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The Electron Microscope

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Table of Contents

Plates

INTRODUCTION

The original light microscope of 250 years ago had a useful magnification of about 20 diameters. All the advances in microscope design of the past two and one-half centuries have achieved a maximum useful magnification of about 1,000 diameters. About 50 years ago, Abbé gave theoretical support to experimental observations that optical instruments could not reveal objects smaller than one-third the wavelength of light. The electron micrsscope, entirely a product of the past twenty years, has already surpassed this limit by a factor of 50 to 100.

In modern research many problems deal with structures measuring less than one micron, or one twenty-five-thousandth of an inch. The science of colloids is concerned with particle sizes ranging from 10 microns to about 3 milli-microns or 30 angstroms. Viruses, large complex molecules and most bacteria are under the limit of optical resolution. The preparation of pigments is essentially a problem in particle size control, the critical size being of the order of the wavelength of visible light, about one fifty-thousandth of an inch. Studies of dusts and smokes indicate that 75 percent of the particles are completely invisible in optical microscopes. In the field of catalysis, the optical microscope reveals little of the distribution and size of active surface areas. Metallography, as a means of observing the internal structure of metals and alloys, is handicapped by the limiting resolution. For example, x-ray diffraction patterns indicate that in age-hardening aluminum alloys, the precipitates are from 2 to 50 atoms thick and from 20 to several hundred atoms long or wide. Such structures can never be observed directly with an optical system. These are but a few examples to illustrate the need for a microscope with at a least one order of magnitude greater resolution than the optical microscope. This need has now been filled by the development of electron misroscopes capable of covering the entire range of structures from ten microns to about 40 angstroms.

CHAPTER I

Uses of the Electron Microscope

In this country, electron microscopes have been available for little more than two years and are beginning to be used in many fields. Biologists are carrying on investigations of immune reactions, staphylococcus and phage, tubercle bacillus, and pneumococcus. For the first time they have been able to observe and photograph viruses. Metallurgists have investigated structures like troostite, unresolvable in the light microscope, and have identified them as very fine pearlite. Industrial chemists are studying minute particles of all types. Carbon particles of colloidal size have been photographed as they appear in numerous substances such as rubber for tires, pigment in printer's ink, and coloring matter in plastics. In the paint industry, particle sizes and the degree of aggregation are now easily observed with the electron microscope. In the field of plastics, interesting micrographs have been published of catalysts such as aluminum oxide gel, silica gel, palladium on asbestos, vanadium pentoxide, etc. These pictures give direct information as to sizes and distributions of active surfaces. In some cases, it is possible to study chemical reactions on a colloidal scale, as has been done in a study of the photo-chemical reduction of silver in silver bromide, followed by the developing and fixing process.

The microscope at the Naval Research Laboratory, which was acquired in November, 1942, thus far has found most of its application in chemistry and metallurgy. The instrument has been used to study pigments, impregnating materials for protective clothing, smokes, non-reflecting surface films and fillers for plastics. The specimen technique for metallurgical studies has been improved to the point where valuable information is now being obtained about the heat treatment of steel.

CHAPTER II

Magnification and Resolving Power of the Electron Microscope

The question most often asked about the electron microscope is, "What is the highest magnification obtainable?" The maximum direct magnification

is of the order of 25,000 diameters. The human eye has a resolution of about 0.1 millimeter, and if the original phtograph did not contain detail smaller than 0.1 millimeter, no thing could be gained by further photographic enlargement outside the microscope. Actually, the electron micrographs contain detail too small for the eye to see even at the highest direct magnifications. It is therefore useful to magnify the image further in a photographic enlarger. Useful magnifications are obtainable up to 100,000 diameters. Most electron micrographs are taken at magnifications between 1500 and 10,000 diameters to include larger fields of view and then enlarged from 5 to 10 times optically.

Of much more significance than the question of magnification is the degree of resolution attained in a microscope. With the electron microscope it is possible to resolve objects from 50 to 100 times smaller than is possible with fine optical microscopes. To understand this gain in resolution, consider the factors that limit the resolving power of an optical system.

Each point in an object is imaged by an optical instrument as a diffraction pattern. A photograph of a point source, such as a distant star, is revealed upon enlargement as a round disk surrounded by alternately light and dark rings. It was shown by Rayleigh that the images formed by an optical system of two close points could just be resolved when the distance between them was such that the central diffraction disk of one just coincided with the first dark ring of the other. Stated mathematically, this condition for the light microscope is given by Abbé's formula:

$$\delta = \frac{0.61\lambda}{\text{N. A.}} \qquad (1)$$

where δ is the limit of resolution or the smallest distance by which two particles may be separated and still be imaged as two particles, λ is the wavelength of the radiation used, and N. A. is the limiting numerical aperature of the objective lens. In the most perfect light microscopes yet built, a practical limit of 1.4 is reached for N. A. If we take 4,000 angstroms as the limit of human vision, formula (1) gives 1,700 angstroms as the limit of visual resolution. In the "ultra-microscope," by

the use of ultraviolet light of wavelength 2,537 angstroms, a resolution of 750 angstroms can be attained. Extreme resolution is achieved in dark field microscopy by resorting to oblique illumination to give higher N. A. Although smaller particles are then seen, no direct measure is obtained of their sizes and shapes. Because of the lack of suitable refracting media for lenses, shorter ultraviolet radiations cannot be used.

The use of electrons in microscopy makes possible a considerable gain in resolution. Fast electrons may be regarded as radiation with a very much shorter wavelength than visible light. The wavelength of an electron of mass m, moving with a velocity v, after acceleration through a potential drop V, is given by

$$\lambda\,(\text{cm}) = \frac{h}{mv} = \frac{1.22\times10^{-7}}{\sqrt{V}} \qquad (2)$$

With an accelerating voltage of 60,000 volts, the electron wavelength is only 0.05 angstroms. Therefore, even with a numerical aperture as small as 0.001 cm, a resolution of 30 angstroms is indicated by formula (1).

Almost equally important is the excellent depth of focus characteristic of electron micrographs. The microscopist who has photographed objects at 1,000 diameters with an optical microscope is well acquainted with the difficulty of obtaining more than a small portion of the field in sharp focus. Only particles within a layer of about 0.2 micron thickness appear sharply focused in any given micrograph. With the electron microscope, the depth of focus is about 1.0 micron if the objective aperture is 0.001 centimeter.

CHAPTER III

Design of the Electron Microscope

Busch showed in 1926 that a stream of electrons could be focused upon passing through a magnetic field in a manner closely resembling the focusing of light by a glass lens. A cylindrically symmetrical magnetic or electric field bends a divergent beam of electrons moving nearly parallel to the axis to form a point image of a point source, as shown in Plate 1. The earliest electron micro-

graphs were emission studies using tubes similar to ordinary cathode ray tubes with some modifications in the electron gun and lenses. In 1932, the first compound electron microscope was built by Knoll and Ruska in Germany. Commercial microscopes are now manufactured by Siemens Halske in Germany and by R. C. A. in the United States.

In its geometrical arrangement of lenses, specimen, and source of illumination, the electron microscope closely resembles the compound light microscope (Plates 2, 3). The electrons are emitted from a hot tungsten filament which is at a high negative potential of from 30 to 60 kv with respect to a cusp shaped grounded anode. The high electron velocity is attained in covering the small gap between the apex of the filament and the aperture in the anode. This electron gun converges the electrons towards the specimen. The illumination of the specimen is further controlled by the condenser lens. Each electromagnetic lens consists of a coil of wire surrounding the microscope axis, and pole pieces shaped so as to give the desired flux distribution. The coils are iron clad and outside the evacuated volume. The sample holder is held inside the objective lens to permit the use of the short focal lengths necessary for high magnification. Those electrons which pass through the object are focused to form an image in the focal plane of the projector lens at a magnification of 50 to 150 diameters. This primary image is then further magnified by the projector lens to form the final image on the fluorescent screen or photographic plate. The final magnification may be anywhere between 1,000 and 25,000 diameters. An interesting difference between the optical and electronic instruments is the use of fixed focal length lenses in the former and variable focal lengths in the latter. To focus with the light microscope, the object distance is varied. With the electron microscope, all the distances are fixed and focusing is achieved by varying the objective coil current.

There are numerous difficulties encountered in electron microscopy that do not enter into optical microscopy. Electrons are scattered easily by gaseous molecules, so that the entire electron path must be in high vacuum (10^{-4} to 10^{-5} millimeters of mercury). This vacuum is obtained by continuously pumping the column with a three jet oil diffusion pump and Megavac fore-pump. Only very thin objects,

of the order of one hundred-thousandth of an inch in thickness, may be examined because of the weak penetrating power of electrons. Many specimens are destroyed by heat absorbed in stopping the electrons. To obtain a resolution of 50 angstroms in the micrograph it is essential that the lens currents be extremely stable, and that the column be free of vibration. Since the deflection of the electron beam in a magnetic lens is a function of the electron velocity, well-regulated high voltage is required to avoid chromatic aberration. Spherical aberration must be reduced by the use of apertures of the order of 20μ in diameter.

Various additions to the electron microscope facilitate its use. These include air locks for introducing specimens and photographic plates without destroying the main vacuum; a moveable stage for scanning specimens; and fluorescent screens for observation of images at all stages of the system.

CHAPTER IV

Electron Diffraction Adapter for the Electron Microscope

The electron microscope is easily converted into a high precision electron diffraction camera. This change is accomplished by removing the original projection lens assembly and replacing it with a unit containing a new projection lens and in addition a specimen holder and special focusing lens (Plate 23). To photograph a diffraction pattern, the specimen is inserted above the special focusing lens. The objective lens serves to form a very fine point image of the source. This insures that any point on the specimen receives electrons from only one direction. The projector lens is not energized. Electrons striking the sample are diffracted in various directions depending on the crystal structure of the sample and the accelerating voltage applied to the electrons. The function of the focusing lens is to concentrate all electrons deflected through a given angle and in a given direction at the same point on the plate.

When the specimen is made up of small crystalline particles in random orientations, the incident electrons may be deflected through a given

angle with equal probability in any azimuth. The resulting pattern is a series of concentric rings called a Debye-Scherrer diagram (Plates 24, 25). The ring diameters are directly related to the spacings of neighboring atoms in the crystal lattice.

Opaque as well as transparent substances may be studied with the diffraction technique. The holder has been designed to permit orienting the specimen so that the electron beam just grazes the surface, giving rise to a reflection pattern. Single crystals yield reflection patterns from which the lattice constants may be determined to within three tenths of one percent.

CHAPTER V

Specimen Preparation

Image formation in the electron microscope is the result of scattering and absorption of part of the incident beam by the specimen material. The final image on the fluorescent screen is produced by those electrons which are not scattered or absorbed. When the object is relatively thick or dense the electron image is merely a silhouette, but if the specimen thickness and density are within the proper range, the images show all the tonal gradations attainable in normal photographic work (Plate 9).

Since the electron beam has so little penetrating power, it is obvious that conventional methods of mounting specimens for optical microscopy are entirely inapplicable to electron microscopy. Glass slides and cover glasses are out of the question. Instead, specimens are supported on thin membranes, stretched across wire mesh screens. These membranes may be formed of collodion, parlodion, formvar, silica, alumina, or beryllium and their thickness need not exceed 100 angstroms. The supporting screens (Plate 4) are 200 mesh nickel. The field observed in the microscope at lowest magnifications is still smaller than the open area of a single mesh.

There are four common methods of mounting specimens: (1) collecting material directly on the screen; (2) depositing the specimen on a thin membrane; (3) sandwiching the object between two layers of a nitrocellulose film; (4) the replica technique.

(1) When the specimen to be mounted is in

the form of needle-like crystals or long fibers, it can be mounted directly on the screen. Smoke particles are quickly collected by holding a screen above the burning material. Particles adhere to the edges of the screen and to each other, forming a lattice work extending well out toward the center of the screen aperture (Plate 7).

(2) The most common mounting technique employs thin supporting membranes. Collodion films spread on a water surface from a 2 percent solution are only 100 angstroms thick and show no resolvable structure in the electron microscope. The mesh screens are placed on the upper film surface after it is formed on water. A screen, together with the portion of the membrane in contact with it is then removed by applying a suitable tool to the underside of the membrane. Water adhering to the film is removed with a capillary tube and the screen with its attached membrane is then permitted to dry. If the material to be studied can be prepared in a water suspension, a droplet may be placed on the film on the side away from the screen and the solvent evaporated, (Plate 10). A number of nitrocellulose films other than collodion can be used. Formvar films, prepared on polished glass or mica surfaces, are tougher than nitrocellulose. Various stages in the above preparation technique are illustrated in Plates 5 and 6.

Exceptionally strong supporting films are prepared by evaporation of silica onto a nitrocellulose film backing. After completing the evaporation, the backing is easily removed by dissolving in alcohol or amyl acetate. Such silica films can be heated to 1,000° C in vacuo without destruction, though they are only 100 angstroms thick.

(3) Materials which are not readily dispersed in water are often prepared by dispersing the particles in nitrocellulose or formvar. The film is then formed by spreading on water in the normal fashion, leaving the particles sandwiched between two layers of membrane (Plates 11, 12). Most pigment particles are studied by this technique.

