EPA-650/2-75-036

ASBESTOS FIBER ATLAS

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Research Grant 801336
Program Element No. 1AA010

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Prepared for

U. S. ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF RESEARCH AND DEVELOPMENT
WASHINGTON, D. C. 20460

April 1975

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Publication No. EPA-650/2-75-036

PREFACE

Asbestos or asbestiform minerals include several types or groups of fibrous crystalline substances with special thermal and electrical properties that have long encouraged their use in the manufacture of such products as roofing, insulation, brake linings, fireproof curtains, etc. Their occurrence as pollutants in the ambient air and in supplies of food and drinking water has caused considerable concern because occupational exposures to asbestos have been found to induce mesothelioma of the pleura and peritoneum, as well as cancer of the lung, esophagus, and stomach, after latent periods of about 20 to 40 years.

Transmission electron microscopy, often together with selected area electron diffraction, has been the principal technique used to identify and characterize asbestos fibers in ambient air and water samples. Because of the poor sensitivity of other analytical methods, electron microscopy is also being used for routine measurement of airborne or waterborne asbestos concentrations, although it is ill-suited for this purpose. Even with the future development of more appropriate quantitative procedures, however, electron microscopy in combination with electron diffraction will continue to be valuable as a reference method and particularly for research applications, e.g., in support of health effects studies where maximal information on fiber counts and size distributions are needed.

In publishing this asbestos fiber atlas consisting of transmission electron micrographs and electron diffraction patterns obtained during a study supported by Research Grant 801336, the Environmental Protection Agency seeks to provide microscopists and others involved in the analysis of asbestos with a convenient guide to fiber identification.

Jack Wagman
Grant Project Officer

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ABSTRACT

Transmission electron micrographs and corresponding selected area electron diffraction patterns of standard specimens of serpentine and amphibole asbestos are presented for use by analysts as an aid in identification. Micrographs and diffraction patterns of typical ambient air samples and of certain minerals that often occur with airborne asbestos are also included. Specimens were uniformly prepared and examined in a single electron microscope.

ACKNOWLEDGMENTS

The authors wish to thank those who made mineral specimens available, and to thank R. J. Amouroux, L. Carpenter, J. Murchio, and S. C. Tocchini for their assistance in the preparation of this atlas.

GLOSSARY

Amosite	An amphibole mineral that is an iron-rich variety of anthophyllite, occurring in long fibers. $Fe_5Mg_2Si_8O_{22}(OH)_2$
Amphibole	A mineral variety belonging to a group of minerals with essentially like crystal structures involving a silicate chain and generally containing three groups of metal ions, the large ions being sodium and calcium, the intermediate being chiefly bivalent iron, magnesium, and manganese, and the small ions chiefly silicon with some aluminum and, rarely, ferric iron.
Anthophyllite	An orthorhombic mineral of the amphibole group that is often clove brown in color and lamellar or fibrous, and is essentially a magnesium ferrous silicate. $(Mg,Fe)_7Si_8O_{22}(OH)_2$
Antigorite	A brownish green lamellar variety of the mineral serpentine. $Mg_3Si_2O_5(OH)_4$
Asbestos	A mineral that readily separates into long flexible fibers suitable for uses where incombustible, nonconducting, or chemically resistant material is required.
Crocidolite	A lavender-blue or leek-green mineral of the amphibole group that is a variety of riebeckite and occurs in silky fibers and in massive and earthy form. $Na_2O \cdot Fe_2O_3 \cdot 8\ SiO_2 \cdot H_2O$
Chrysotile	A fibrous, silky variety of the mineral serpentine. $Mg_3Si_2O_5(OH)_4$
Lizardite	A variety of the mineral serpentine. $Mg_3Si_2O_5(OH)_4$
Serpentine	A rock composed of chrysotile and antigorite often in layers with or without other minerals, having usually a dull green color often with a mottled appearance or a red or brownish hue; occurring in masses (as antigorite) or in fibrous form (as chrysotile). $Mg_3Si_2O_5(OH)_4$
Tremolite	A mineral of the amphibole group that is white or gray calcium magnesium silicate and occurs in long blade-shaped or short stout crystals and also in columnar, fibrous, or granular masses. $Ca_2Mg_5Si_8O_{22}(OH)_2$

ASBESTOS FIBER ATLAS

INTRODUCTION

Purpose

This atlas consists of transmission electron micrographs (TEM) and corresponding selected area diffraction patterns of serpentine and amphibole asbestos to be used by the analyst as an aid in identification. Micrographs of certain minerals known to occur with asbestos in the air and some typical ambient air samples are also included.

Sources of Standards

Mineral specimens were collected through the offices of three individuals. Dr. LeRoy Balzer, Department of Environmental Health Sciences, School of Public Health, University of California, Berkeley, kindly supplied amosite, anthophyllite, chrysotiles, and crocidolite. Dr. Norman Page supplied specimens of antigorite and clinochrysotiles from the collection of the U.S. Geological Survey in Menlo Park, California, and lizardites from the Department of Geology and Geophysics, University of California, Berkeley. Mr. H. L. Weber of Fiberboard Paper Products Corporation, Emeryville, California, kindly supplied the amosite-Penge. Tremolite was purchased from Baker and Adamson, calcite and magnesite from J. T. Baker, brucite from Matheson, Coleman and Bell, and quartz from Research Organic/Inorganic Chemical Co., Sun Valley, California. In all cases, the identity of these compounds was confirmed using X-ray diffraction.

Preparation and Arrangement of Micrographs

Most of the asbestos standards were received already ground. However, three samples of lizardite were received as small pieces of rock. It was therefore necessary to grind a small piece of each of these samples in a small agate mortar and pestle. A portion of each ground sample was transferred to a glass microscope slide which had been placed into a covered plastic Petri dish.

Electron microscope grids of 300-mesh nickel were covered with a thin layer of collodion. The asbestos specimens were mounted by lightly touching the collodion-covered grid to the sample in the Petri dish. An amount sufficient for electron microscopy adhered to the collodion after the excess had been removed by tapping the grid held with forceps.

The electron microscope used was the Siemens I with selected area diffraction capability. Most transmission micrographs were obtained at a magnification of 20,000. Two prints taken at 40,000 magnification show the characteristic internal capillary of chrysotile. The selected area diffraction micrographs represent a circular area 1 μm in diameter centered in the corresponding transmission micrographs.

The serpentines are described before the amphiboles, followed by the associated minerals, gold, and ambient air samples. Within each class the minerals are sequenced in alphabetical order. The micrographs, which are contact prints of the plates, are numbered consecutively in order of appearance. A description of important morphologic and diffraction pattern characteristics of the standard asbestos samples precedes each group of plates. Additional detail has been published elsewhere.[1-5]

The air samples were collected at several sites (see p. 47). In all cases, after collection of particulate matter, the filters were ashed, and the residue was dispersed in water and deposited on a polycarbonate (Nuclepore) disc by filtration. The disc was transferred to a microscope grid, the polycarbonate was dissolved; and the fibers were counted, identified, and sized as described[6] in AIHL Method 38.

Discussion

Asbestos is identified by ascertaining the fiber morphology and diffraction pattern. Characteristically, chrysotile exhibits a central channel running longitudinally through the fibril. Identification of asbestos by morphology alone is possible only for chrysotile. To avoid confusion with similar non-asbestos fibers, substantial information about the source of the sample should be available. Confirmation should be made by electron diffraction techniques.

