonaceous materials, but is also useful for black and white reproductions. The sensitive tint plate is usually made from optical quality quartz, gypsum or mica and is inserted in the optic axis between the polarizer and analyzer assemblies. The plate rotates the plane of polarization of the light resulting in blue and purple hues for most graphite materials. Easily detected color changes are produced by slight rotations of the microscope stage.(7) Sensitive tint illumination frequently reveals minute difference in structure not clearly defined by other types

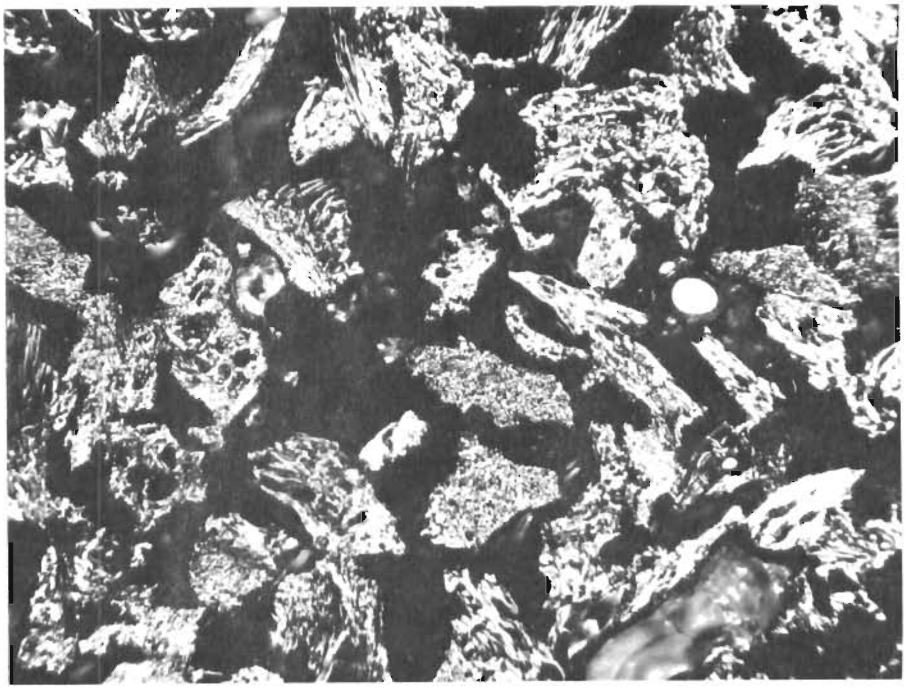

Figure 21. Completely Polarized Light, Same Sample Area Shown in Figures 25 and 26, 100 X.

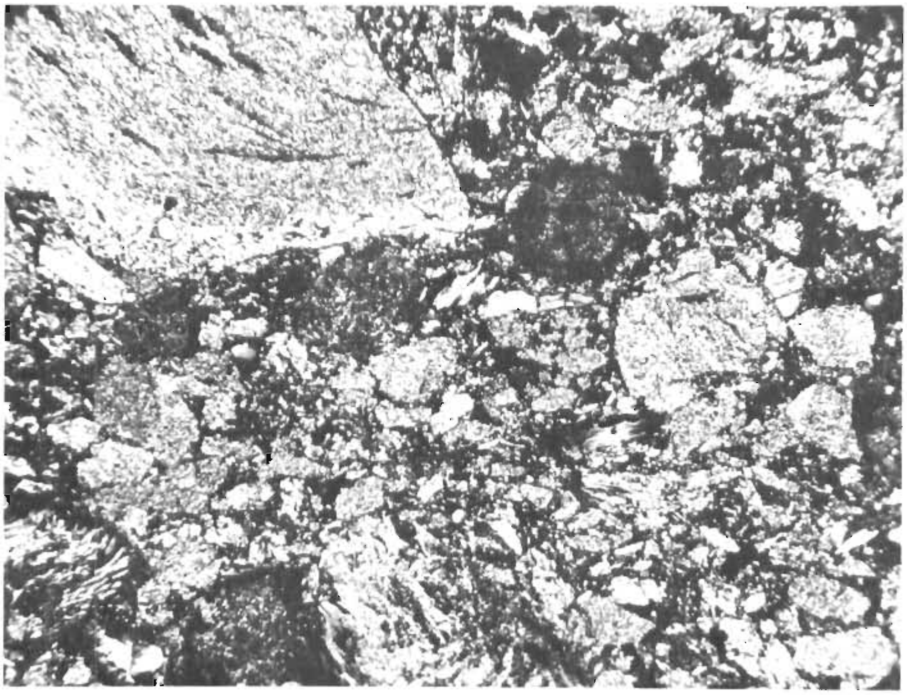

Figure 22. Completely Polarized Light, Same Sample Area Shown in Figures 23 and 24, 100 X.

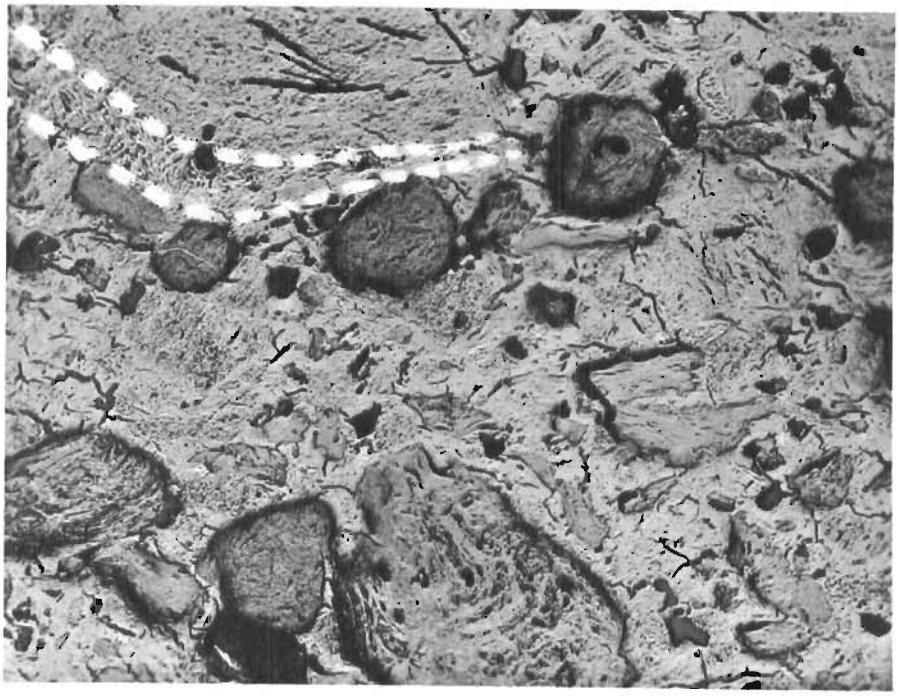

Figure 23. Specimen Photographed with Bright-Field Illumination, Yellow and Green Filters, 100 X.