(4) Replica techniques make possible the study of surfaces with the electron microscope in the usual transmission arrangement. The methods of replica preparation may be classed under three headings: (a) natural surface films; (b) single step synthetic and metallic surface films; (c) double replica techniques.

(a) The natural surface film technique is best applied to those metals which form thin protective oxide coatings. Aluminum and nickel and most of their alloys quickly form oxide coatings of the order of 50 to 100 angstroms in thickness. These films can be removed from the base metal surface by chemical means without injury and show no inherent structure of their own in the electron microscope.

(b) The method of casting a thin film of nitrocellulose on a surface, from which it is stripped after setting, has long been used in preparing replicas of diffraction gratings for spectroscopy (Plate 16). A thin layer of collodion or Formvar solution spread on a solid surface, takes the form of the surface on the contact side as the solvent evaporates. The upper surface dries smoothly under the influence of surface tension forces. The film is then scored with a needle or a razor blade and can be removed from the solid by dipping in water, provided the solid material is wet by water. As the film floats off the metal, it is caught on a mesh supporting screen and mounted for viewing in the microscope. High spots of the original surface appear bright in the micrograph. While this technique is fast and does not destroy the specimen, it doesn't offer the most in contrast and resolution.

Another single film process consists of evaporating a light metal, such as beryllium, onto the specimen surface. Such films, although thin enough for transmission of the electron beam, are strong enough to be stripped from the specimen after evaporation and mounted in the usual fashion.

(c) The double replica techniques are designed to eliminate the mechanical removal of synthetic films from specimen surfaces. The first successful method of this type was the silver-collodion process. The prepared surface is reproduced on silver by evaporating a heavy silver coating and stripping it. Silver films do not adhere appreciably to materials with no silver content. A thin film collodion replica is then made of this silver impression and removed by dissolving the silver in nitric acid.

At present, the most effective replica technique is the polystyrene-silica method, in which the first replica is molded polystyrene and

the final replica is evaporated silica. Since the best metallurgical replicas have been prepared this way, the procedure will be discussed in detail.

The metal specimen must be prepared with a high degree of polish, preferably an electrolytic polish and etched very lightly. For steel specimens, etching in a 0.1 percent nital solution for fifteen seconds is generally sufficient. Following the etching, it is desirable to mold the specimen in polystyrene with as little delay as possible. Granulated polystyrene is placed above the specimen surface in sufficient quantity to give the completed mold a thickness of about 1/2 inch. To avoid deformation of the surface by pressure of hard particles, it is essential to heat the mold to about 120°C before applying pressure. The pressure is slowly increased to about 3,500 pounds while the mold temperature is raised to 160°C. The mold is then slowly cooled under constant pressure until the temperature drops below 80°C. This is a necessary precaution to prevent air bubble formation.

To remove the metal specimen from the mold all excess polystyrene is first sawed away from the sides. If the metal has been properly etched, the polystyrene can be separated from the specimen by a light tap. If the specimen exhibits appreciable undercutting, it is necessary to dissolve it in acid.

The evaporation of silica onto the polystyrene replica is accomplished by normal evaporation technique (Plate 17). A tungsten spiral cone is closely wound of 30 mil wire. Convenient dimensions for the cone are a 30° apex angle and a depth of about 6 to 8 millimeters. The cone is filled with splinters of fused quartz or preferably powdered quartz in the form of Santocell. To melt the silica, the current through the tungsten spiral is slowly raised to about 25 amperes. The evaporation is then carried out by bringing the current up to 40 amperes. The proper film thickness is obtained by holding the styrene six inches from the spiral and evaporating for five seconds. Exceptionally good vacuum is not absolutely essential. Successful evaporation is obtained with pressures as high as one micron of Hg.

The property which makes silica so ideally

suited to this replica method is its high mobility on polystyrene and on silica itself. After evaporation, the sides and back of the styrene as well as the molded face are uniformly covered. The undesired portions are freed of silica by rubbing with emery cloth and the replica surface is cut in 1/8 inch squares (Plate 17). The back of the styrene block is then cemented to a small plate of glass with the help of some ethyl bromide and placed in a petri dish containing sufficient ethyl bromide to cover the silica surface. In a few minutes, the silica squares are freed by the solvent and float to the surface. Side illumination with a red filter under the dish aids in observing them. The bulk of the styrene on the glass plate is then removed and the silica films may be caught on the mesh supporting screens. The task of catching the silica squares while they are in ethyl bromide, however, is quite difficult. The ethyl bromide is highly volatile and near its boiling point at room temperature, keeping the squares in constant motion. The problem is simplified by diluting the ethyl bromide with absolute alcohol. In this mixture, the squares can be caught much more easily. A final wash in ethyl bromide clears away any polystyrene still adhering to the silica.

The high contrast and resolution obtainable with the polystyrene-silica method are illustrated in Plates 18-22.

CHAPTER VI

Electron Micrographs

Various electron micrographs are shown in the Plates. These are for the most part self-explanatory, but some additional details are mentioned in the following paragraphs.

Plates 7 and 8 are electron micrographs of metallic smokes. In each case, the smoke particles have characteristic shapes. The particles of zinc oxide smoke have the form of four spikes making angles of 1$\frac{1}{2}$0 degrees with each other. Magnesium and cadmium oxide smokes are small cubes. Tungsten oxide smoke exhibits a variety of cubic and rhombohedral shapes. Aluminum oxide smoke consists of minute spherical globules. Information of this type is interesting in relation to the chemistry of combustion. In regard to toxic and irritant smokes, many of the properties are strongly dependent on the physical shape of the particles.

The aluminum oxide monohydrate of Plate 9 is in the form of very thin scale called diaspore. The particles are unusual for their uniformity of shape and size.

The specimens of Plate 10 (A, B, C) were prepared by chemical precipitation. A very fine precipitate of lead chromate was obtained by reacting concentrated lead acetate solution with concentrated sodium dichromate. When first prepared, the precipitate appeared as in the micrograph of Plate 10 (B). After digesting for one day, the precipitate had formed the crystals shown in Plate 10 (C). The barium sulphate crystals of Plate 10 (D) were prepared by adding barium chloride solution to sulphuric acid.

Diatomaceous earth is commonly used as a filler for paints and as filter material. The minute holes in these specimens measure less than one-half the wavelength of visible light Plate 11, (B, C).

For optimum optical properties, the particle size of paint pigments must be in the neighborhood of the wavelength of visible light. Optical microscopes show almost no distinguishable differences between pigments of different types, but the electron microscope reveals true shapes and particle sizes. The contrast between optical and electron micrographs is illustrated by Plate 12.

Plate 13 shows electron micrographs of supposedly identical chemical substances which behaved very differently in their application. In

each case, the micrographs show that the behaviors are attributable to particle shape differences. Two arsenic insecticides differ considerably in their effectiveness. The better insecticide as shown in (A) is a plate-like structure having great covering power. The relatively ineffective compound (B) is granular. Optical micrographs revealed no differences. In another instance, a calcium carbonate formed by precipitation, exhibited unusual chemical activity. Plate 13 (C) shows that the large particles (unresolved optically) are aggregates of fine crystals, giving an over-all corrugated effect. The surface area of such particles is far greater than that of the ordinary calcite form of calcium carbonate shown in Plate 13 (D).

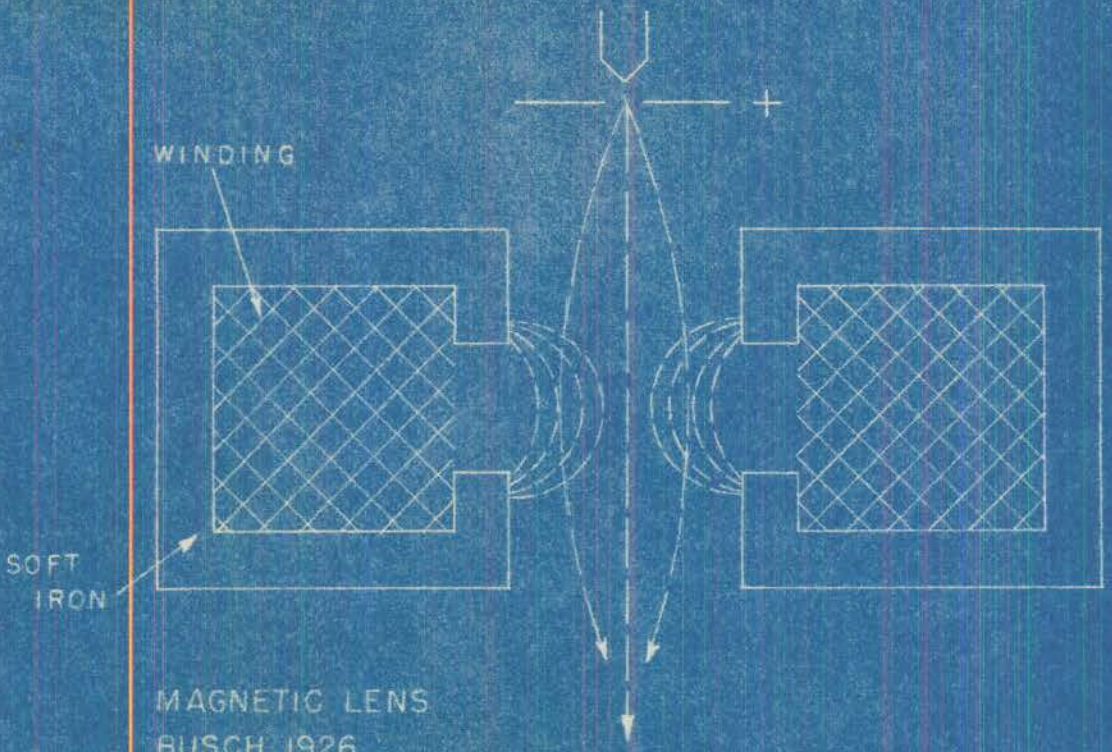
The effectiveness of finely divided carbon as a reinforcing agent of rubber depends directly on its fineness of division. The most common source of finely divided carbon is the partial combustion of natural gas. Plate 14 (A) is an electron micrograph of carbon black caught above a benzene flame. Similar micrographs show that the distributions from various parts of the flame are very different. Carbon black from a camphor flame is very homogeneous, the individual particles measuring about two millionths of an inch in diameter.

Some interesting electron micrographs have been obtained of the structure of plastics. Plate 14 (B) shows the unusual structure of cuprene. The vinylite in 15 (A) is only slightly polymerized. In this form, it is used for the manufacture of hemp rope. The koroseal of Plate 15 (B) is an artificial rubber obtained by more complete polymerization of vinyl chloride. The minute dark specks appearing throughout the structure are of the order of 30 angstroms in diameter and may represent single molecules. For Plate 15 (C) the polystyrene was prepared in the form of a thin film. Where the film is broken, thread-like fibers, 30 to 50 angstroms in diameter are evident. In 15 (D) a ruptured strip of thin polystyrene ribbon also shows a thread-like structure at the tear.

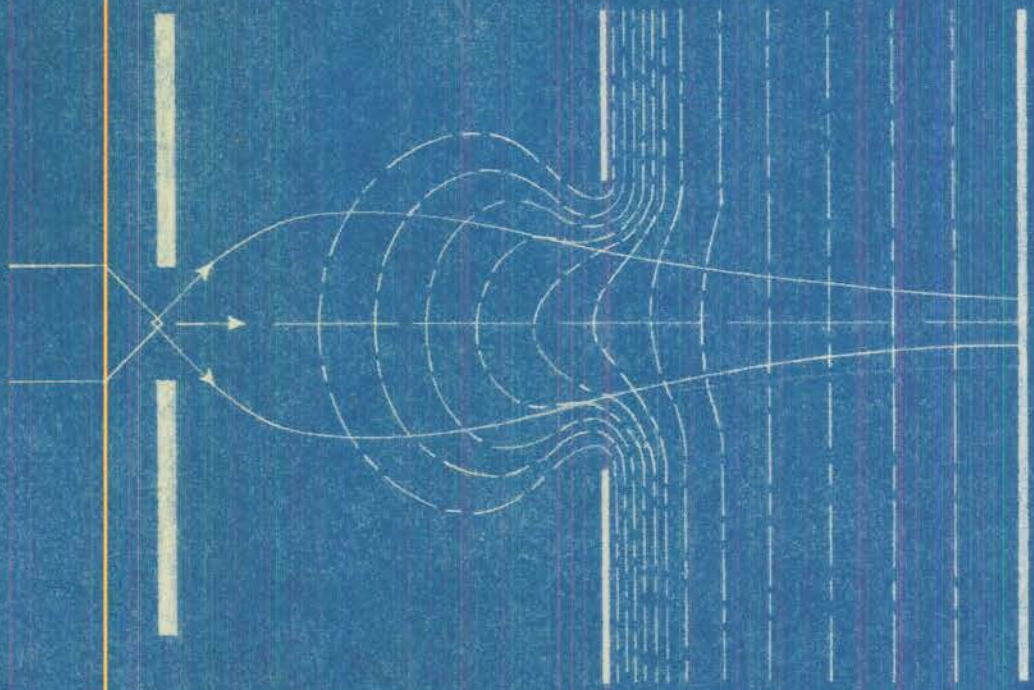
Plates 18 to 22 are electron micrographs of silica replicas of armor plate steel. The advantages over optical metallography are evident from comparison with the optical micrographs at 750x. The micrographs illustrate effects of various heat treatments of the same steel sample. In three cases, the specimen was heated to 1550°F and

then oil quenched. Plate 21 shows the "as quenched" specimen. Tempering for two hours at 500°F yielded the structure of Plate 20. Tempering at 800°F gave the structure of Plate 22. The pearlitic structures were derived by furnace cooling from 1595°F. The entire electron micrograph of Plate 18 corresponds to one of the dark unresolved patches in the optical micrograph.