This atlas enables the microscopist to differentiate between chrysotile and amphiboles. However, it is difficult to differentiate specific amphiboles by the use of the atlas alone. In using this atlas for identification, a preliminary selection should be made by comparison of the specimen with the bright field TEM plates shown for each asbestos type at the specified magnification.

Confirmation is then made by selected area electron diffraction utilizing techniques described in References 5, 7, and 8. Crystal lattice parameters aid in the identification of various asbestos types when compared with standard ASTM X-ray diffraction data referenced in the Table.

When the selected area includes many fibers or very large fibers, the diffraction pattern consists of a series of continuous rings resulting from the polycrystalline nature of the diffracted area. Interplanar spacings may be easily calculated for polycrystalline diffraction rings as follows:

The average instrument factor (K) is calculated by the formula $K=D \cdot d$, where D is the ring diameter (mm) in the diffraction pattern and d is a known interplanar space in Å. Gold is normally used in determining the instrument factor K, which for this atlas is 38.8 mmÅ, based on Figure 131. The actual interplanar spacing is then determined from the diffraction pattern where $d = \frac{K}{D}$, and compared with standard ASTM diffraction data.

Diffraction of a single fiber produces a spot pattern, rather than the ring pattern produced by many randomly oriented crystals. The single crystal pattern is interpreted by use of reciprocal lattices as described in Reference 5 or by diffraction pattern measurement and pattern recognition as described in Reference 7.

Table

REFERENCE NUMBERS TO ASTM FILES
ON X-RAY DIFFRACTION PATTERNS

Asbestos type	File No.	Fiche No.
Serpentines		
Antigorite, $Mg_3Si_2O_5(OH)_4$ (Non-asbestiform)	9-444 10-402 7-417	I-33-F1 I-37-B11 I-26-F1
Chrysotile (Clino) $Mg_3Si_2O_5(OH)_4$	10-380 10-381	I-37-B3
Lizardite, $Mg_3Si_2O_5(OH)_4$ (Non-asbestiform)	18-779	I-95-D5
Amphiboles		
Amosite, $Fe_5Mg_2Si_8O_{22}(OH)_2$	none	none
Anthophyllite $(Mg,Fe)_7Si_8O_{22}(OH)_2$	9-455	I-33-F6
Crocidolite, $Na_2O \cdot Fe_2O_3 \cdot 8\ SiO_2 \cdot H_2O$	none	none
Tremolite, $Ca_2Mg_5Si_8O_{22}(OH)_2$	13-437	I-54-E12
Associated minerals		
Brucite, $Mg(OH)_2$	7-239	I-25-D10
Calcite, $CaCO_3$	5-586	I-18-E4
Magnesite, $MgCO_3$	8-479	I-30-B10
α-Quartz, SiO_2	5-490	I-18-C2
Gold, Au	4-784	I-16-E11

ELECTRON MICROGRAPHS

AND

CORRESPONDING DIFFRACTION PATTERNS

ANTIGORITE

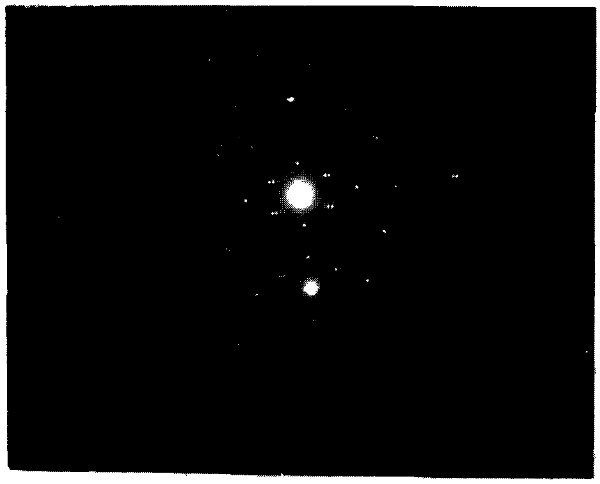
Electron Micrographs

The morphology is plate-like with some pointed material (Fig. 3). Some large fibers occur. The plate-like and fiber types seem to be made up of sheets and exhibit undulating striations. The fibers never consist of bundles of smaller fibrils, but have a sheet-like appearance.

Electron Diffraction Patterns

The diffraction patterns of antigorite are in hexagonal array with multiple spots of 3 at each diffraction point (Fig. 2, 4). Some patterns exhibit irregular parallel streaks (Fig. 6).

1



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3

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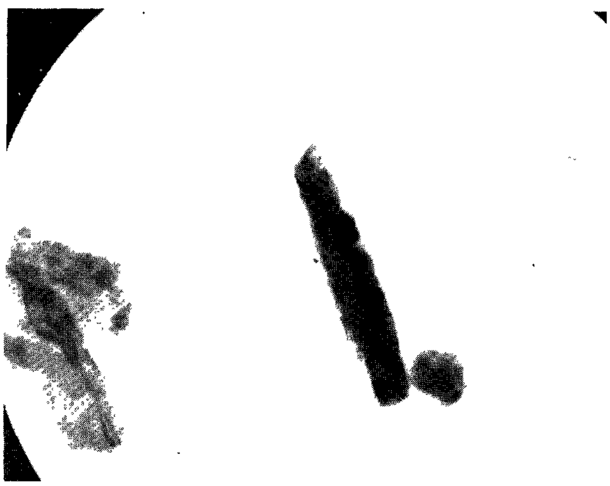


5

6

ANTIGORITE, 38-NA-62 USGS

1 μm

20,000X

CHRYSOTILE (ASBESTOS)

Electron Micrographs

Chrysotile, as the bright-field micrographs show, consists of single fibrils of small diameter and various lengths (Figs. 7, 9) and bundles of fibers of various diameters and lengths (Figs. 9,11). Occasionally a mass of fibers occurs in a single structure (Figs. 23, 25). Large tangles of single fibers also occur (Fig. 19).

A very important characteristic of the morphology is the occurrence of a channel running throughout the length of the individual fibrils (Figs. 7, 9, 11, 13, 15, 17, 21, 27, 31, 32). The range of the diameters of single fibrils is from 19 to 50 nm. The most frequent diameter[2] is 26 nm. The diameter of the internal channel ranges from 5 to 11 nm. The most frequent diameter of the tube[2] is 11 nm. An average fiber diameter of 37.5 nm has been reported for Jeffrey (Canada) chrysotile asbestos and 27.5 nm for New Idria (Coalinga, California) chrysotile asbestos[3]. The Coalinga asbestos fibers are described as overlapping fibrils 0.5 to 2 μm long cemented together, while the Canadian asbestos occurs as long individual fibrils[3].

Electron Diffraction Patterns

Diffraction patterns range from parallel streaks for single fibers (Figs. 12, 10), with or without a suggestion of concentric circles

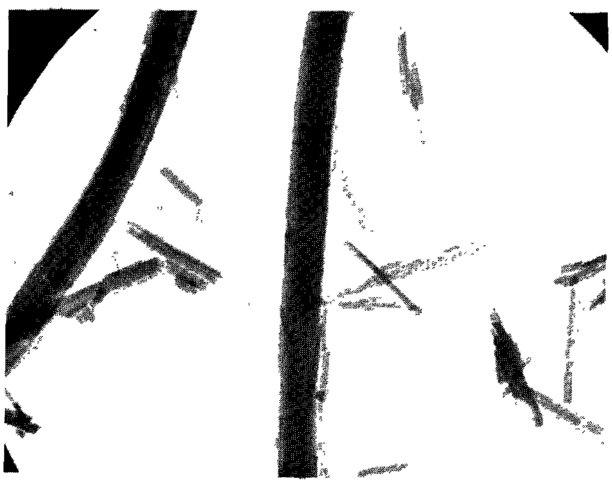
(Figs. 10, 22), to concentric circles for a mass of fibers (Figs. 20, 26). Small bundles or masses of fibers have a pattern of an array of 2 or 3 parallel streaks at acute angles to each other superimposed on an array of incomplete concentric circles (Figs. 14, 28, 30). A very large mass of fibers can produce a hexagonal array superimposed on parallel streaks (Fig. 24).