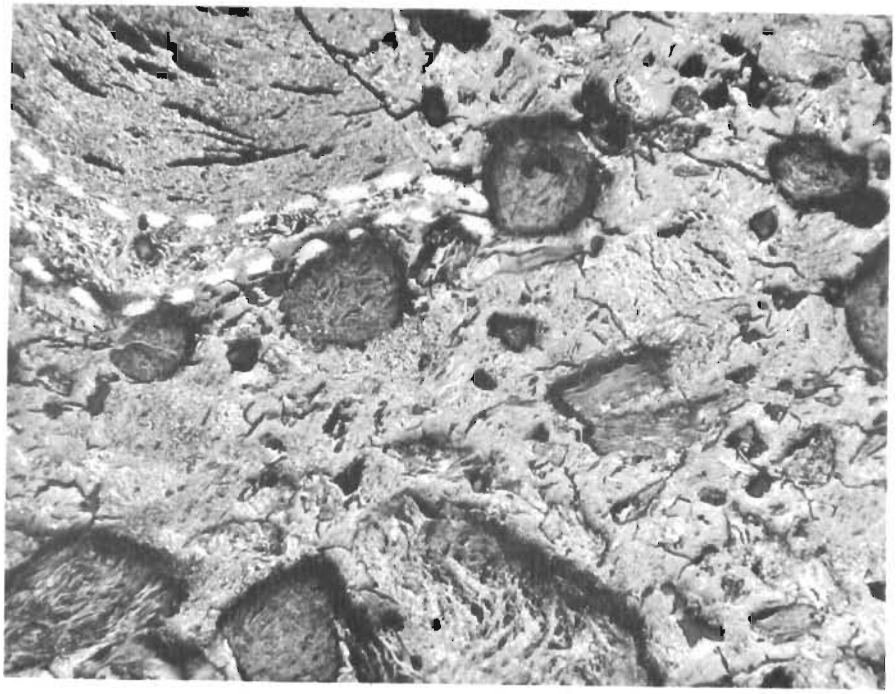

Figure 24. Specimen Photographed with Sensitive Tint Illumination, 100 X.

of lighting. Figures 23 and 24 are photomicrographs of the same specimen, the former being filtered bright-field illumination while the latter is sensitive tint. The dotted lines surround areas of graphitized binder which is more easily detected by the sensitive tint photographed in black and white in Figure 24. For some materials, sensitive tint offers little advantage over filtered bright-field illumination as illustrated by the nearly identical photographs in Figures 25 and 26.

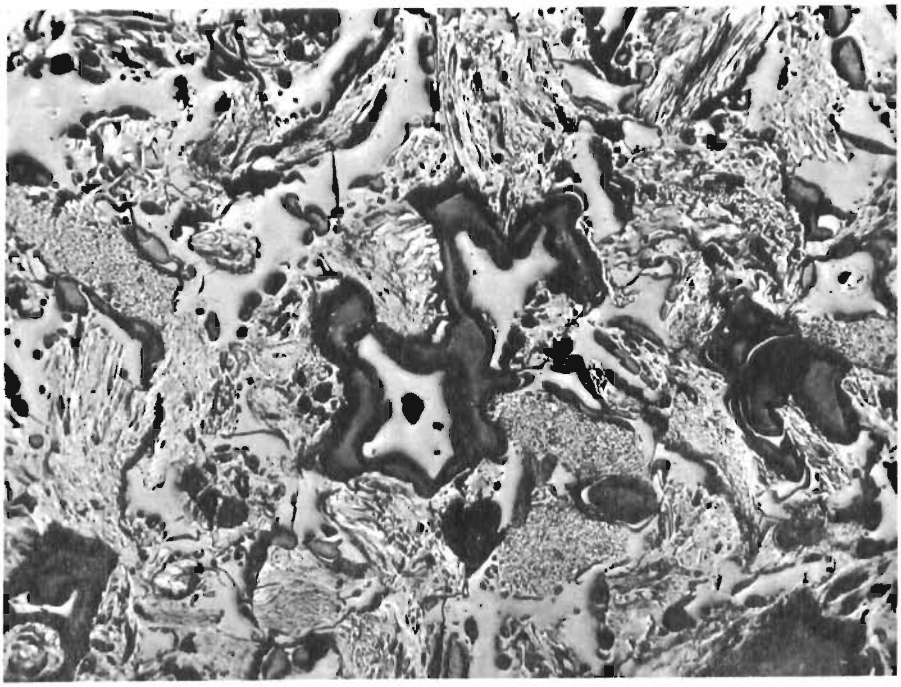

Figure 25. Specimen Photographed Using Bright-Field Illumination, Yellow and Green Filters, 100 X.

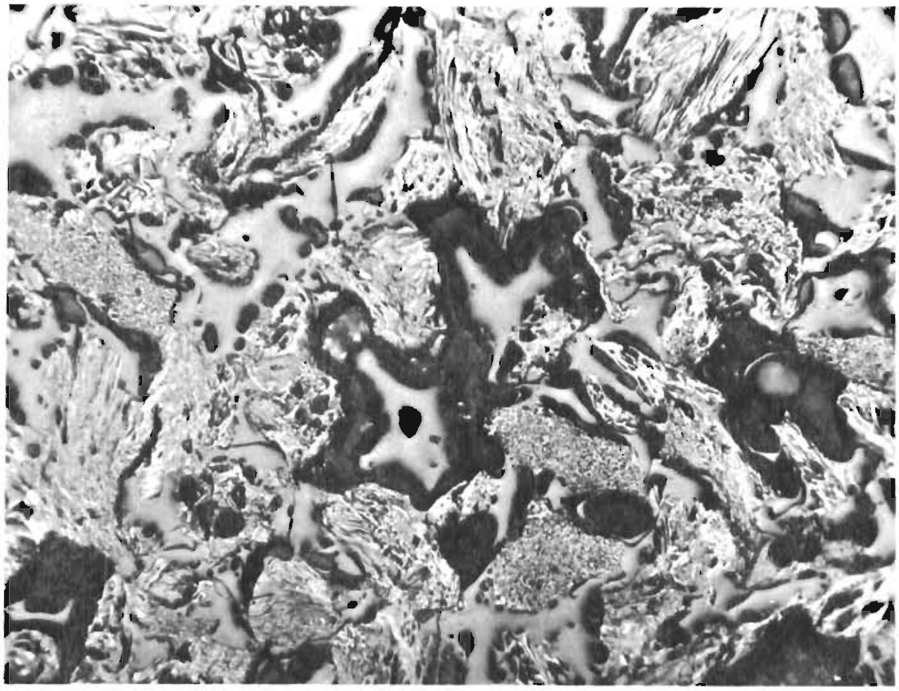

Figure 26. Specimen Photographed Using Sensitive Tint Illumination, 100 X.

Varying degrees of polarization can be obtained by rotating the polarizer the desired amount if the metallographic unit is so equipped. Partially polarized light is advantageous for examination of some materials. For example, Figure 27 is a photomicrograph of graphite cloth taken with partially polarized light as the illumination. The difference in fiber orientation, accentuated by the darkening of certain regions, is clearly visible. Complete polarization of the light, Figure 28, destroys the contrast and results in a photomicrograph with little detail. On the other hand, filtered bright-field illumination produces the relatively dull, uniform shading shown in Figure 29.