Plates 24 and 25 are electron diffraction patterns of the Debye-Scherrer type, taken with the diffraction adapter for the R. C. A. microscope. Patterns (B) and (C) of Plate 24 and the pattern of Plate 25, identify the crystal structure and chemical composition of the particles shown in Plate 9, Plate 8 (A), and Plate 7.



MAGNETIC LENS
BUSCH 1926



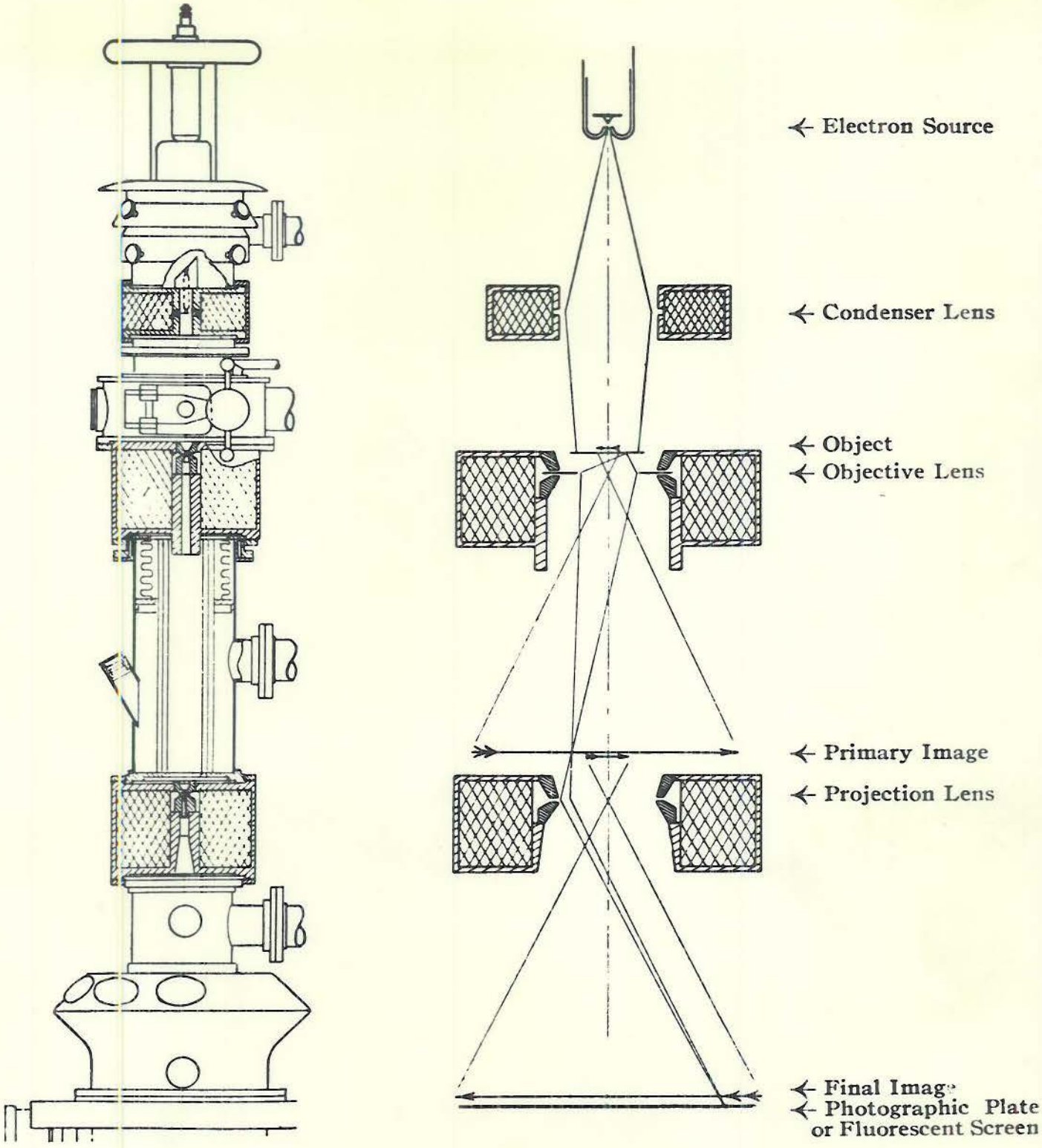
ELECTROSTATIC LENS

MAGNETIC AND ELECTROSTATIC LENSES

PLATE I

ELECTRON MICROSCOPE ROOM AT
NAVAL RESEARCH LABORATORY

PLATE 2



The electron microscope.

On the left is a scale drawing and on the right is a schematic diagram of the instrument showing the arrangement of lenses and the formation of images.

SCHEMATIC DRAWING OF
ELECTRON MICROSCOPE

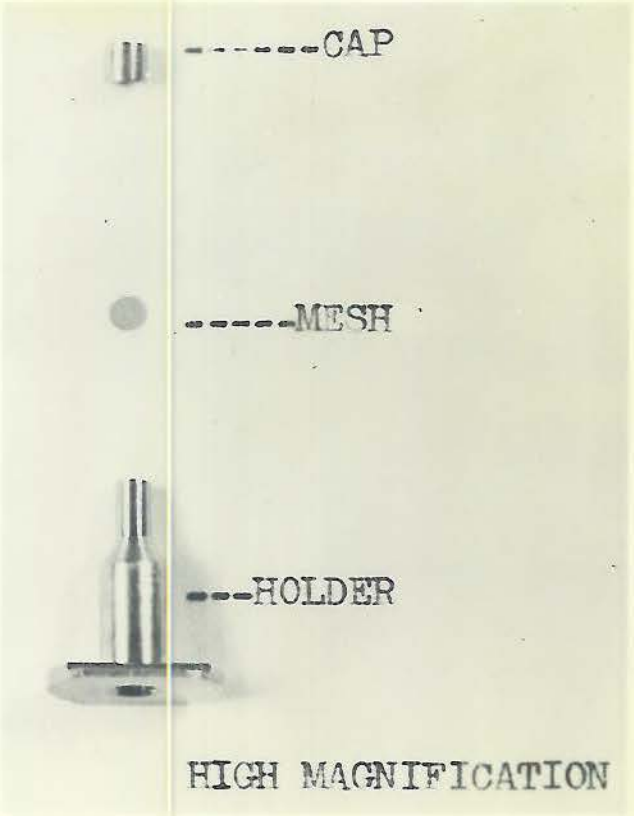
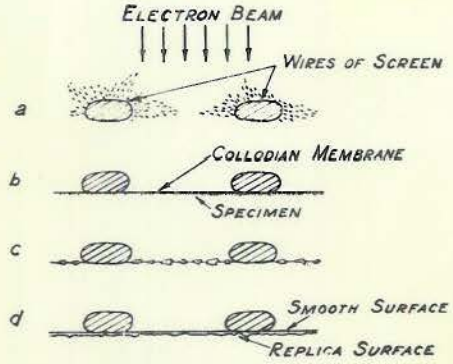


FIG.1 SPECIMEN HOLDER UNIT



Specimens prepared in different ways for the electron microscope: (*a*) by collection directly on screen; (*b*) by collection on a thin membrane; (*c*) by inclusion of specimen in membrane; and (*d*) by the preparation of a membrane, one surface of which is a replica of the solid surface to be studied. Note that membranes are mounted below the screen so that the screen protects the membrane from electron bombardment as much as possible.

FIG.2 METHODS OF SPECIMEN PREPARATION

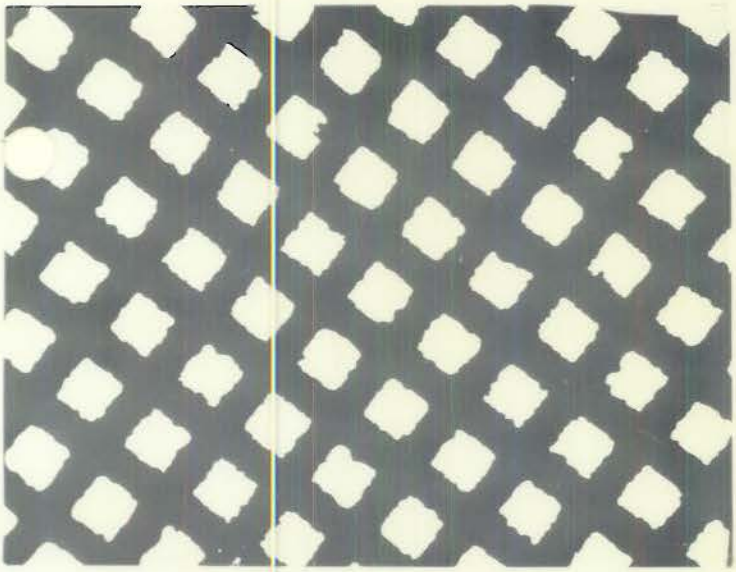


FIG.3 200 MESH NICKEL SCREEN (75X)

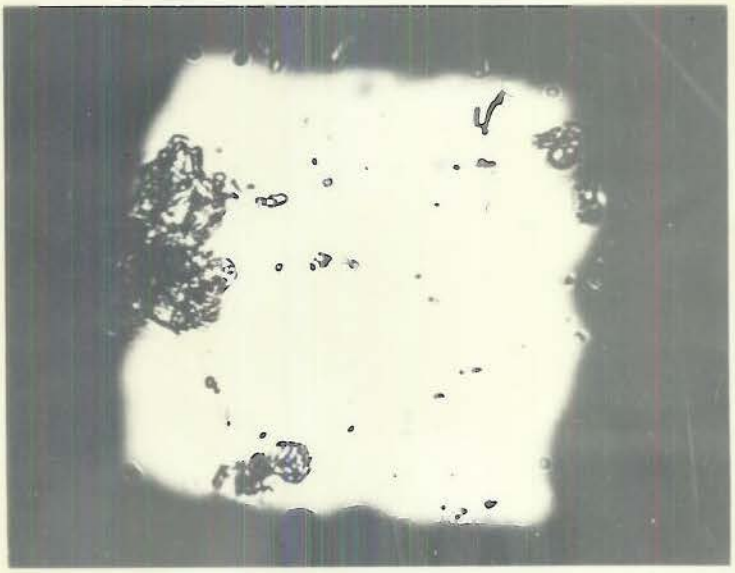
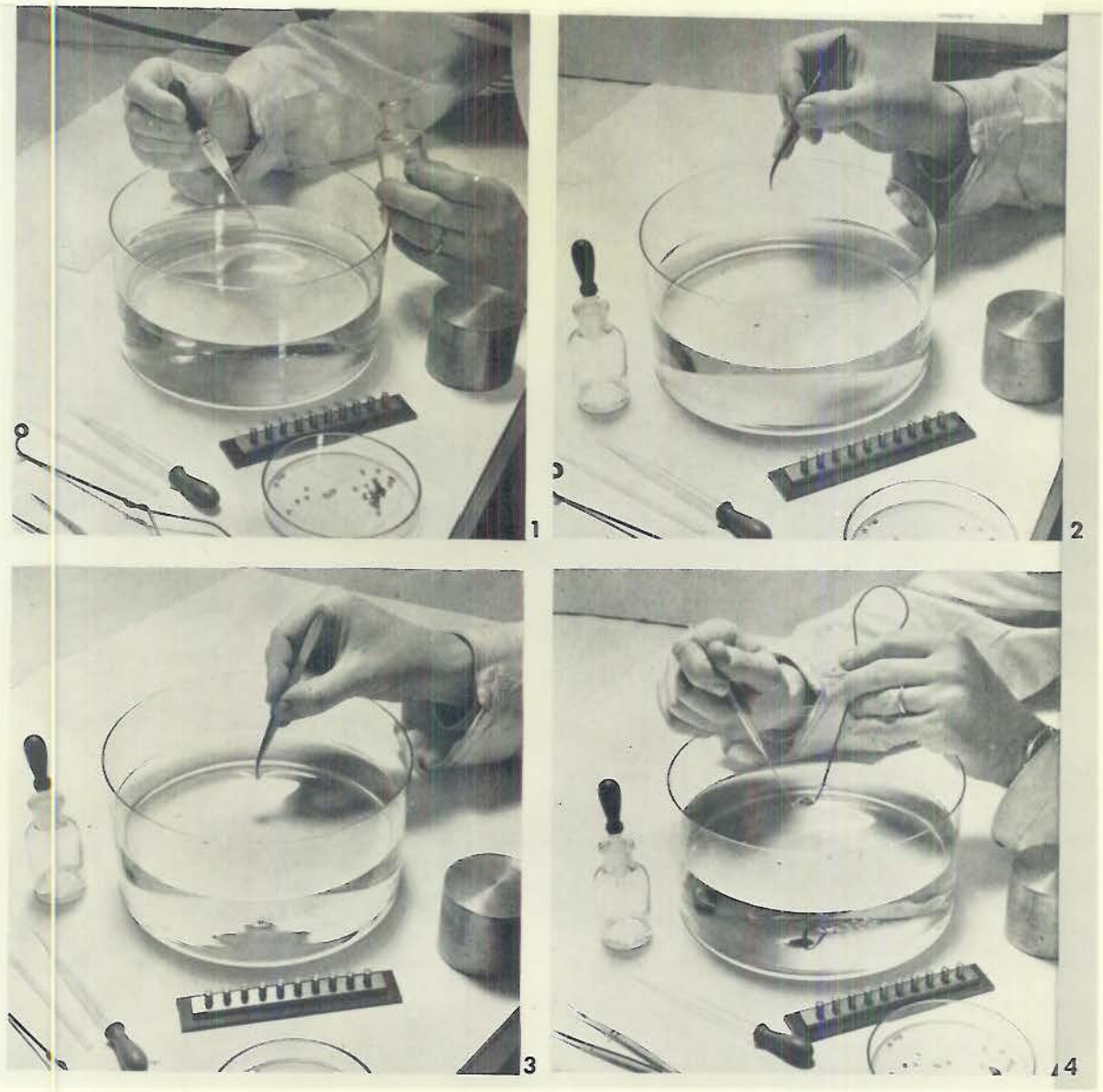