7

8

9

10



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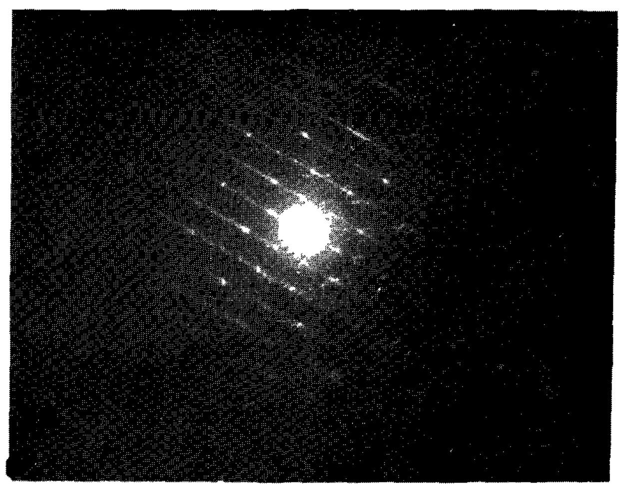
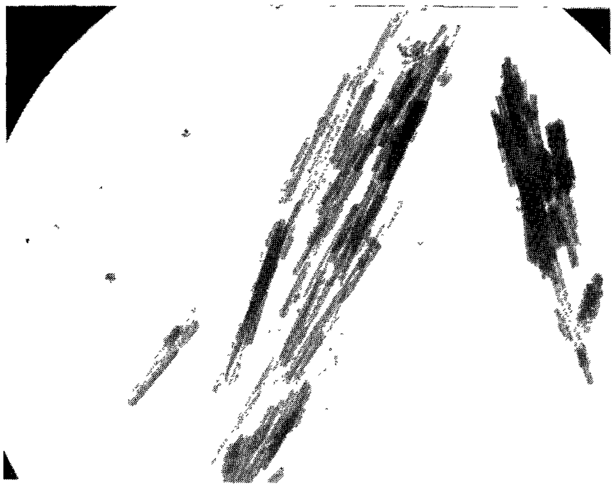


12

CHRYSOTILE, A UICC

1 μm

20,000X



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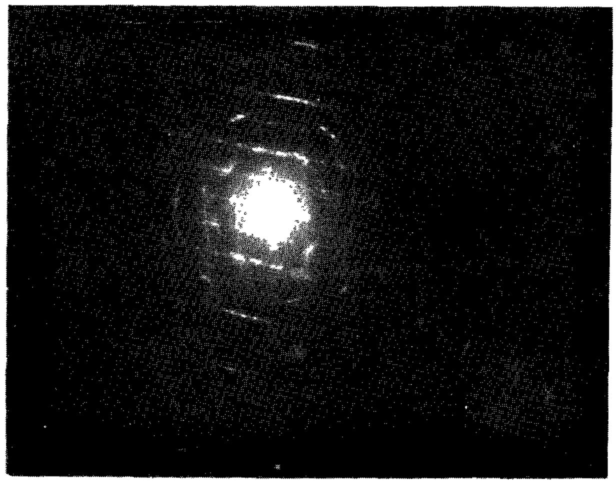

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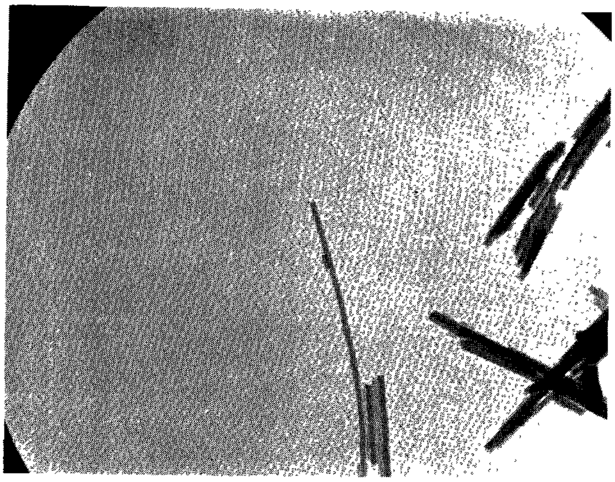

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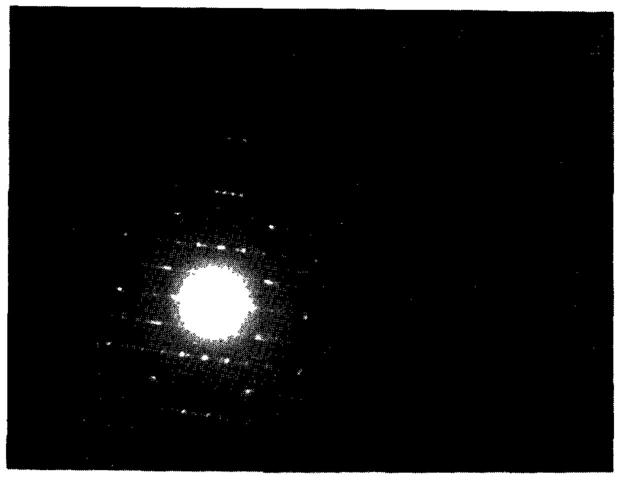
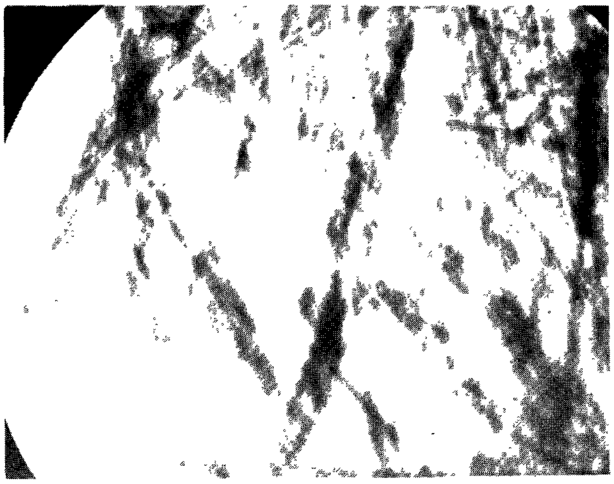


18

CHRYSOTILE, B UICC

1 μm

20,000X



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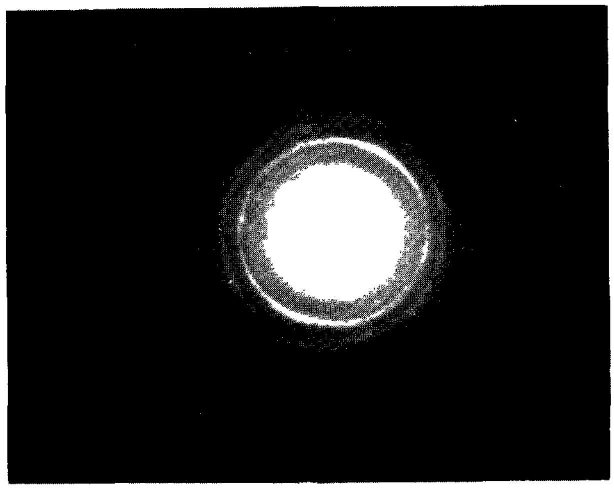