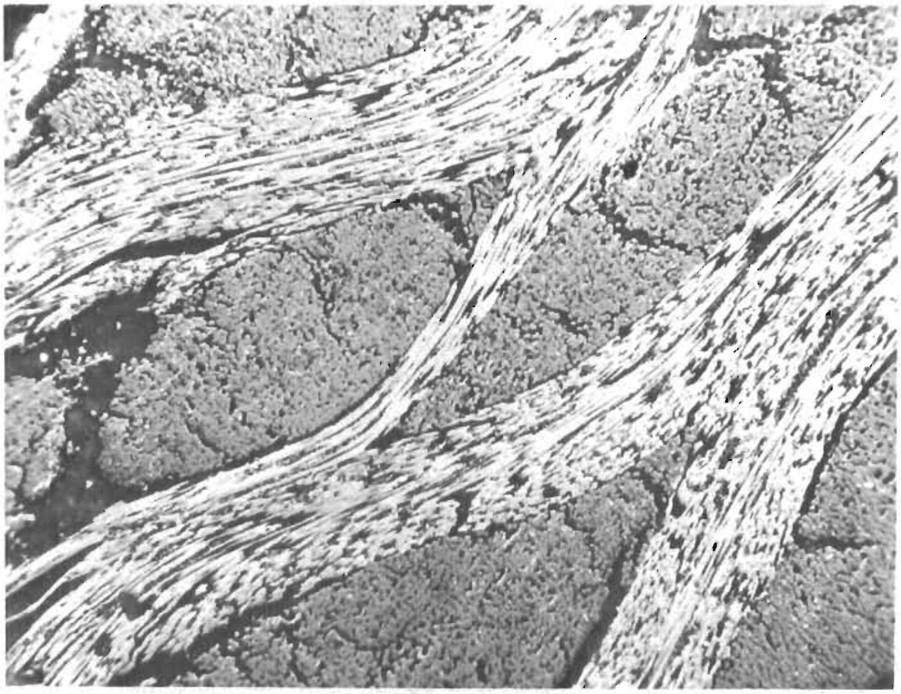

Figure 27. Graphite Cloth Using Partially Polarized Light, 100 X.

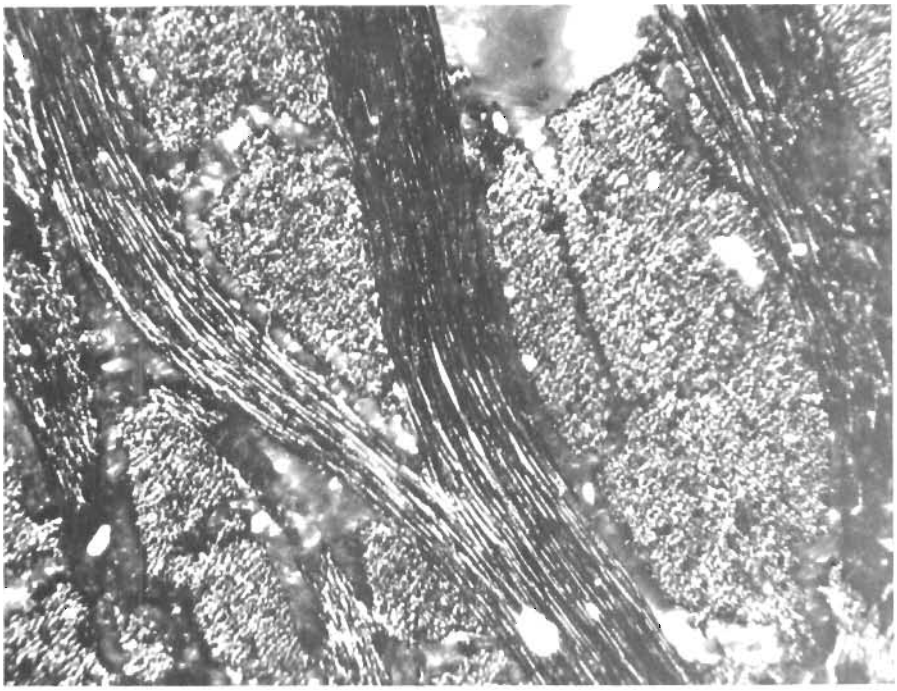

Figure 28. Graphite Cloth Using Completely Polarized Light, 100 X.

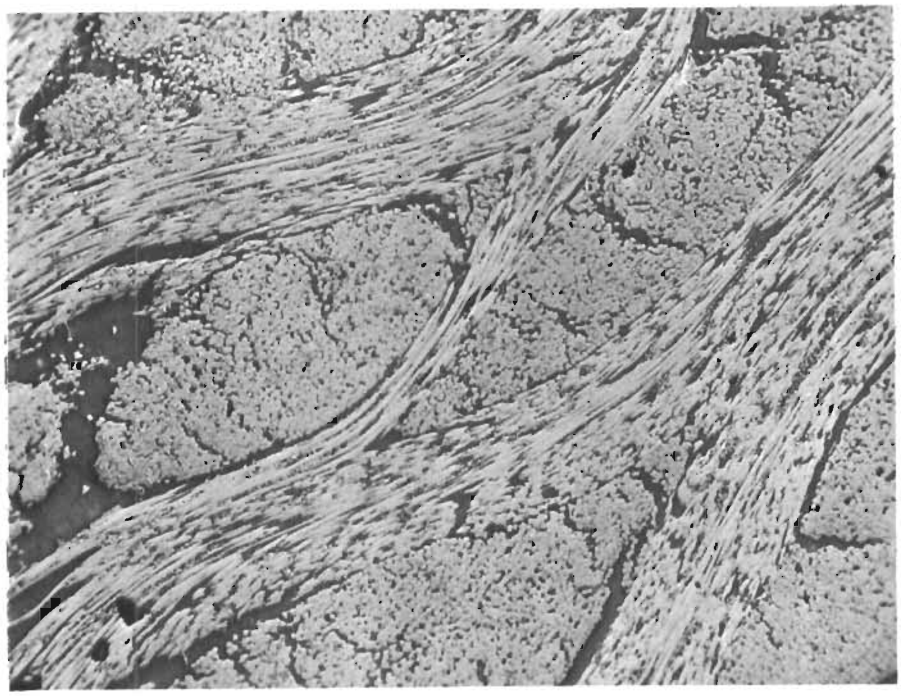

Figure 29. Graphite Cloth Using Bright-Field Illumination, 100 X.

3.2.3. Dark-Field

Dark-field illumination, the opposite of bright-field, produces an image having a dark background with a light structure and is sometimes very useful in photomicrography of carbon and graphite specimens. Most carbon and graphite appear extremely diffuse in dark-field illumination as seen in Figure 30, which shows the same sample area as that in Figures 23 and 24. The visible microstructure has been practically destroyed, making interpretation nearly impossible.