FIG. 4 OPEN AREA OF MESH CONTAINING SPECIMENS OF DIATOMACEOUS EARTH

SPECIMEN PREPARATION (STEPS 1-4)



1 - THIN FILM OF COLLODION IS PLACED OVER SURFACE OF WATER IN VESSEL BY MEANS OF A MEDICINE DROPPER

2 - TINY DISCS OF EXTREMELY FINE WIRE MESH ARE PLACED ON THE FILM OF COLLODION

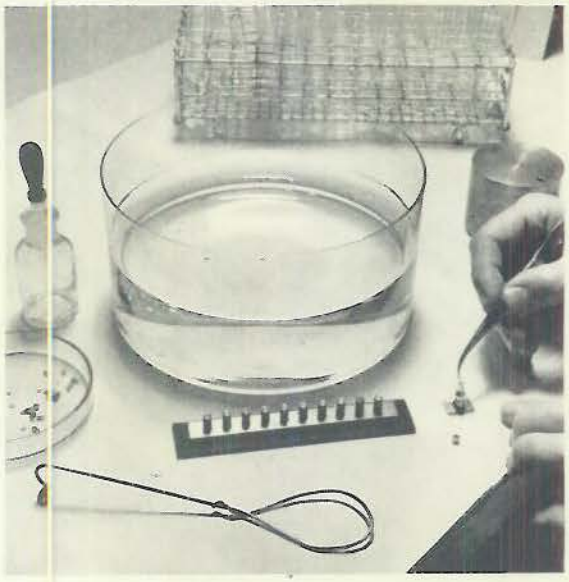
3 - EACH DISC IS GENTLY PRESSED DOWN SO THAT A FILM OF COLLODION WILL ADHERE TO THE MESH

4 - SPECIAL TOOLS ARE USED TO FREE AND LIFT THE COLLODION-PLATED DISCS.

SPECIMEN PREPARATION (STEPS 5-8)


5


6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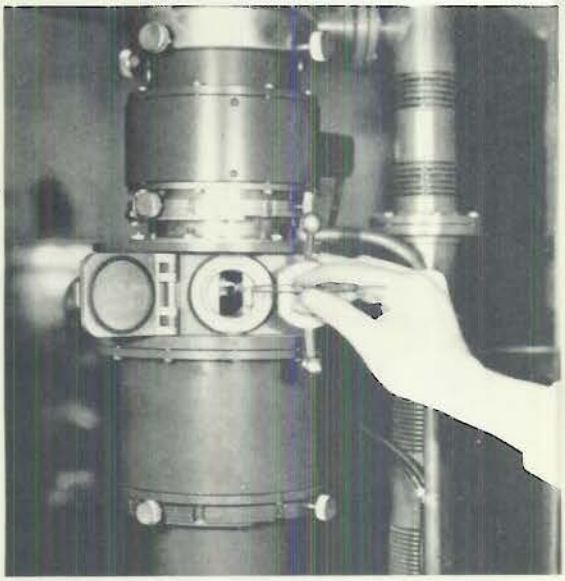

8

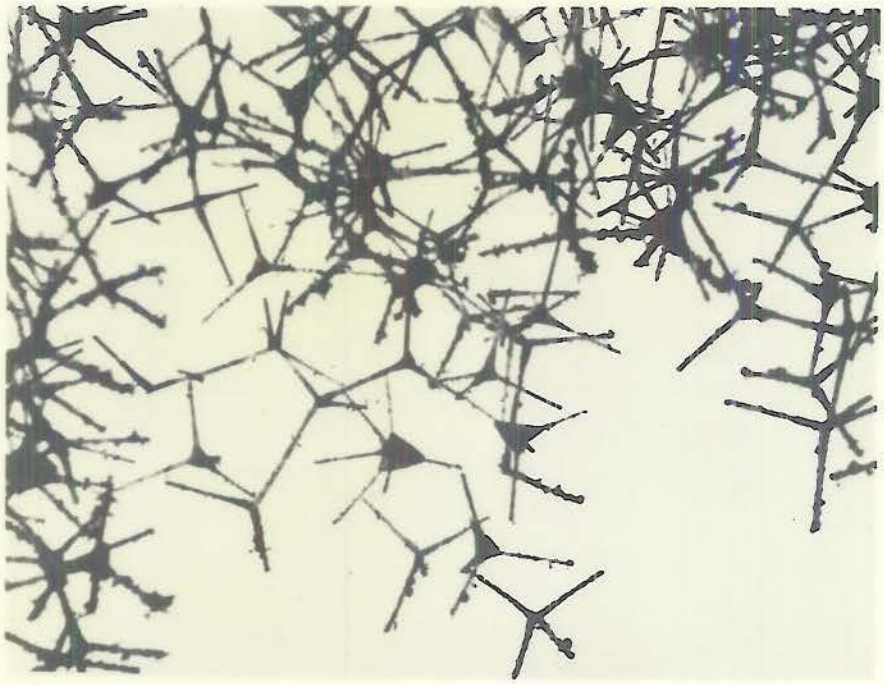
5- COLLODION-PLATED DISC IS PLACED ON SMALL CYLINDRICAL PEDESTAL AND EXCESS WATER IS DRAWN OFF

6- TECHNICIAN APPLIES SOLUTION CONTAINING SPECIMEN TO THE MOUNTED DISC.

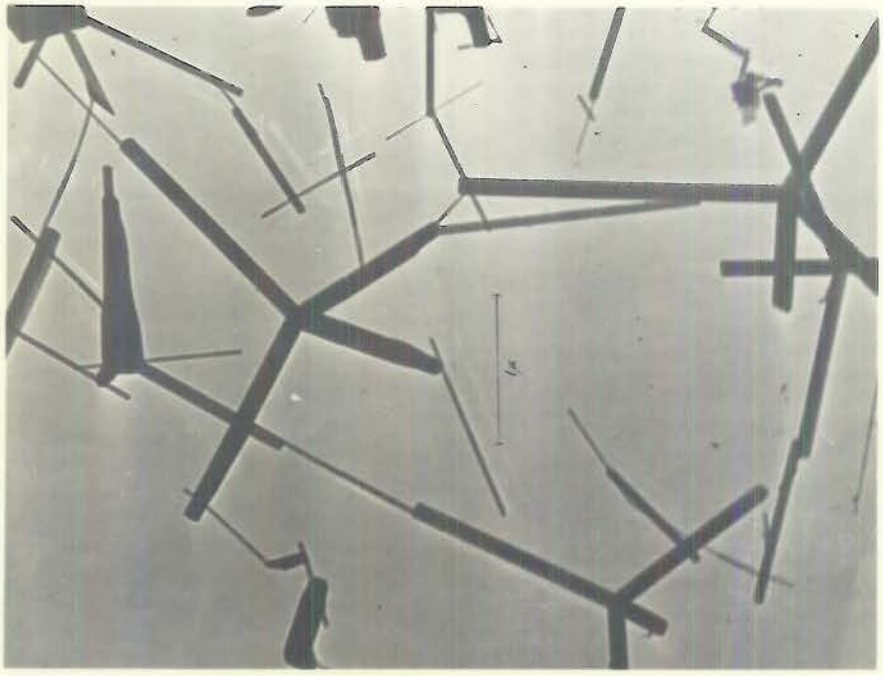
7- DISC IS THEN PLACED OVER OPENING ON END OF THE MICROSCOPE CARTRIDGE

8- CARTRIDGE IS LOADED INTO OBJECT CHAMBER OF THE MICROSCOPE.

ZINC OXIDE SMOKE - PARTICLES ADHERING TO MESH WITHOUT SUPPORTING FILM

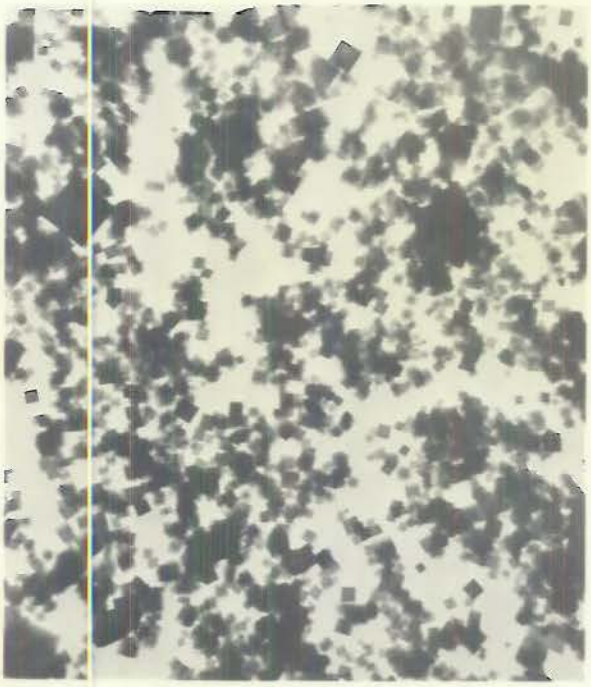


A - PARTICLES CAUGHT CLOSE TO CRUCIBLE - (15,000X)

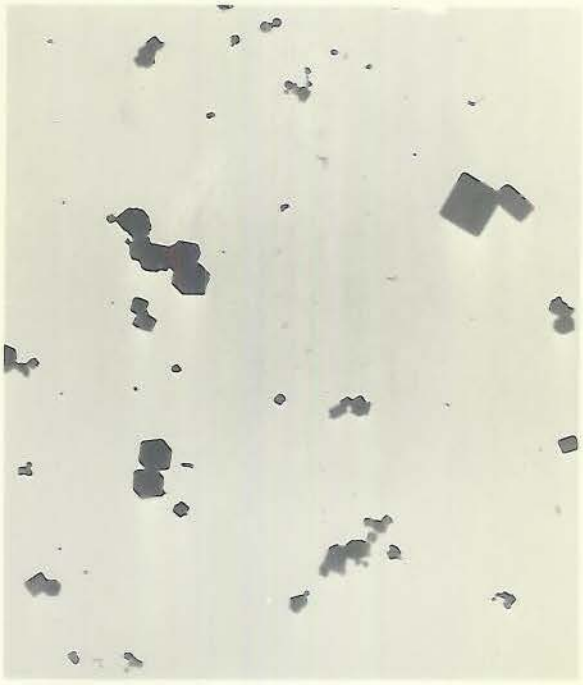


B - PARTICLES CAUGHT HIGH ABOVE CRUCIBLE (30,000X)

SMOKES

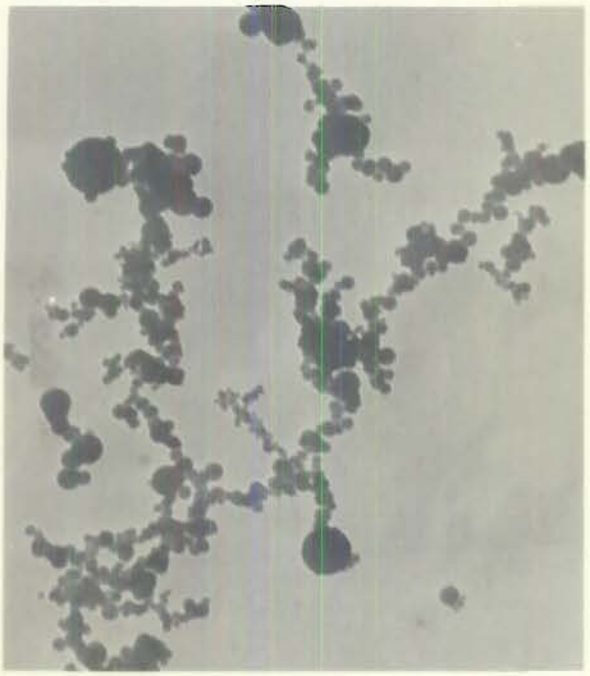


A MAGNESIUM OXIDE (14,000X)



B- CADMIUM OXIDE (18,000X)

C- TUNGSTEN OXIDE (20,000X)



D - ALUMINUM OXIDE (25,000X)