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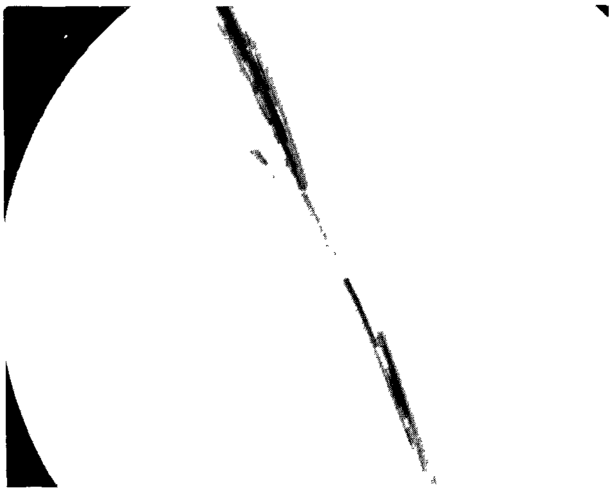

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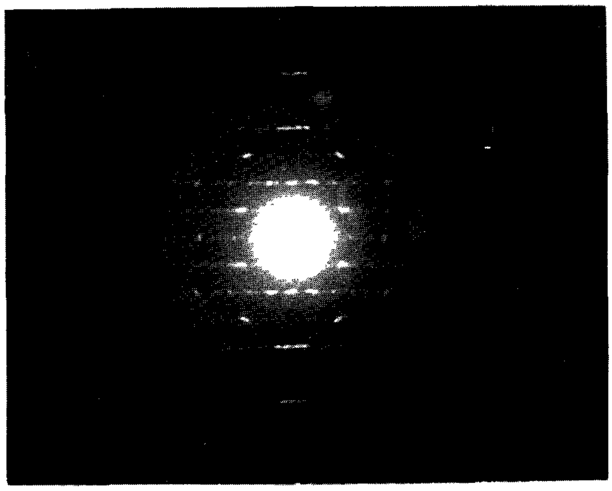

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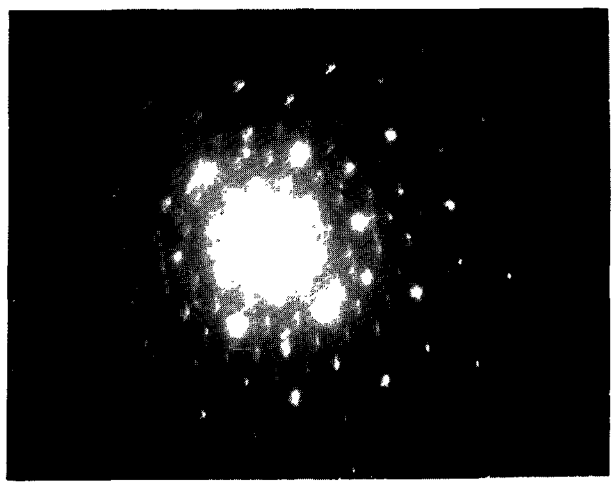
23

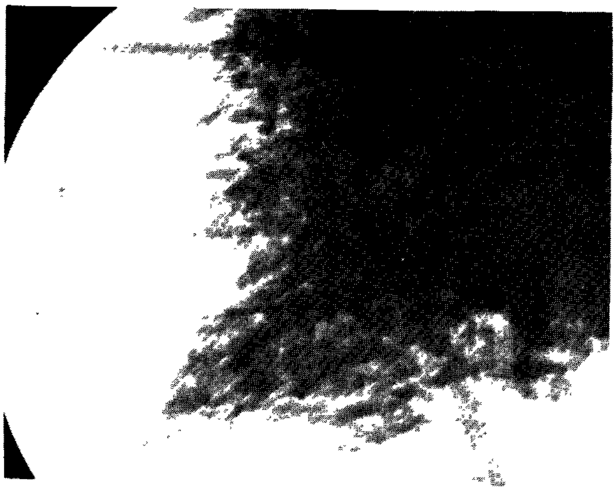


24

CLINOCHRYSOTILE UNION CARBIDE, USGS

1 μm

20,000X



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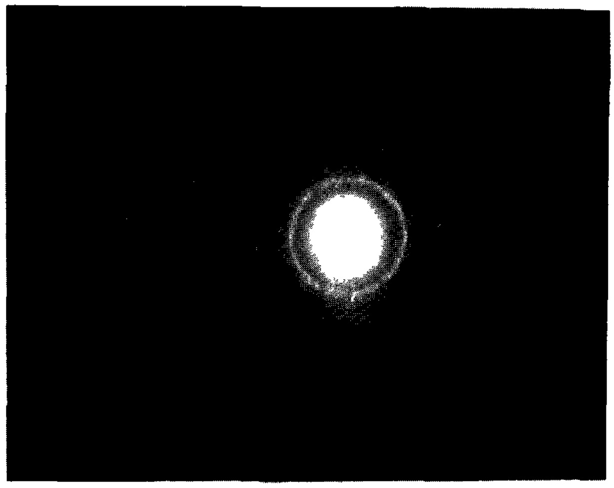

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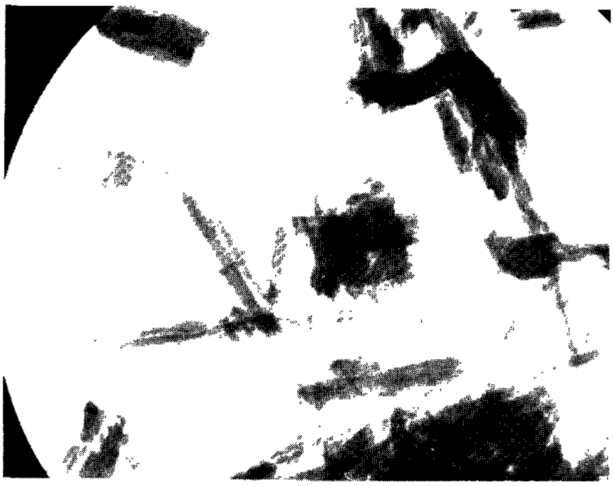

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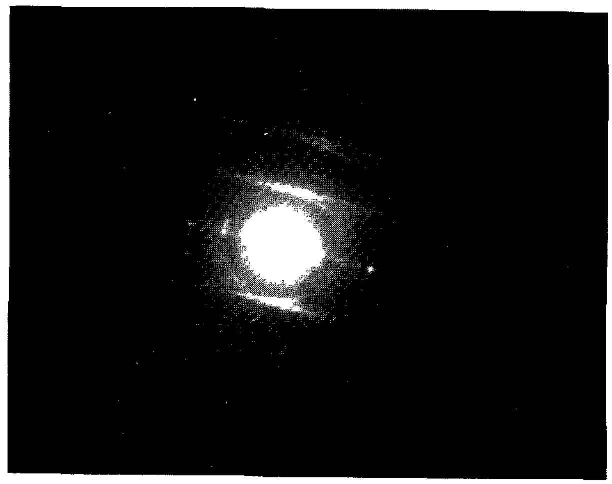
28