Dark-field lighting can be used with graphite cloth to show structural relationships between fibers or groups of fibers, as illustrated in Figure 31. Contrast is extremely high permitting easy detection of most individual fiber ends. It can also be used to highlight impregnant material as illustrated in Figure 32. The styrene is white, the impregnant coke is black, and the graphite particles are a mottled grey. For this particular specimen, which is the same as shown in Figures 25 and 26, dark-field lighting has made it possible to easily locate the impregnant.

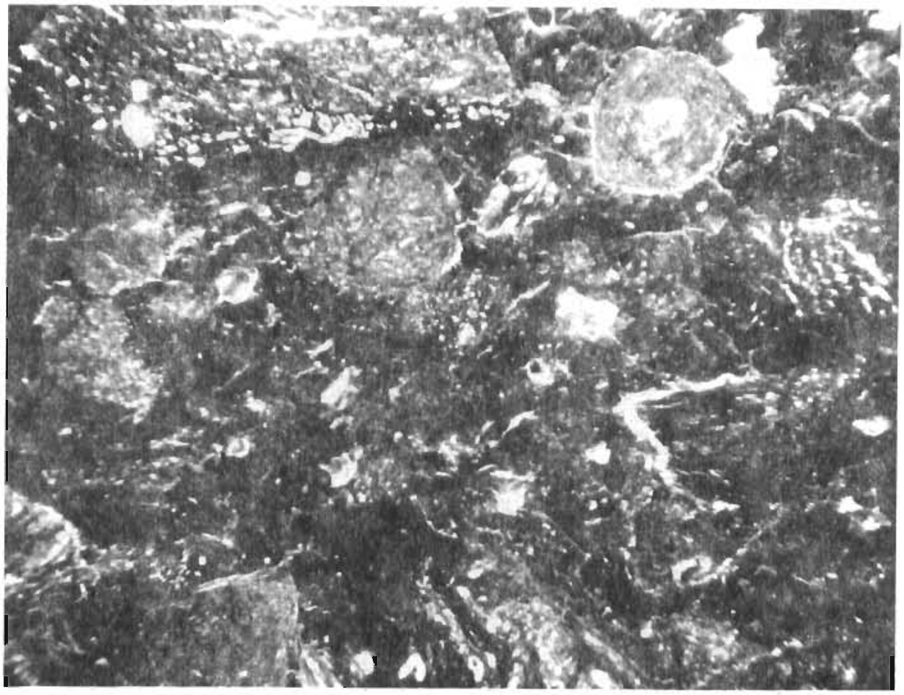

Figure 30. Dark-Field Illumination, Sample Area Shown in Figures 23 and 24, 100 X.

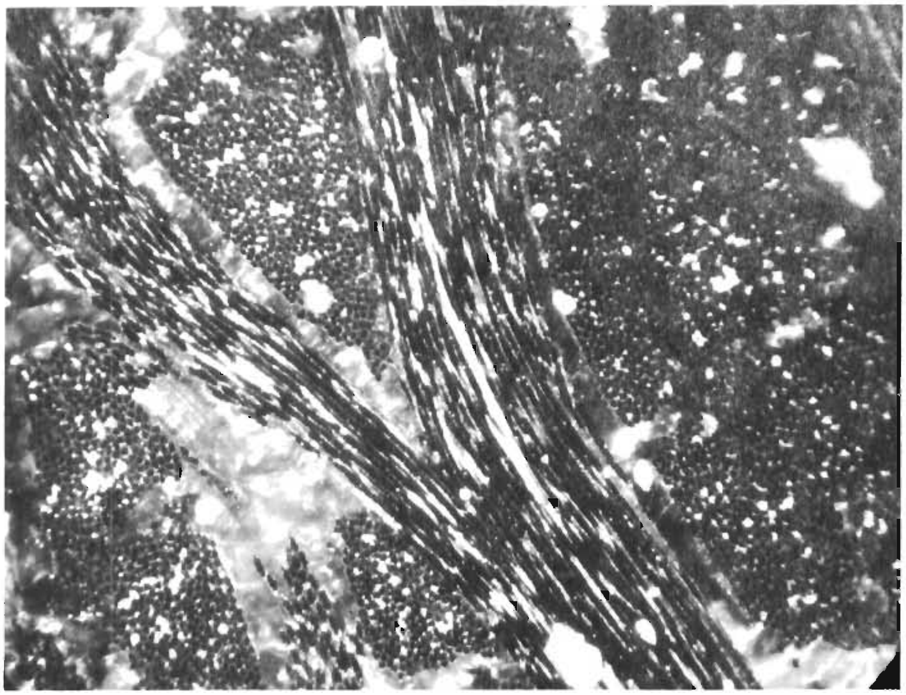

Figure 31. Dark-Field Illumination of Graphite Cloth, 100 X.

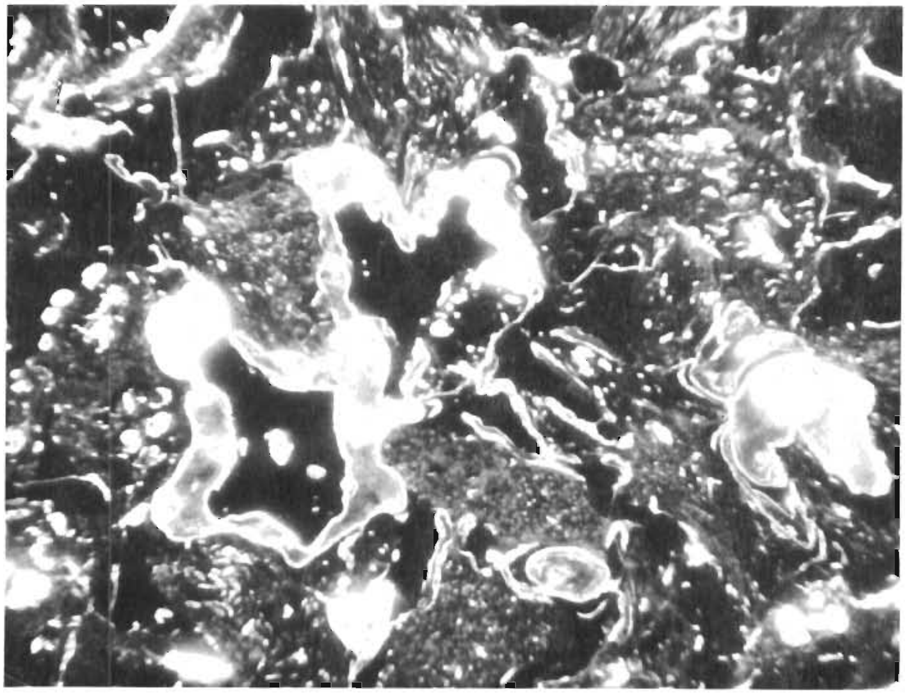

Figure 32. Dark-Field Illumination, Same Area Shown in Figures 25 and 26, 100 X.