ALUMINUM OXIDE MONOHYDRATE (40,000X)
DEPOSITED ON PARLODION FILM
FROM WATER SUSPENSION

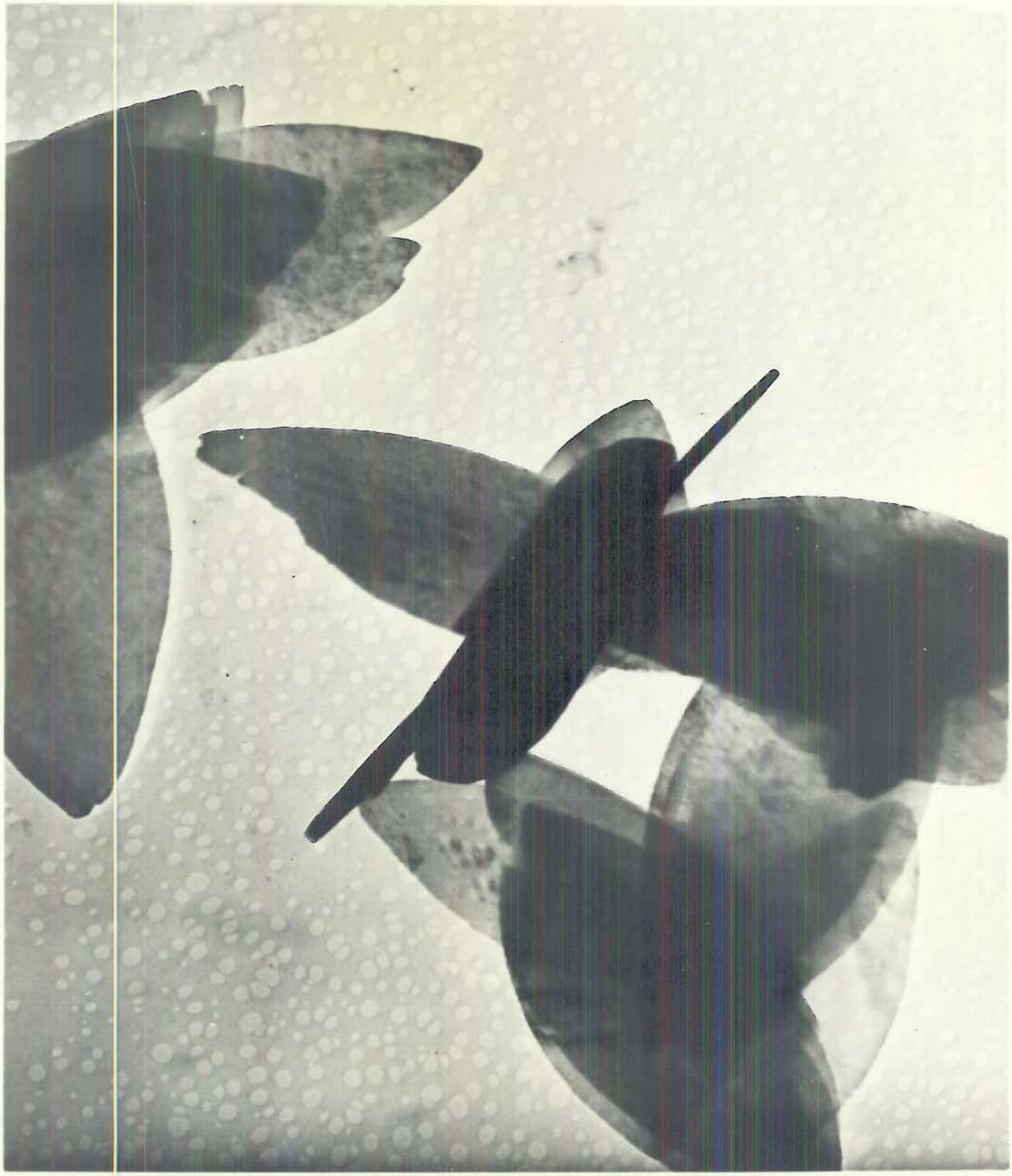
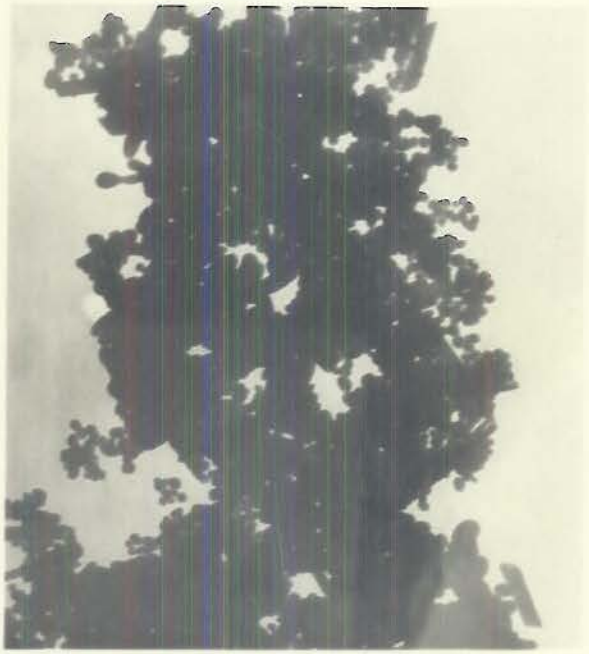


PLATE 9

PRECIPITATED CRYSTALS - DEPOSITED ON FORMVAR FILM FROM WATER SUSPENSION

A- LEAD CHROMATE (OPTICAL 675X) CRYSTALS WELL DEVELOPED.

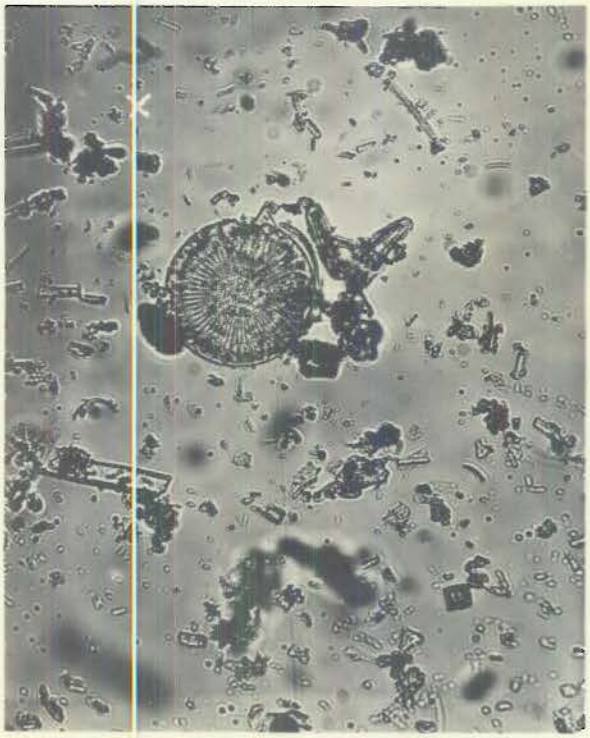


B- LEAD CHROMATE (ELECTRON MICROGRAPH 23,000X) BEFORE APPRECIABLE CRYSTAL GROWTH

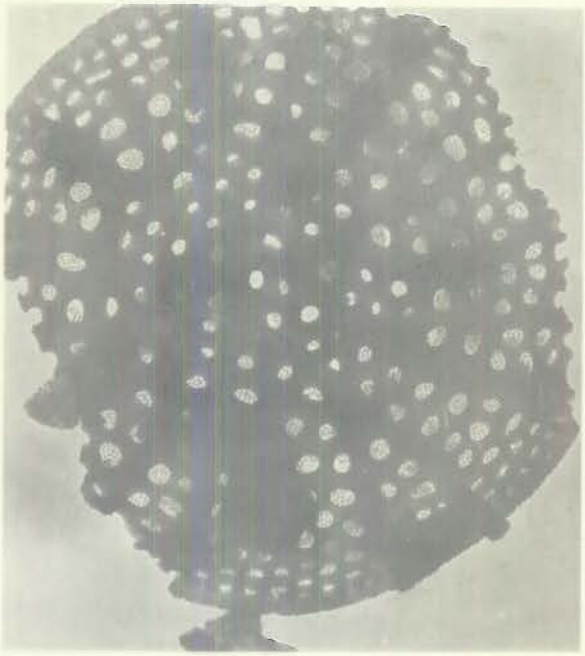
C- LEAD CHROMATE - CRYSTALS AFTER I DAY GROWTH (ELECTRON MICROGRAPH - 13,000X)

D- BARIUM SULPHATE CRYSTALS (ELECTRON MICROGRAPH - 11,000X)

DIATOMACEOUS EARTH DISPERSED IN FORMVAR FILM



A- OPTICAL MICROGRAPH (350X)



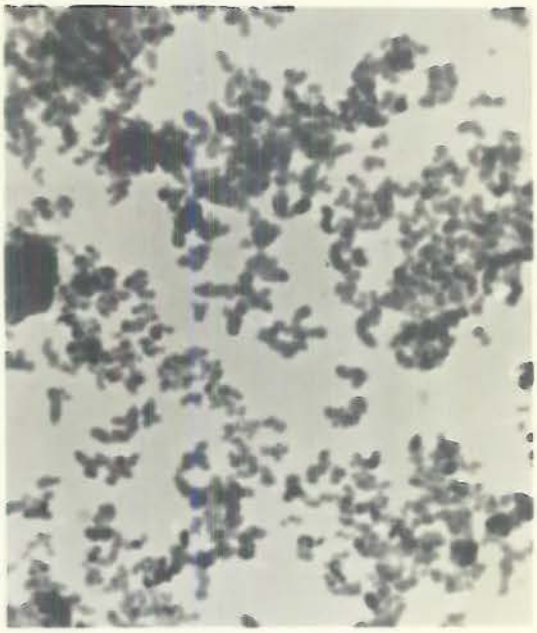
B- ELECTRON MICROGRAPH (18,000X) OF PARTICLE X IN FIG. A

C- ELECTRON MICROGRAPH (20,000X)

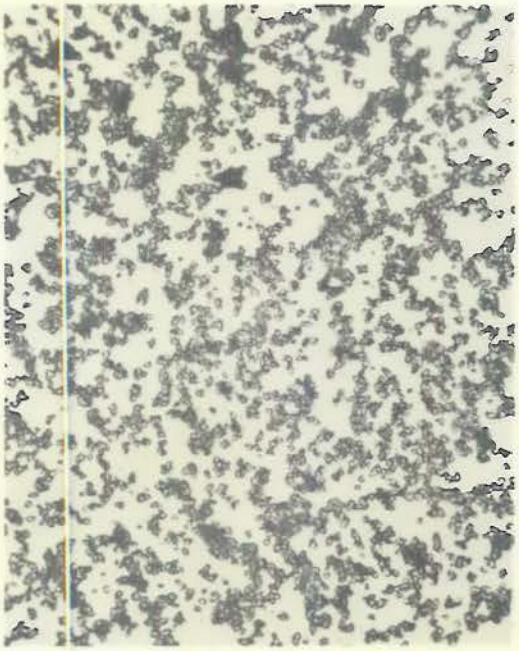
PLATE II

PAINT PIGMENTS

A- IRON BLUE
(OPTICAL MICROGRAPH 675X)



B- IRON BLUE (ELECTRON MICROGRAPH 25,000X) DISPERSED IN FORMVAR

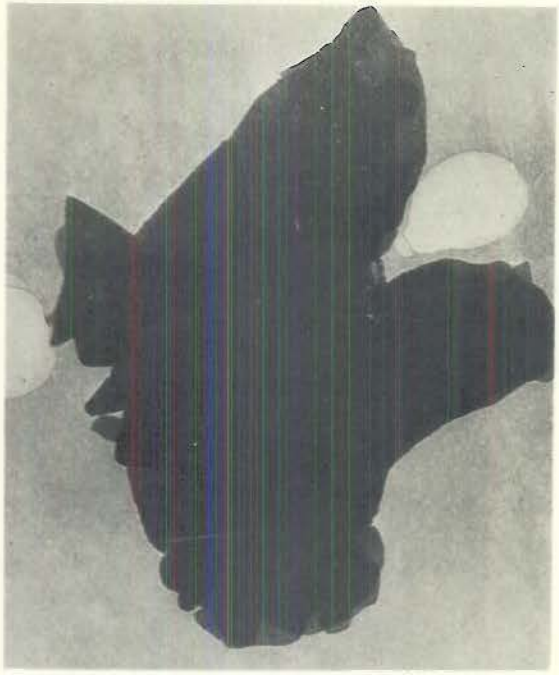


C- ZINC YELLOW (OPTICAL MICROGRAPH 675X)

D- ZINC YELLOW (ELECTRON MICROGRAPH 10,000X) PARTICLES DUSTED ONTO BERYLLIUM SUPPORTING FILM

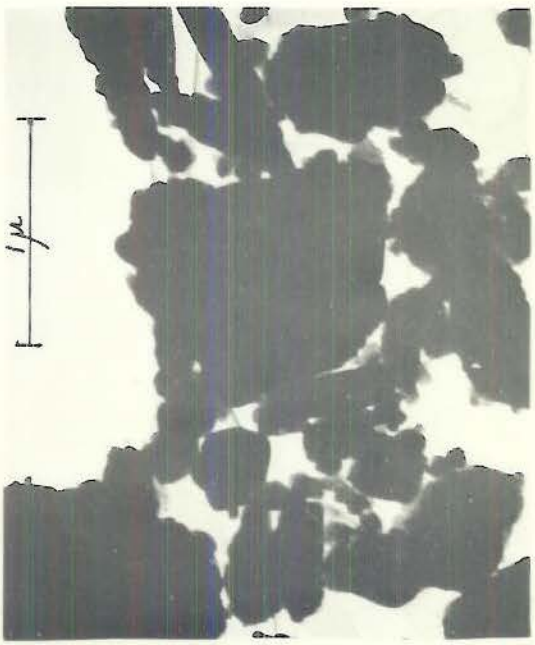
PLATE 12

A AN ARSENIC INSECTICIDE SHOWING A PLATE-LIKE STRUCTURE (25,000X)