29



30

CLINOCHRYSOTILE, 14-NI-63A USGS

1 μm

20,000X

31

32

JEFFREY CHRYSOTILE ASBESTOS

0.5 μm

40,000X

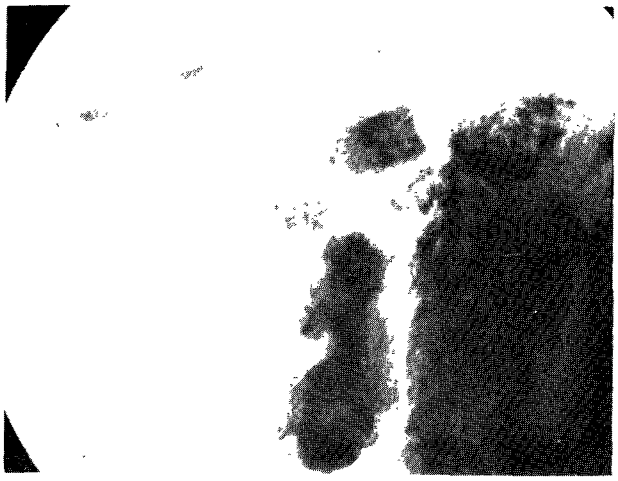
LIZARDITE

Electron Micrographs

Lizardite is a plate-like form of the serpentine class. It occurs in the hard rock associated with some chrysotile asbestos. Lizardite tends to be contaminated with chrysotile. Plates observed in the electron microscope are shown in Figs. 35, 39. Many particles are agglomerates of a jumble of small plates (Fig. 37). Chrysotile fibers can be seen as occlusions in some of the particles and also as single fibers and occasionally as a small mass of chrysotile fibers.

Electron Diffraction Patterns

The diffraction patterns of most of the particles examined are hexagonal arrays, that is, an overall pattern of points arranged in repeating hexagons (Fig. 34). Some patterns have multiple spots at several diffraction points (Figs. 36, 38). Parallel streaks are not observed.



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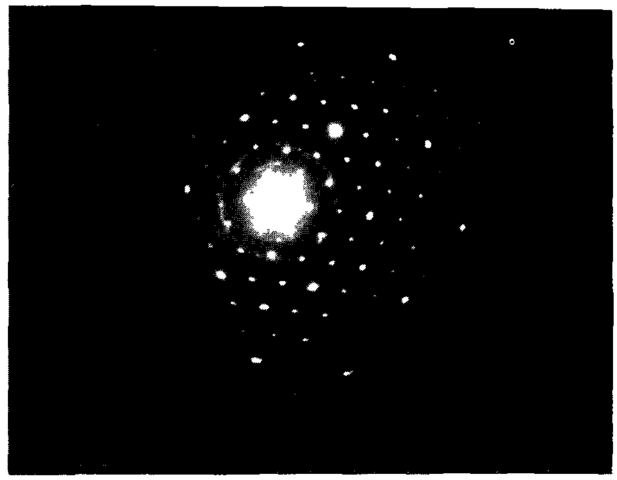

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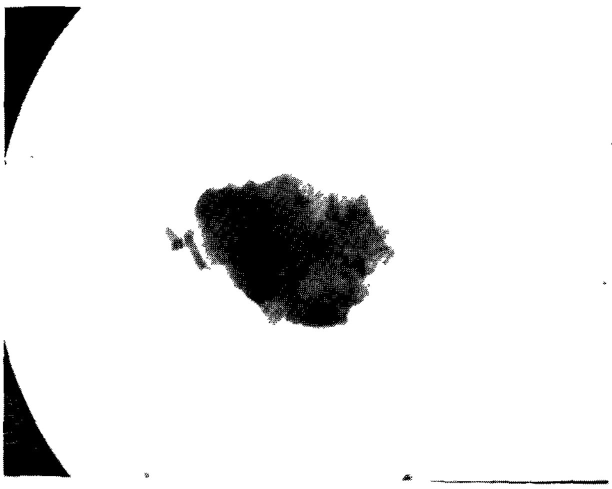

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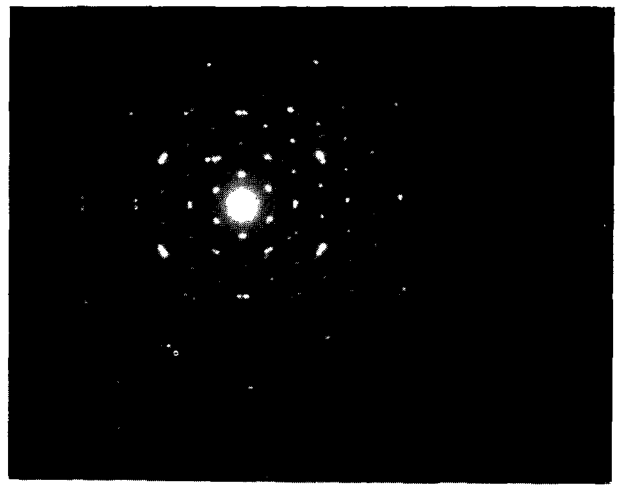

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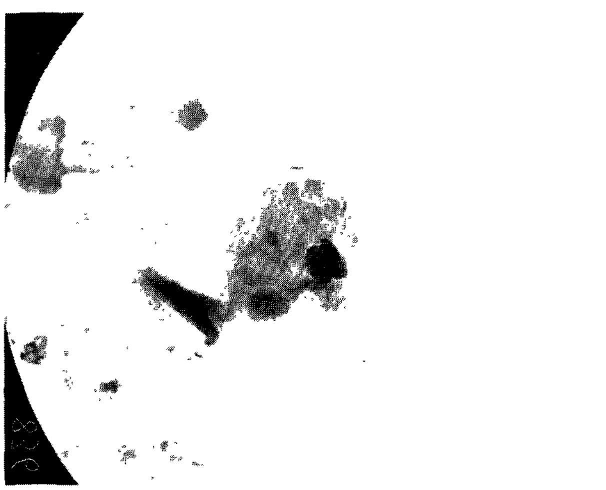

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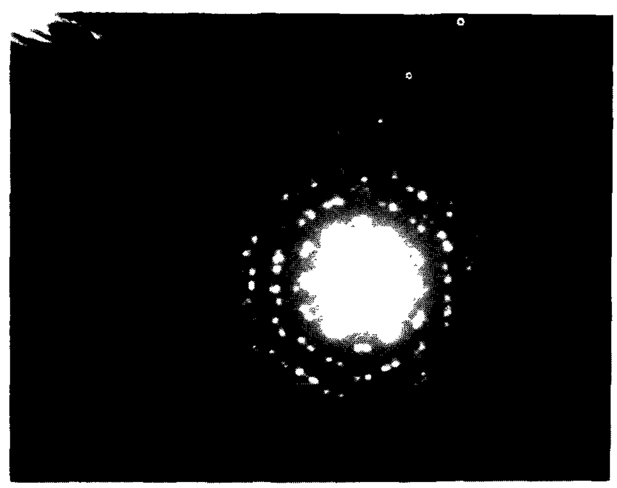