3.2.4. Color

Color photomicrographs are obtained by using a sensitive tint plate with polarized light and any of several types of available color film. Color is often a great aid in revealing differences in structure not readily discernible in black and white, particularly differences in crystallinity and orientation.(8) Further discussion of color photomicrography is beyond the scope of this report but the technique has been discussed briefly by Pincus and Gendron.(2)

3.3. Photography

The production of good-quality photomicrographs is dependent upon the selection of the appropriate photographic film or plates, developer and printing paper.

3.3.1. Films and Plates

Photographic "M" plates were used and found to be well suited for work with carbon and graphite materials since they possess good image contrast between light and dark areas and reveal fine structural details. If film is preferred, a fine-grain, thin-emulsion film is available which is comparable to the photographic "M" plates. Both plates and film have fine-grain emulsion and can be enlarged several times without a grainy appearance. The major advantage of film over plates is the ease with which film is handled and stored without breakage.

High resolution plates are useful for material with uniform structure. Much greater relief perspective is imparted to the specimen structure as illustrated by the difference shown in Figures 33 and 34. The greatest disadvantage of high resolution plates is their slow emulsion speed requiring long exposure times, particularly with polarized light or heavily filtered illumination. A wide selection of films and plates permits a choice to produce practically any negative contrast desired, but those just described were most commonly used by the authors for carbon and graphite materials during this work.

The entire surface of a specimen may be photomicrographically scanned by mounting a motion picture camera on one tube of a binocular eyepiece. The other tube may be equipped with an eyepiece allowing visual and photographic scanning to be done simultaneously, thus assuring good photographic coverage. Good black and white and color picutres have been obtained with high speed motion picture films.

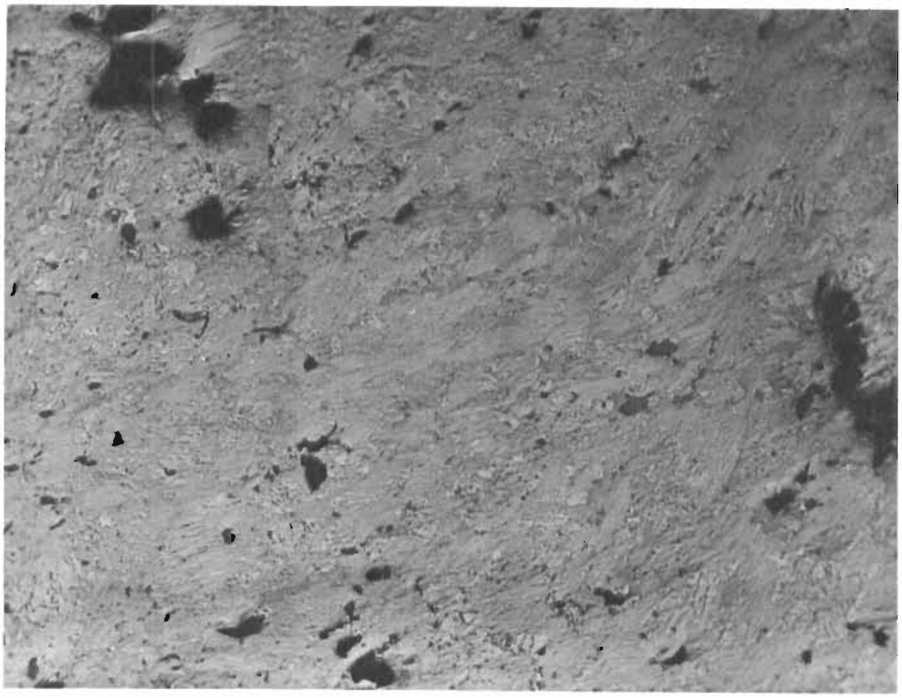

Figure 33. ZTE Graphite Photographed on "M" Plate, 100 X.

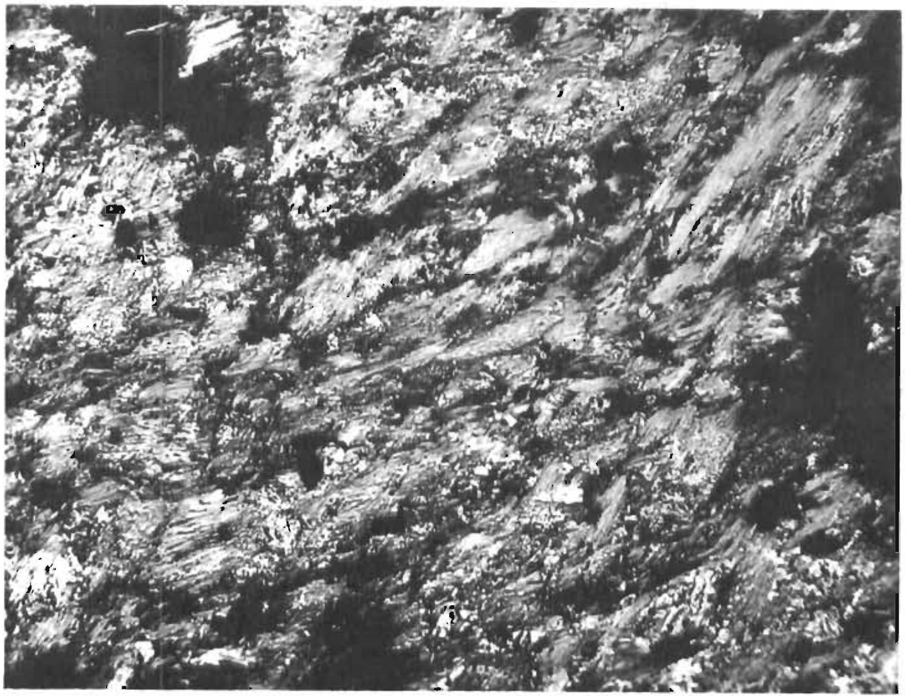

Figure 34. ZTE Graphite Photographed on High Resolution Plate, 100 X.

3.3.2. Developers

From the wide variety of commercially available developers three types (D-19, DK-50 and D-76) were used for most of the photomicrographic applications herein discussed. D-76 is a low-contrast developer and is used for practically all film and plates. The loss in image contrast is compensated for by the increase in fine detail. DK-50 is a moderately high-contrast developer giving slightly greater contrast to the negative than D-76 but frequently results in a loss of the fine detail produced by using D-76. D-19, a high-contrast developer, is seldom used for routine photomicrography. It is useful when detail is not important and high contrast is desired. Figures 35 and 36 show how the extreme contrast of the plate developed in D-19 (Figure 36) has resulted in a great loss of structural detail apparent on the plate developed in D-76.