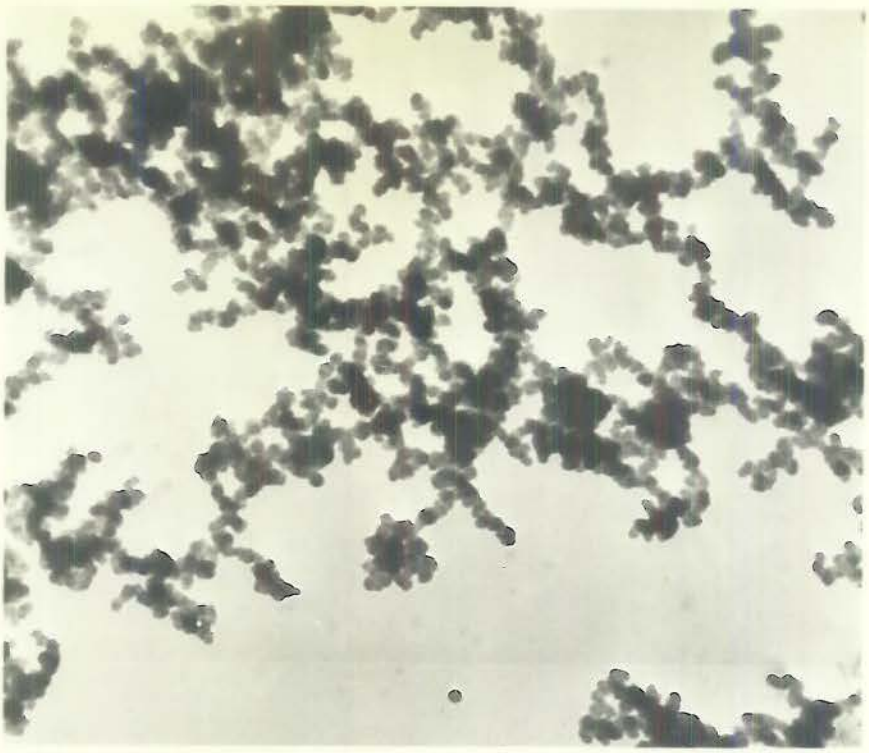


B- A COMPOUND OF ARSENIC WITH GRANULAR STRUCTURE.

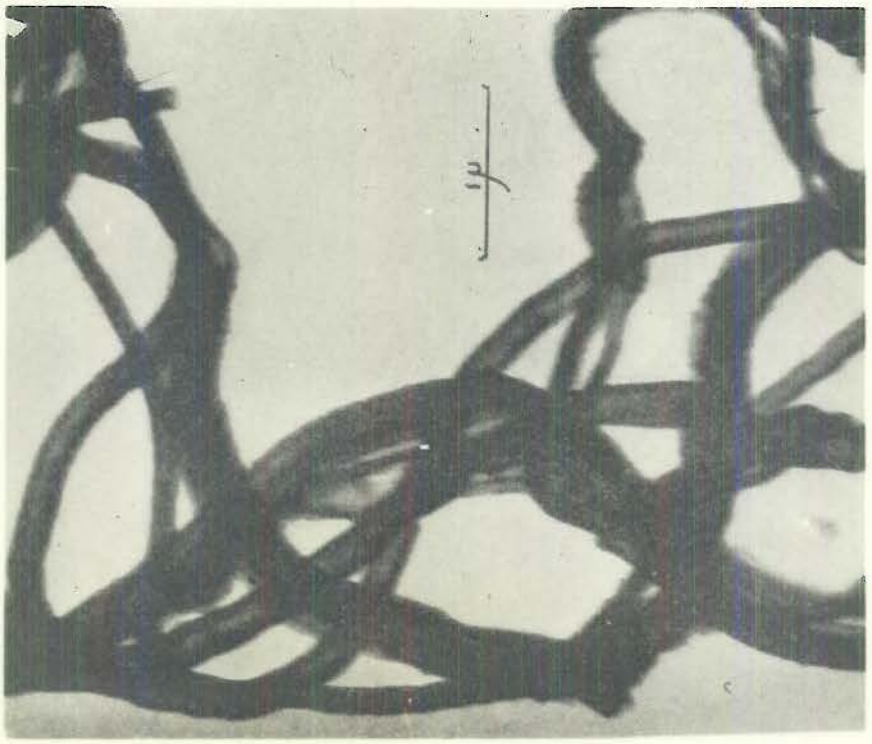
C- PRECIPITATED CALCIUM CARBONATE (30,000X)



D- CALCITE FORM OF CALCIUM CARBONATE (30,000X)



A- CARBON BLACK COLLECTED
FROM BENZINE SMOKE
(25,000X)

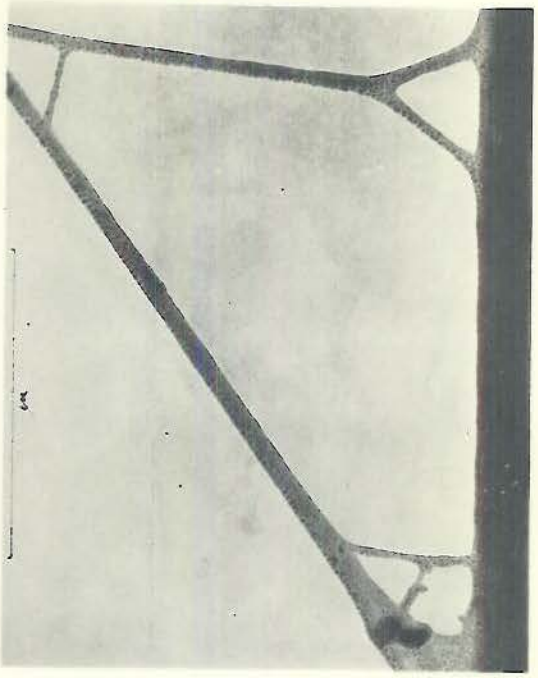


B- CUPRENE, A POLYMER OF
ACETYLENE FORMED IN THE
PRESENCE OF COPPER
(23,000X)

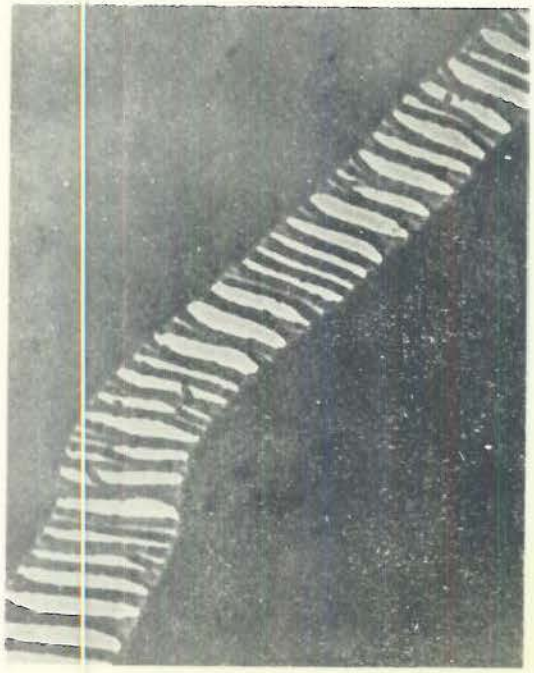
PLATE 14

PLASTICS

A- VINYL CHLORIDE PARTIALLY POLYMERIZED (15,000X)



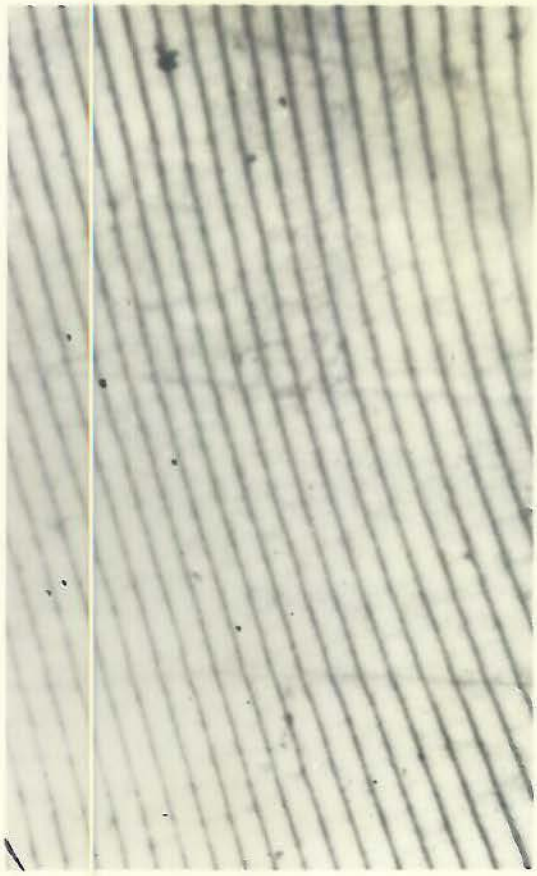
B- KOROSEAL (41,000X)



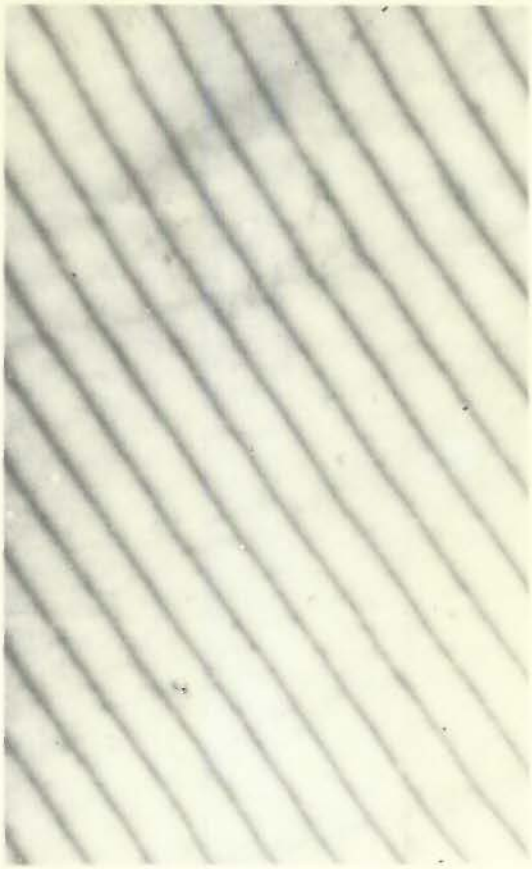
C- POLYSTYRENE (35,000X)

D- RUPTURED POLYSTYRENE RIBBON (20,000X)

FORMVAR REPLICAS OF DIFFRACTION GRATING



(2,000X)



(4,000X)

(26,000X)

PLATE 16

SILICA-POLYSTYRENE REPLICA METHOD

A- PRESS FOR MOLDING METALLIC SPECIMENS IN POLYSTYRENE

B- EVAPORATION OF SILICA ONTO POLYSTYRENE IMPRESSION

C- MARKING EIGHTH INCH SQUARES ON SILICA

D- SILICA REMOVAL IN ETHYL BROMIDE

PEARLITE IN STS ARMOR PLATE (7,500X)