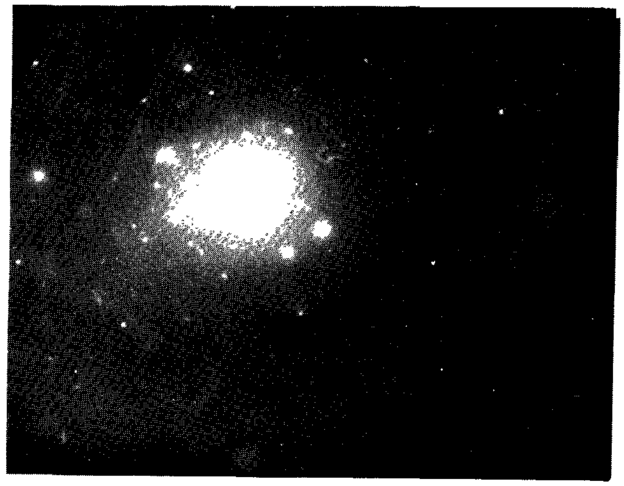
38

LIZARDITE, M4421 UC
LIZARDITE, 16308 UC
LIZARDITE, 16367 UC

1 μm

20,000X

39



40

LIZARDITE, 16308 UC

1 μm

20,000X

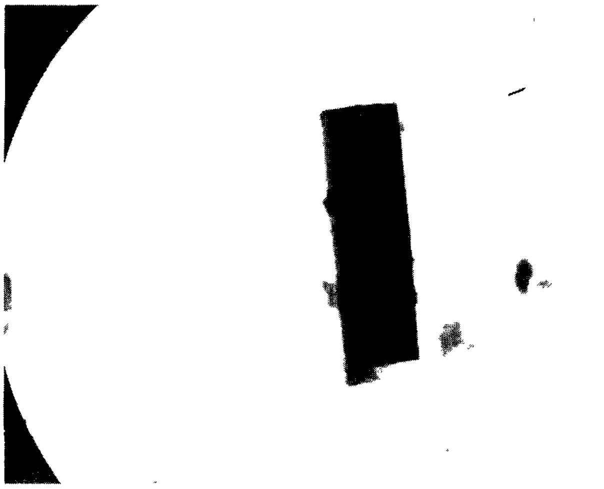
AMOSITE (ASBESTOS)

Electron Micrographs

Some pointed amosite fibers can be confused with the antigorite in that they have striations (Figs. 43, 49); however the striations are less pronounced than those of antigorite. The fibers examined were in a diameter range of 100 to 500 nm. The fibers are not made up of bundles of smaller diameter fibers as are found in chrysotile.

Electron Diffraction Patterns

The diffraction patterns of fibers are parallel lines with diffraction spots much more distinct than those of chrysotile (Figs. 42, 46, 55). An incomplete circle pattern occurs but is not as pronounced as in chrysotile (Fig. 44). Some asymmetrical spots occur in the parallel streaks (Fig. 46). Diffraction of crossed fibers leads to distinctively crossed diffraction lines (Fig. 57).



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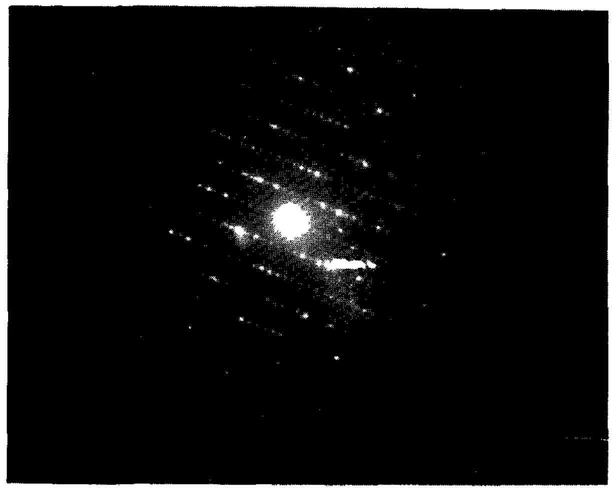

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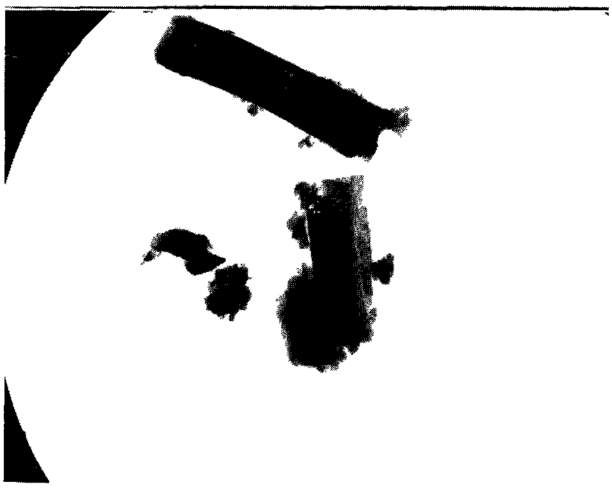

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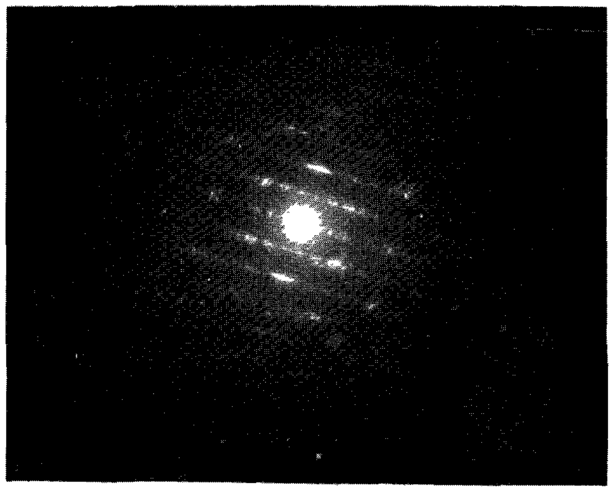

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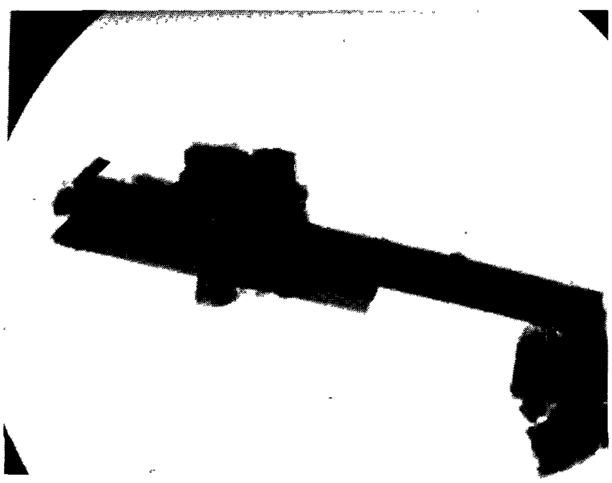

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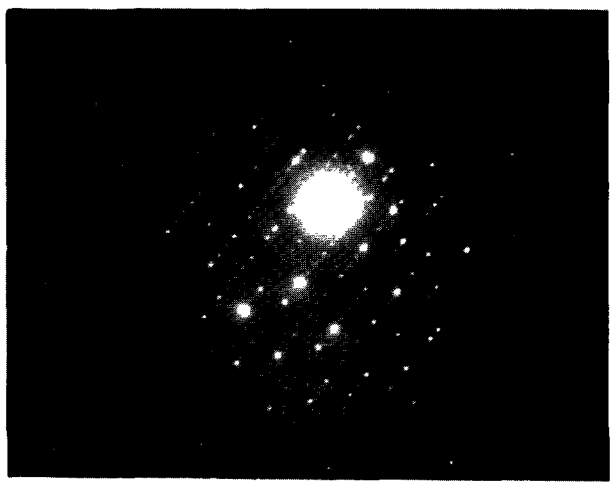
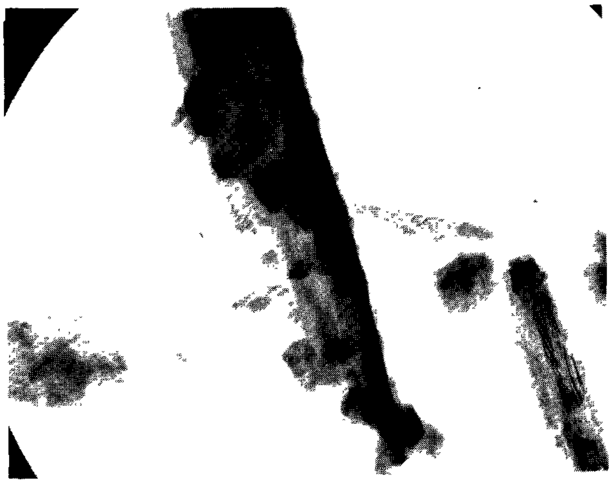