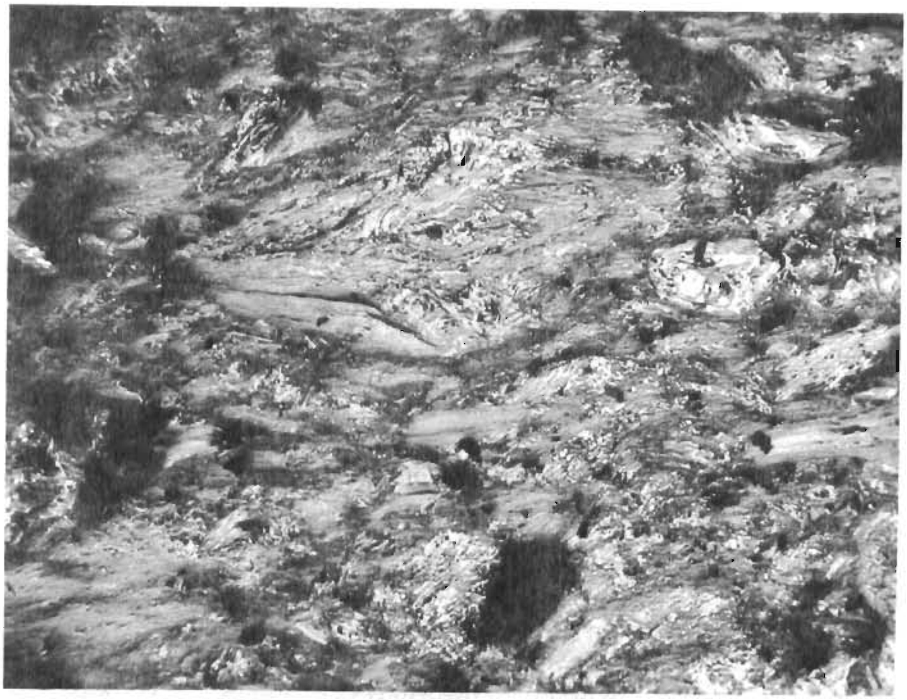

Figure 35. ZTE Graphite Photographed on High Resolution Plate, Developed in D-76, 100 X.

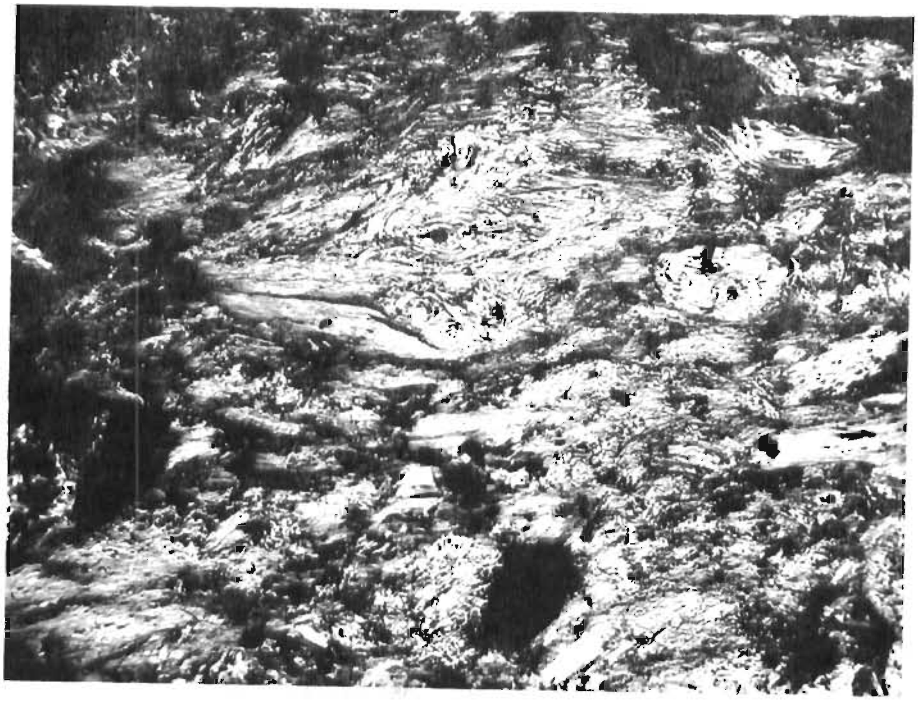

Figure 36. ZTE Graphite Photographed on High Resolution Plate, Developed in D-19, 100 X.

3.3.3. Printing

The choice of printing paper depends upon the contrast of the negative and the contrast desired on the final print. A paper of relatively low contrast, such as No. 2, is required where detail is of paramount importance. If structural contrast is desired, No. 4 or 5 paper is necessary. No. 3 paper is used when both detail and structural contrast are desired. Although No. 3 paper sacrifices some detail for contrast, the resulting print is generally preferable for routine work. None of these papers can be used to correct for defects arising from improperly exposed negatives. Although some papers may give partial correction, it is strongly recommended that time be taken to establish correct film and plate exposure times.

4. PYROLYTIC GRAPHITE

Pyrolytic graphite offers excellent examples of the microstructural details that can be revealed by various types of illumination. Great care must be taken to remove all scratches during specimen preparation so that they will not mask the true microstructure of the pyrolytic graphite.

Figures 37 and 38 are macrographs (6 X magnification) of a pyrolytic coating on a graphite rod. Figure 37, made in bright-field illumination, shows very little structural detail while in Figure 38 the use of polarized light has clearly revealed the conical structure typical of some pyrolytic graphites. Figures 39 and 40 show similar specimens taken at 250 X magnification using filtered bright-field illumination and polarized light respectively. Even at this higher magnification the bright-field illumination does not reveal the conical structure which is clearly shown with polarized light. In both figures the layers, formed as the pyrolytic graphite is deposited, are visible but are much more clearly defined in Figure 40.

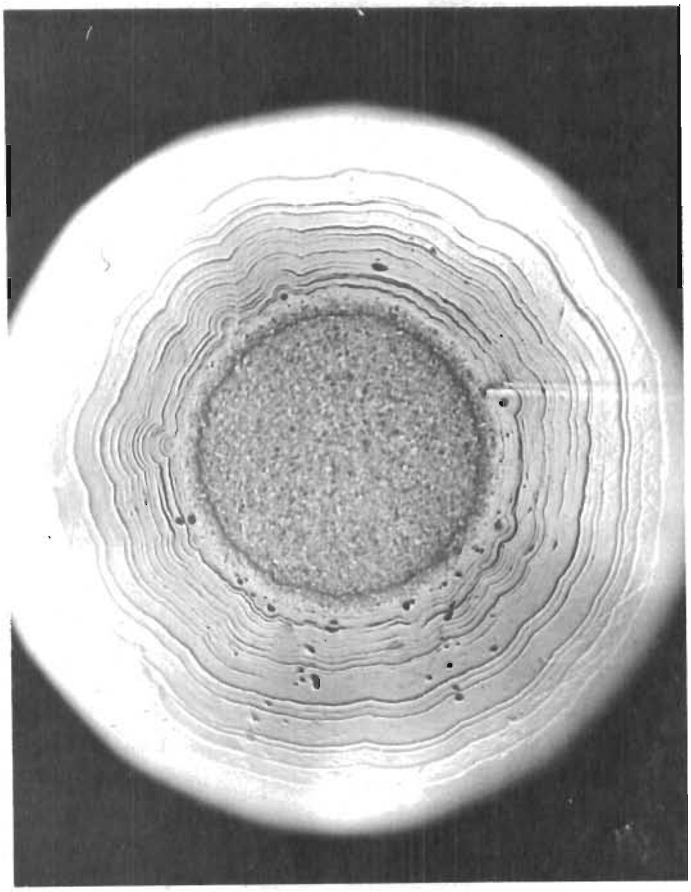

Figure 37. Pyrolytic Coating on Graphite Rod, Bright-Field Illumination, 6 X.