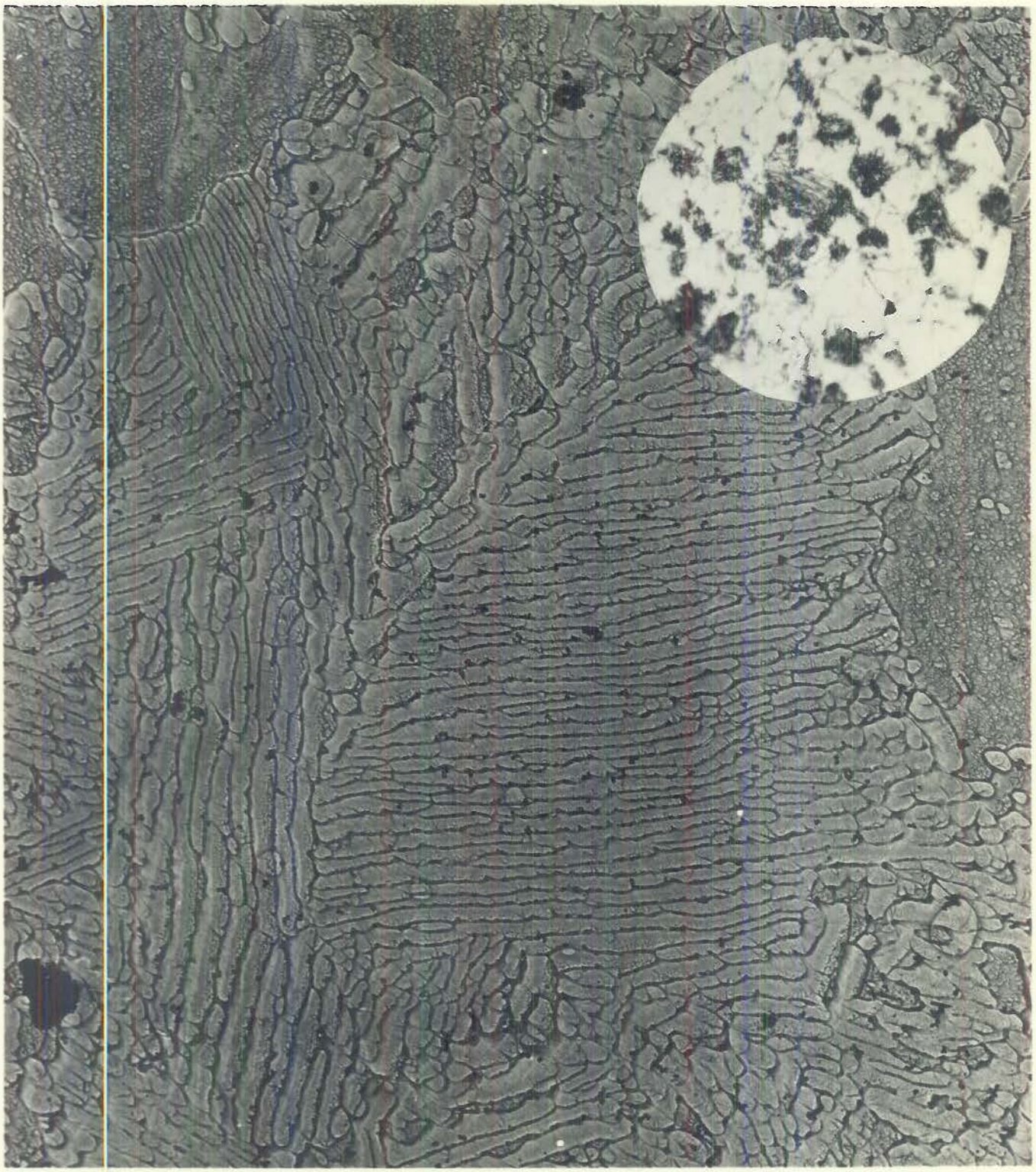


PLATE 18

PEARLITE IN STS ARMOR PLATE (40,000X)

PLATE 19

MARTENSITE IN STS ARMOR PLATE(7,500X)

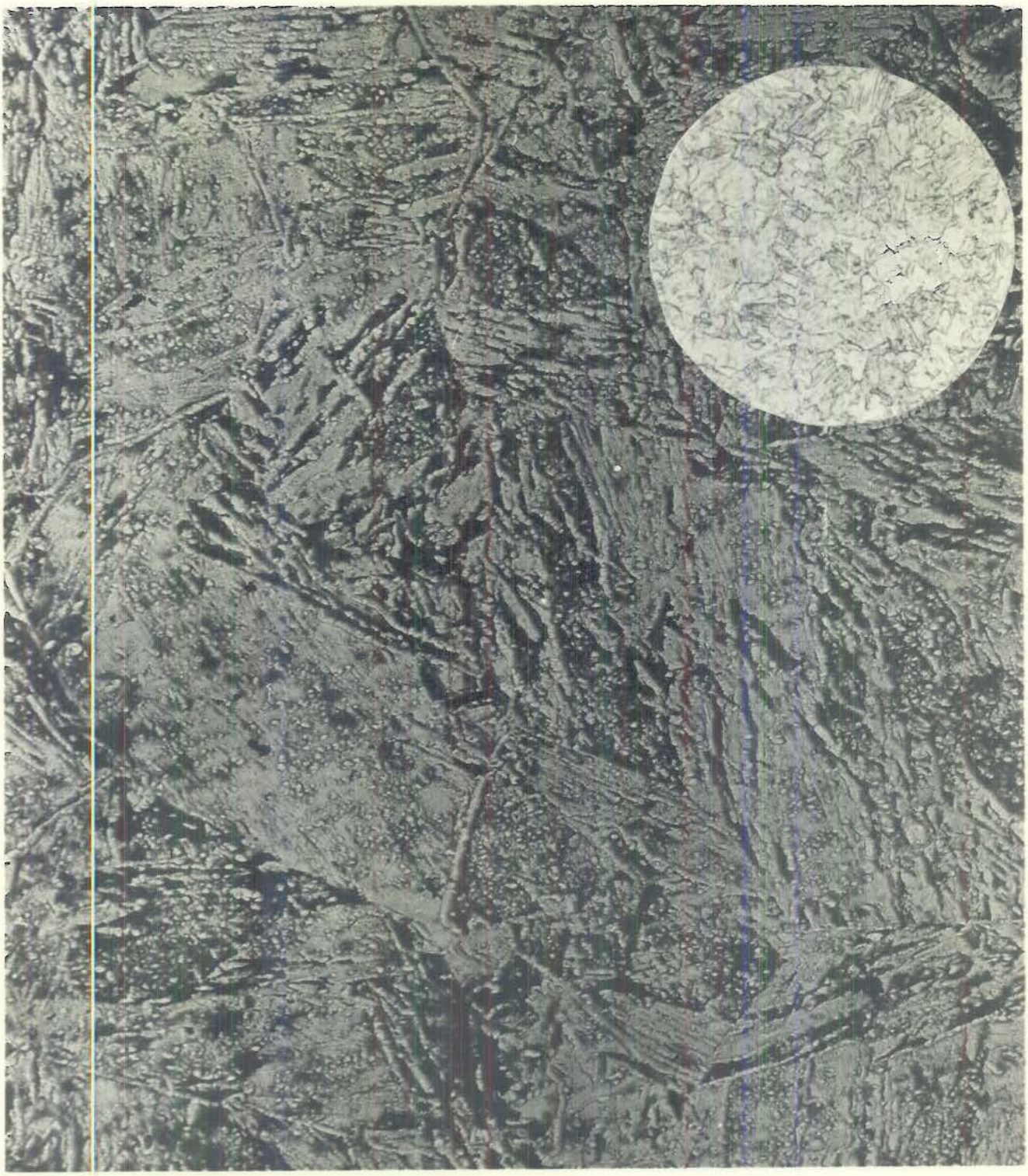


PLATE 20

MARTENSITE IN STS ARMOR PLATE (7,500X)

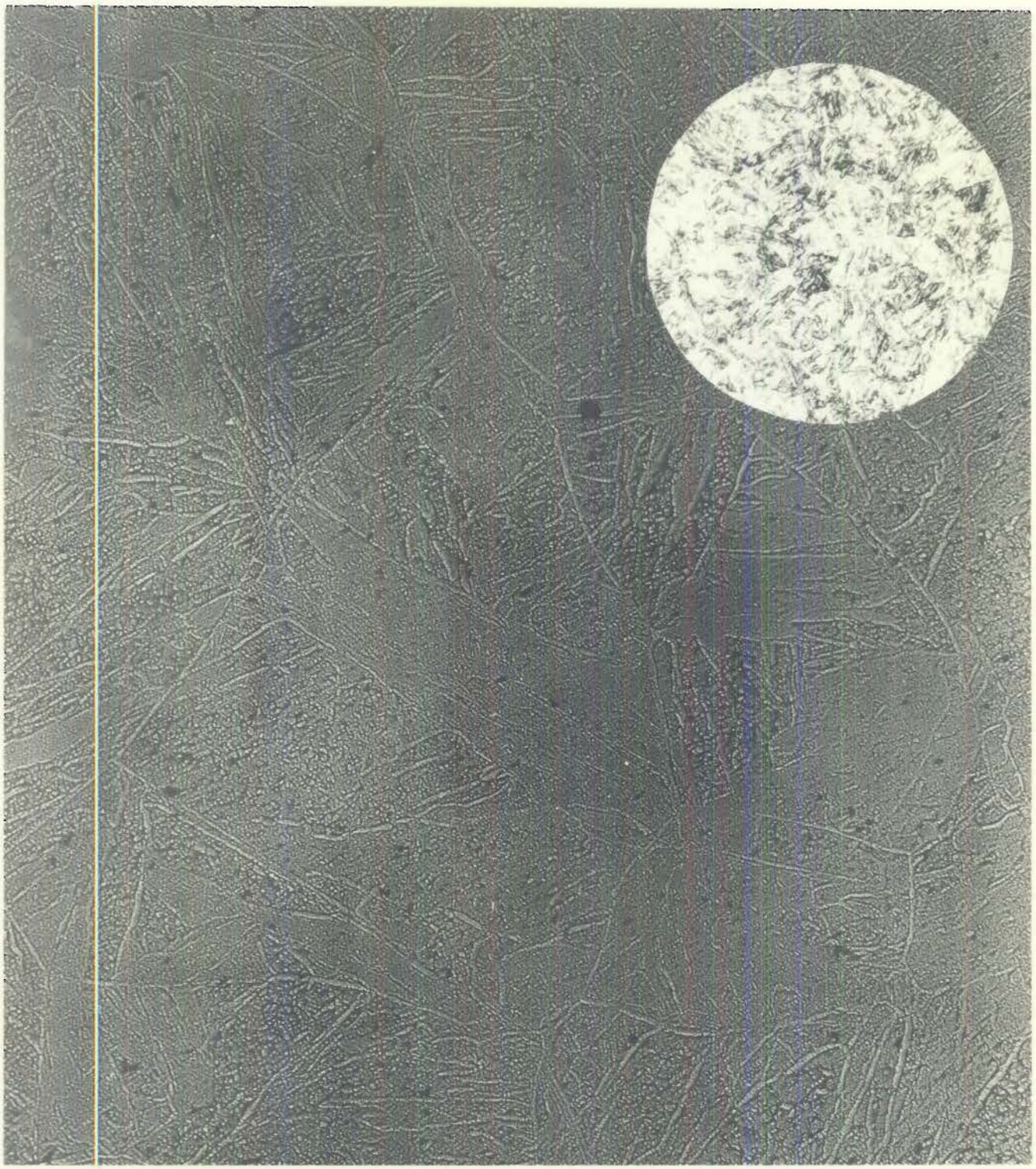


PLATE 21

GRAIN BOUNDARY JUNCTION IN STS ARMOR PLATE (60,000 X)

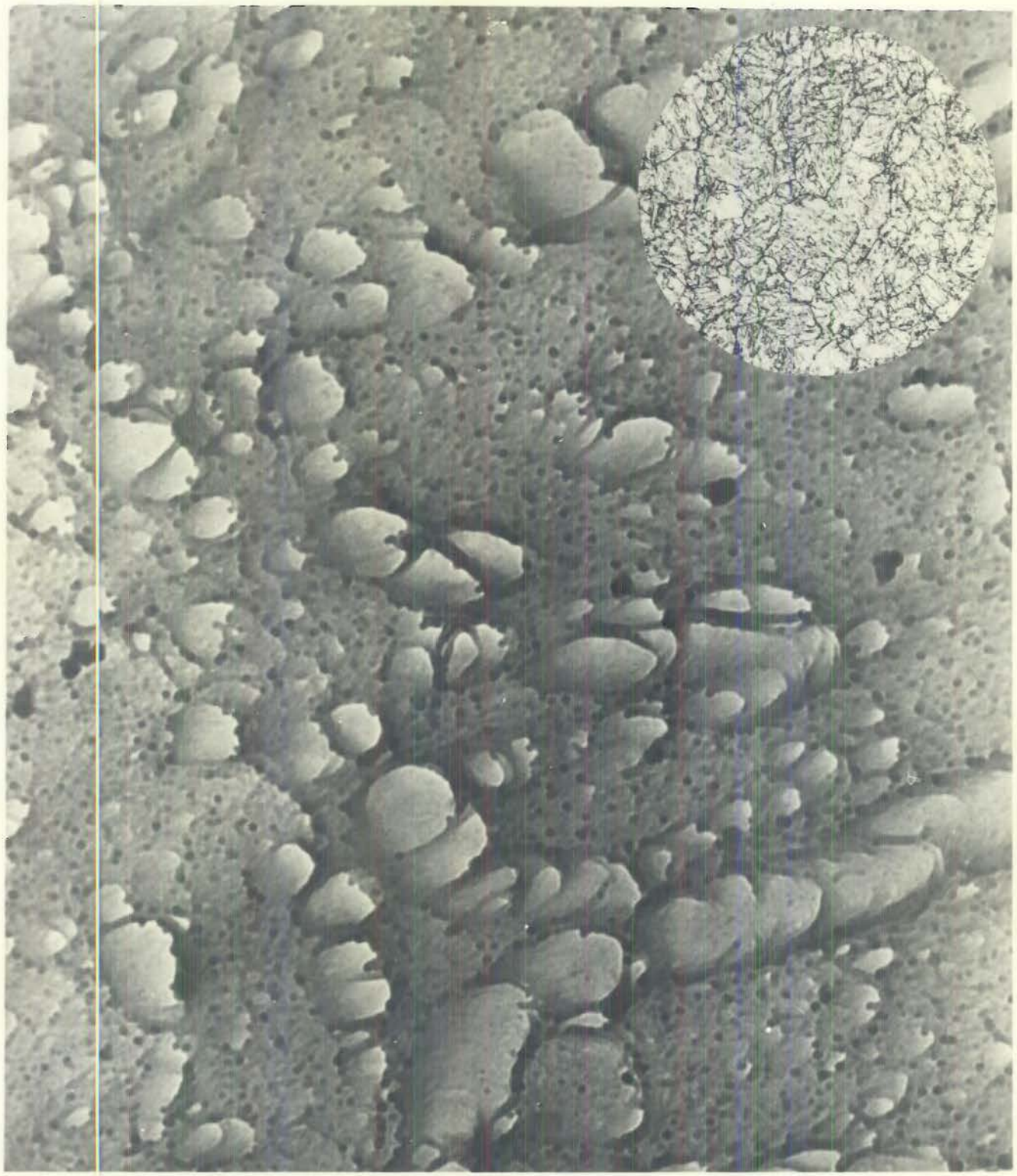
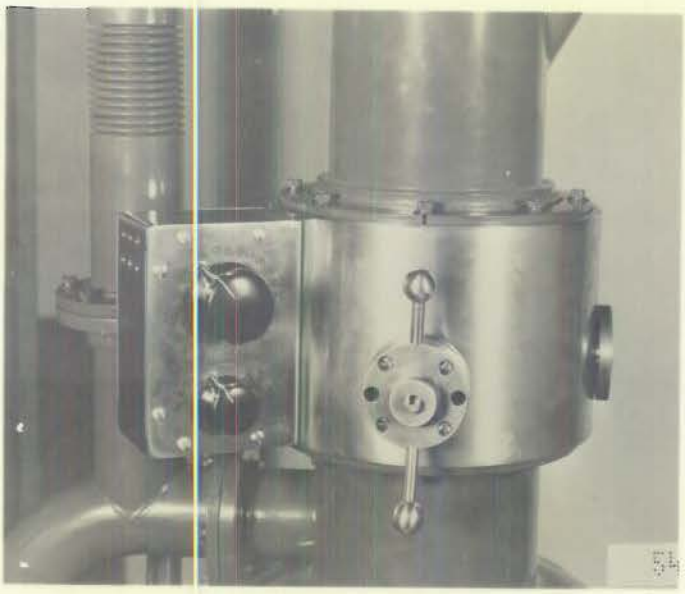