46

AMOSITE, USPHS

1 μm

20,000X



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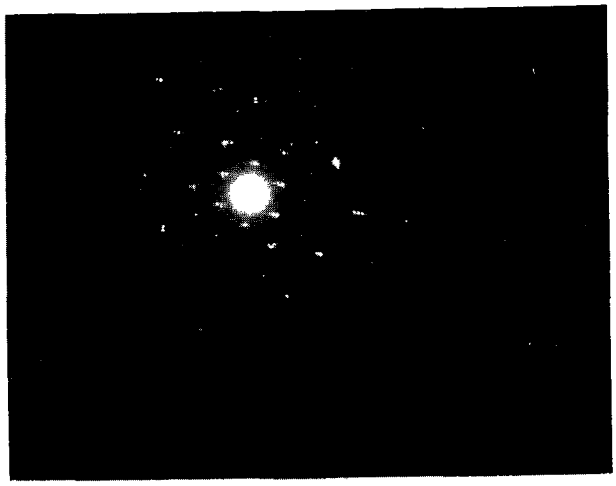

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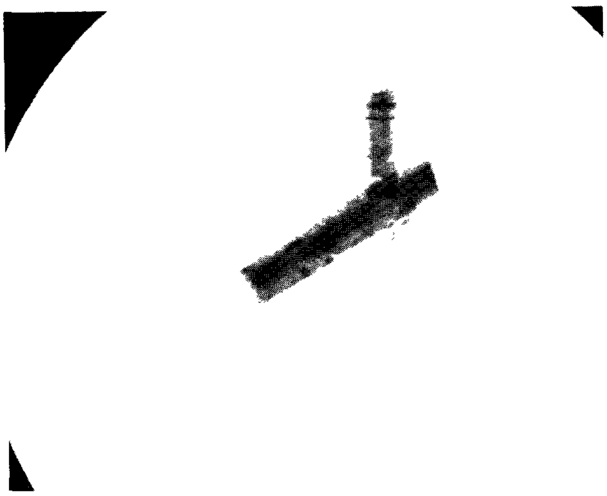
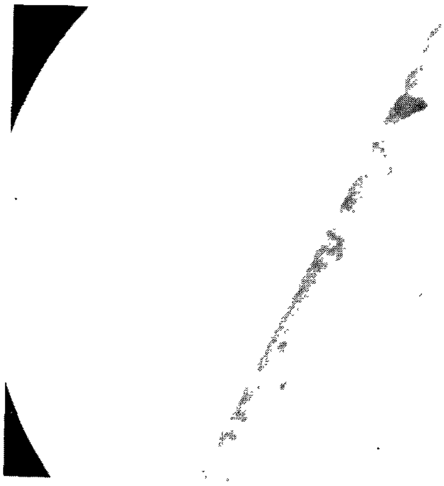


49

AMOSITE, USPHS

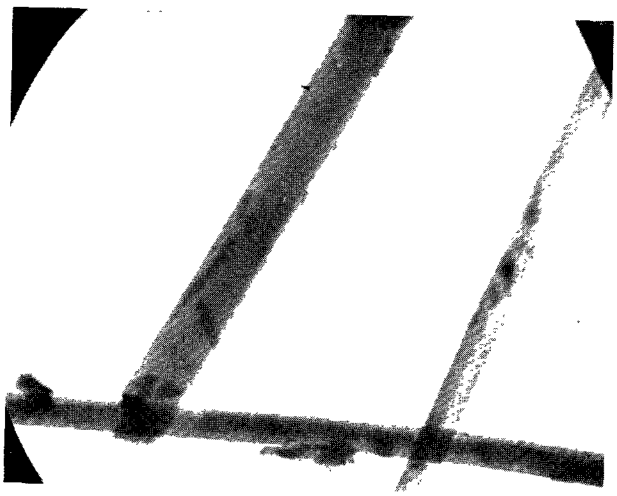
1 μm

20,000X

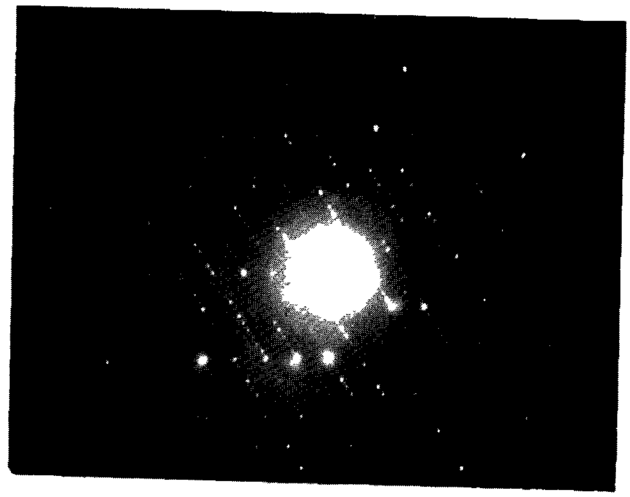


50

51



52



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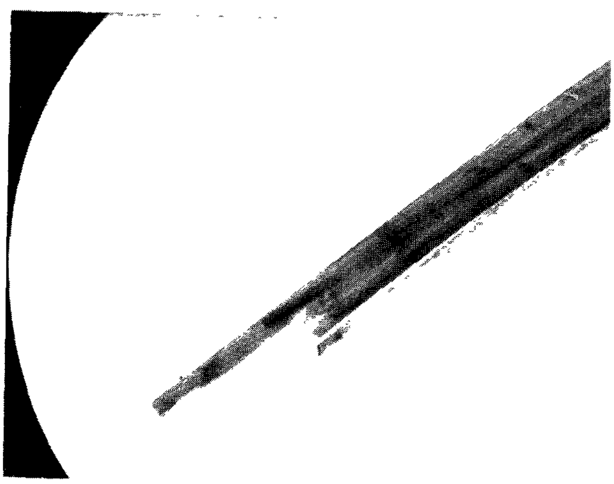