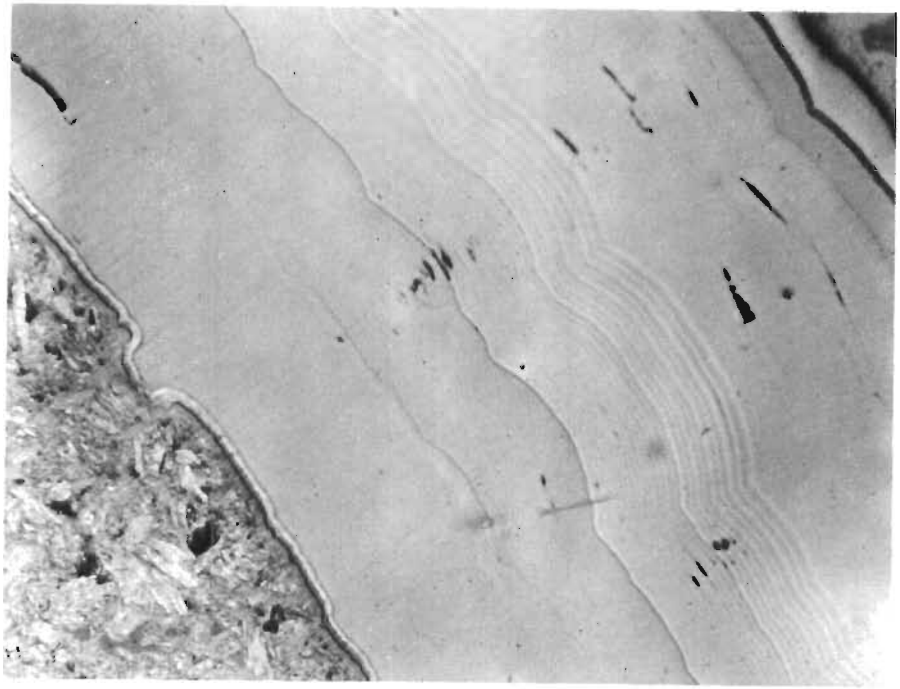

Figure 39. Pyrolytic Coating, Bright-Field Illumination, 250 X.

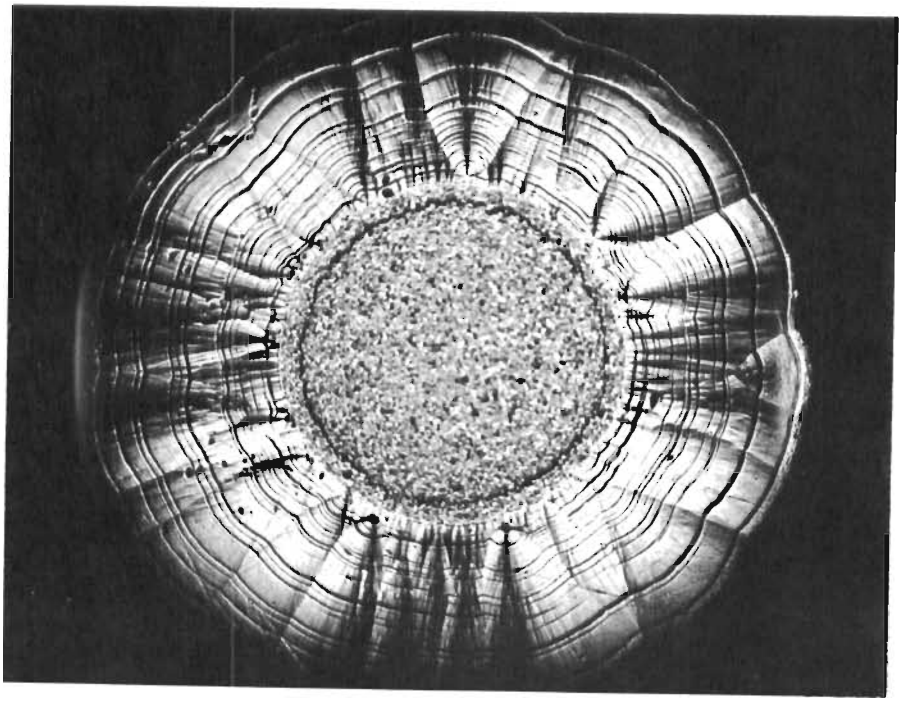

Figure 38. Pyrolytic Coating on Graphite Rod, Polarized Light, Illumination, 6 X.

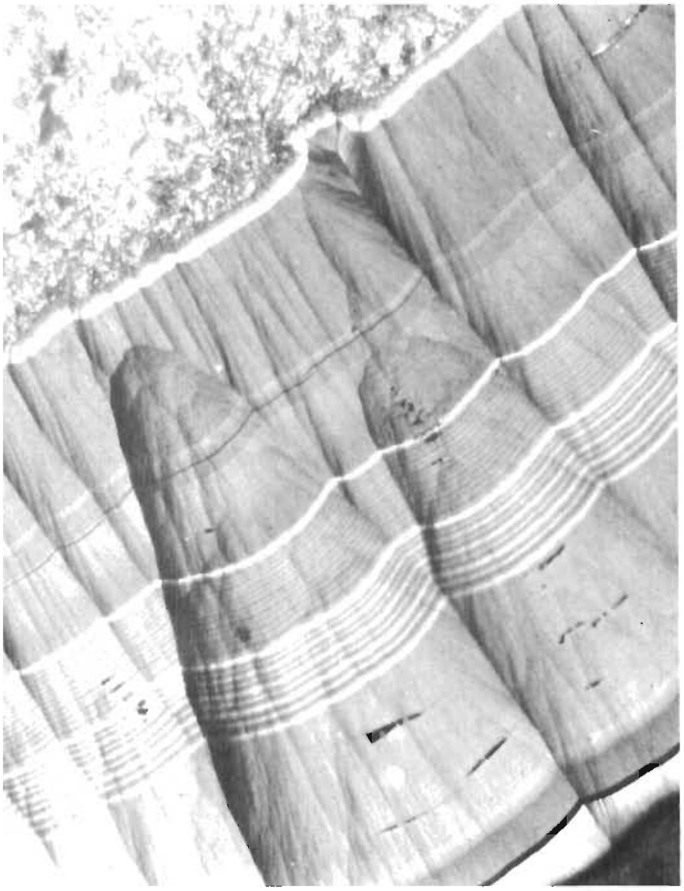

Figure 40. Pyrolytic Coating, Polarized Light Illumination, 250 X.