PLATE 22

ELECTRON DIFFRACTION ADAPTER FOR R.C.A. ELECTRON MICROSCOPE



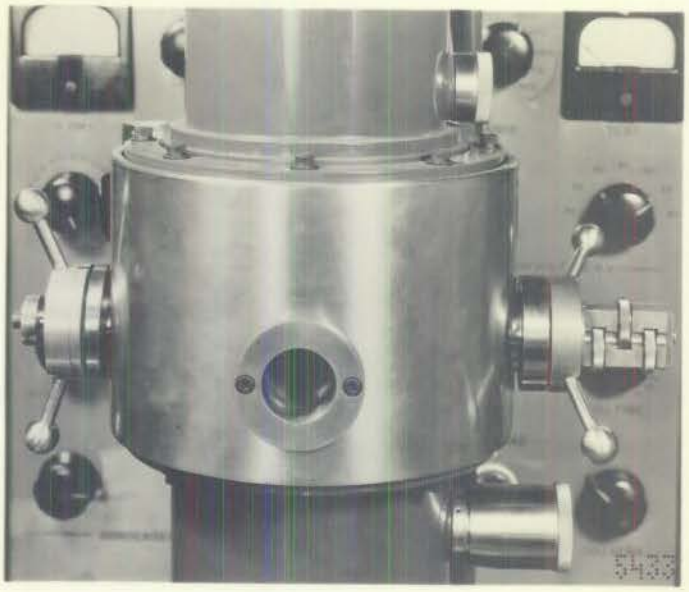
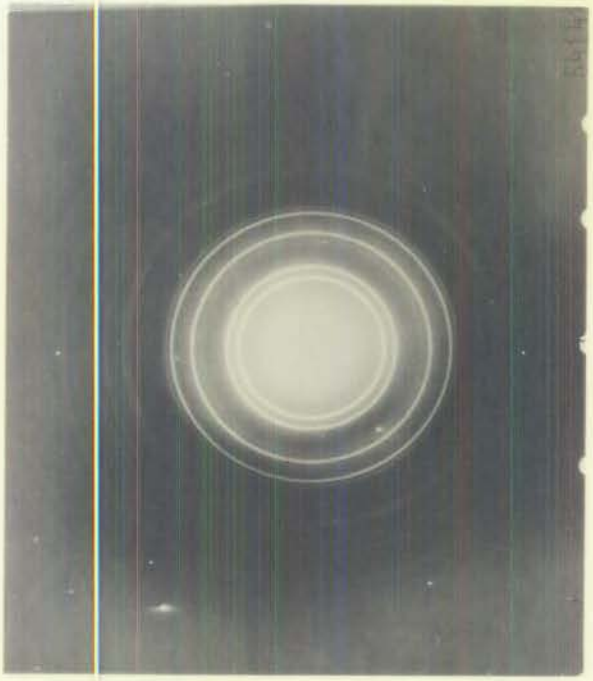
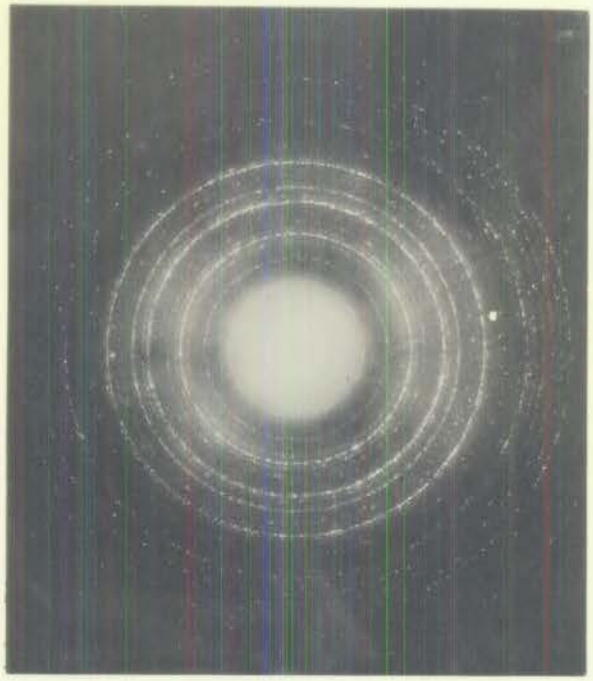


PLATE 23

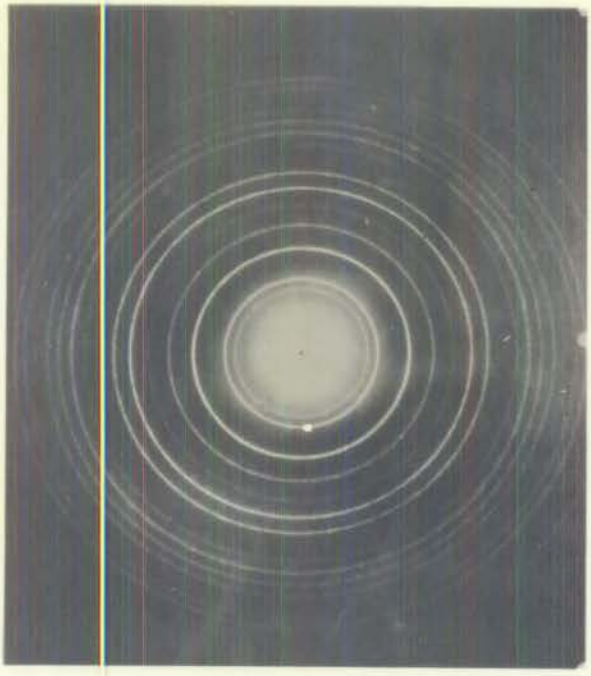
ELECTRON DIFFRACTION PATTERNS



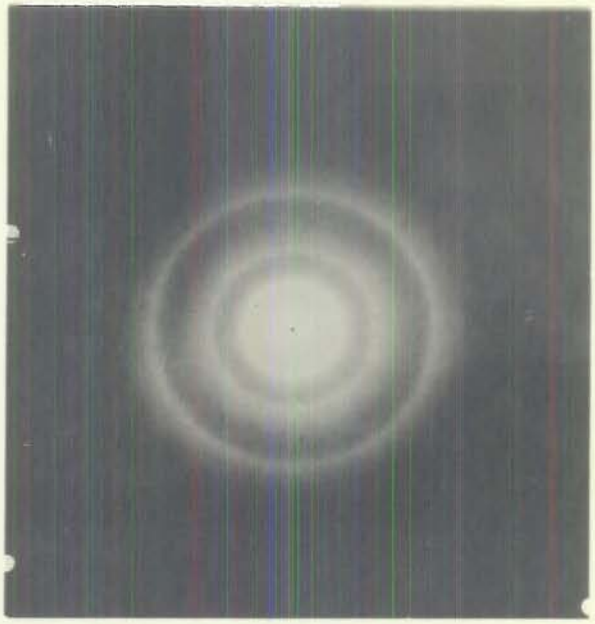
A- ALUMINUM FILM EVAPORATED ONTO NITROCELLULOSE



B - ALUMINUM OXIDE MONOHYDRATE



C - MAGNESIUM OXIDE SMOKE



D- CARBON BLACK OBTAINED BY BURNING CAMPHOR IN AIR.

ELECTRON DIFFRACTION PATTERN
OF ZINC OXIDE SMOKE

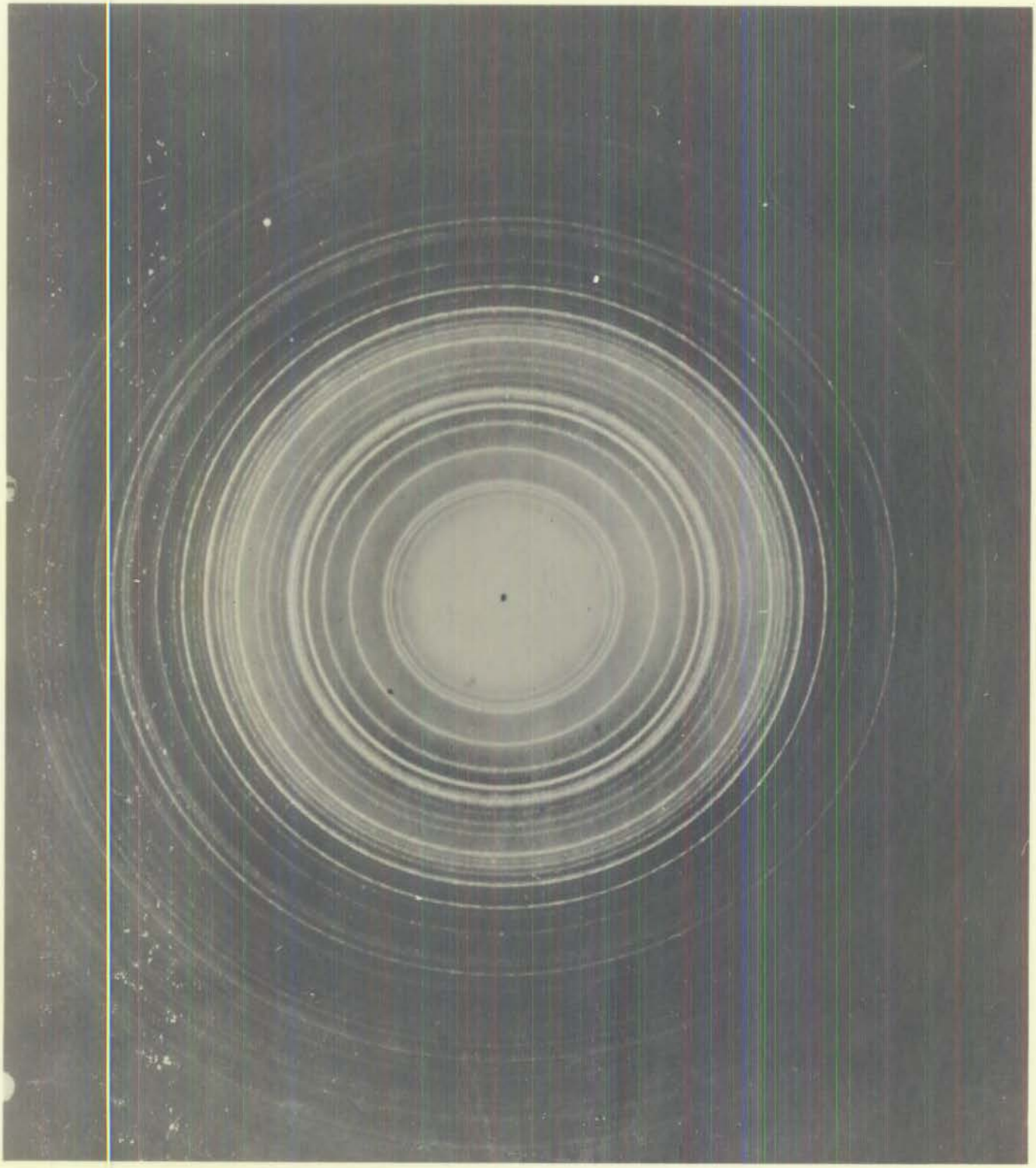


PLATE 25