Full polarization of the light with the sample rotated to "extinction" renders the high-contrast image shown in Figure 41. Although the structural orientation is more evident, some of the detail is lost because of the extreme contrast. The same specimen was photographed using sensitive tint illumination in Figure 42 and more clearly shows the fine detail. Dark-field lighting is extremely poor for the study of pyrolytic graphite. In Figure 43 dark-field lighting has reduced the detail of the large cone to a shadowy outline and the smaller cones are obliterated.

Sensitive tint and polarized light is the preferred method of illumination for detailed studies of pyrolytic graphite microstructure. However, with polarized light, care must be taken to properly orient the stage so as to avoid extreme contrast.

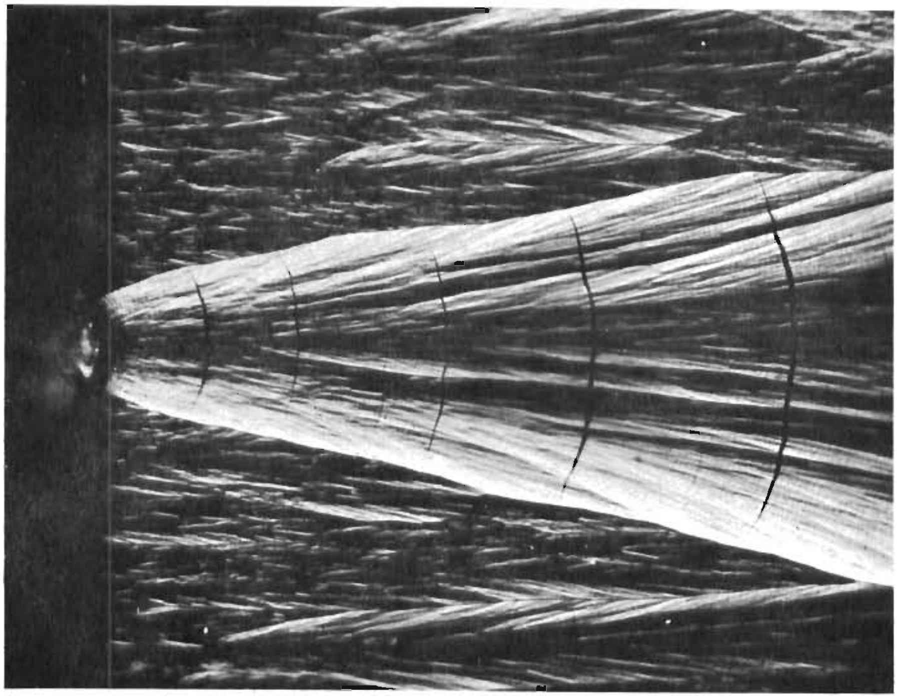

Figure 41. Pyrolytic Graphite in Polarized Light Illumination, 100 X.

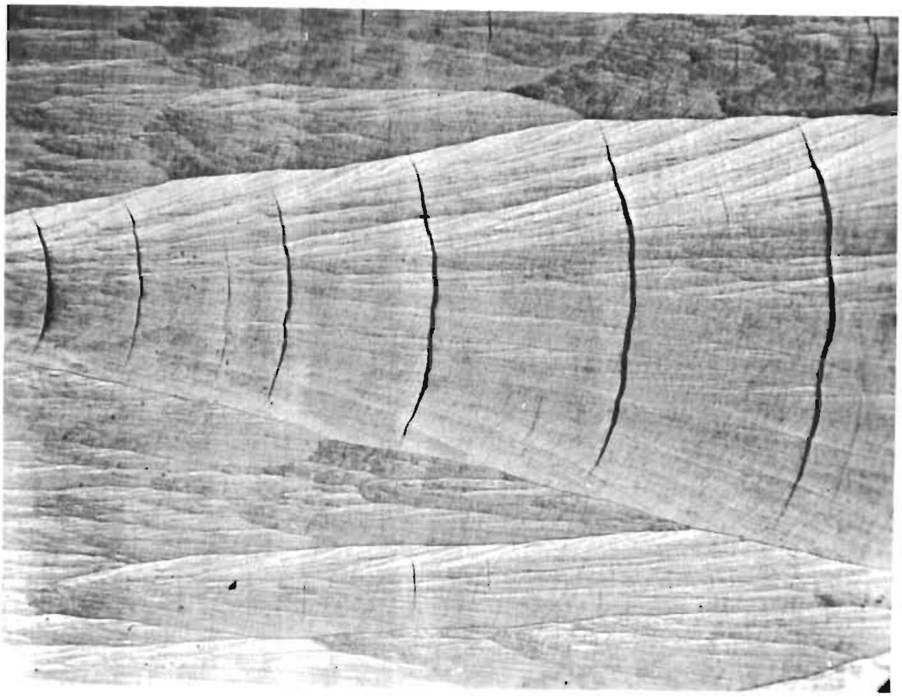

Figure 42. Pyrolytic Graphite in Sensitive Tint Illumination, 100 X.

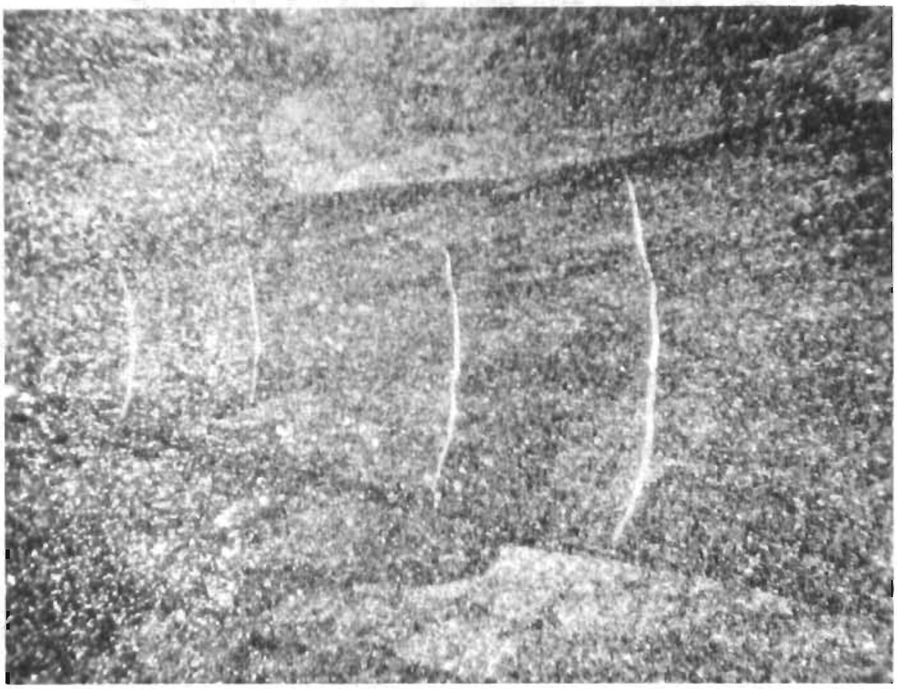

Figure 43. Pyrolytic Graphite in Dark-Field Illumination, 100 X.

5. REFERENCES

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