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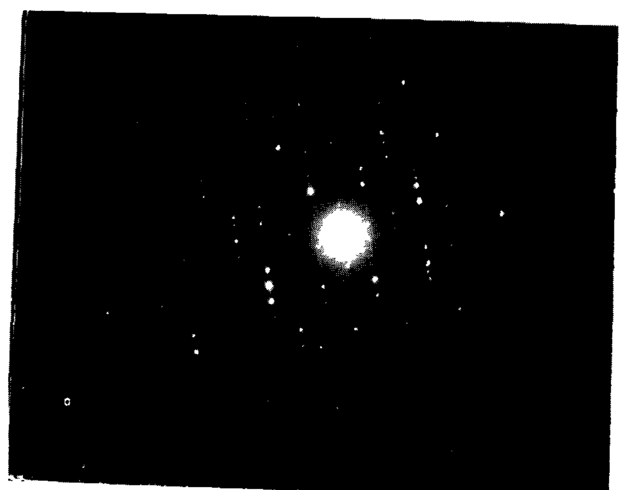
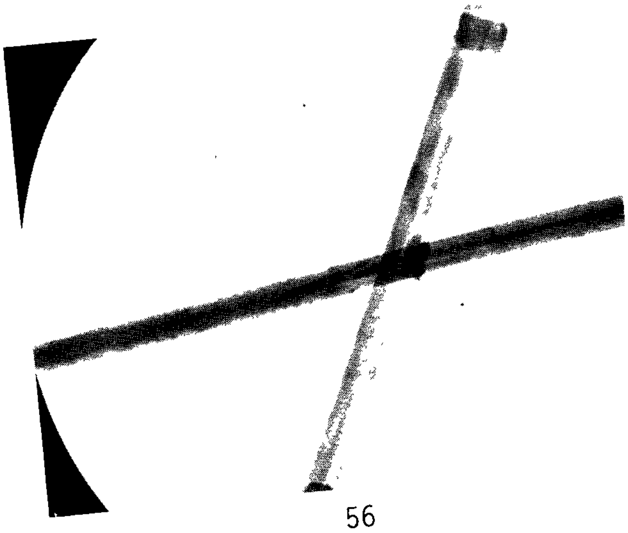


55

AMOSITE, PENGE

1 μm

20,000X



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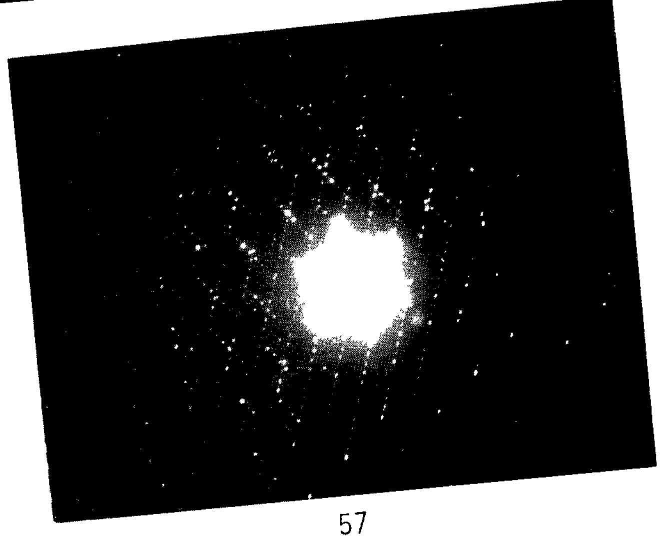


57

ANTHOPHYLLITE (ASBESTOS)

Electron Micrographs

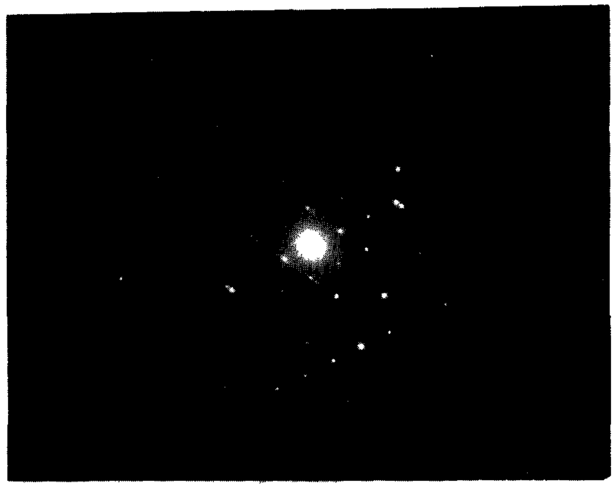
The morphology of anthophyllite fibers is similar to that of amosite and crocidolite (Fig. 60) and is in the same diameter range.

Electron Diffraction Patterns

The electron diffraction patterns are similar to those for amosite, crocidolite and chrysotile. Figure 63 is the pattern of a fiber with streaks like amosite and chrysotile. Patterns of fibers are streaked something like those of amosite (Fig. 59) and chrysotile (Fig. 61). No partial circular patterns are observed in the same patterns that are streaked.

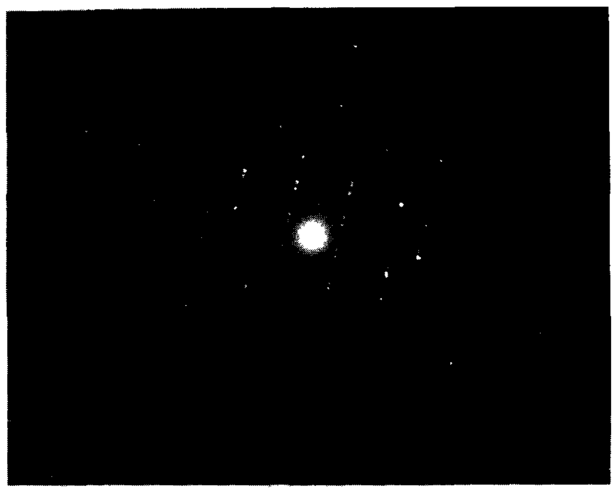
Anthophyllite is difficult to distinguish from the other amphibole asbestos fibers by electron diffraction and is readily distinguishable only from chrysotile by morphology.

58



59

60



61

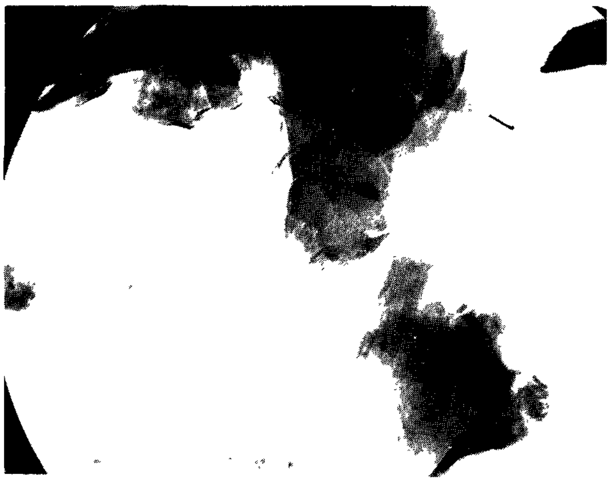
62

63

ANTHOPHYLLITE, UICC

1 μm

20,000X



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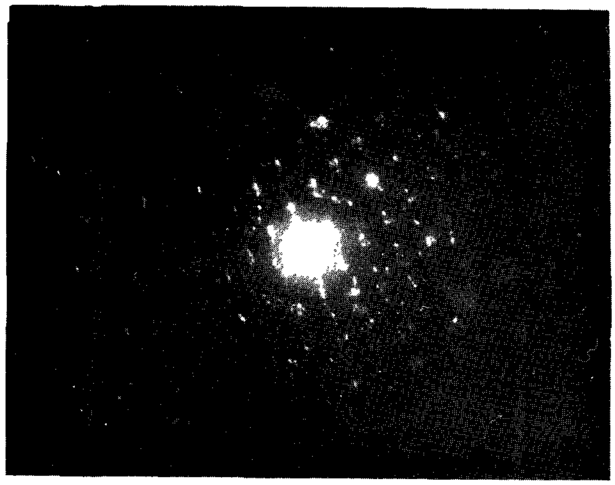

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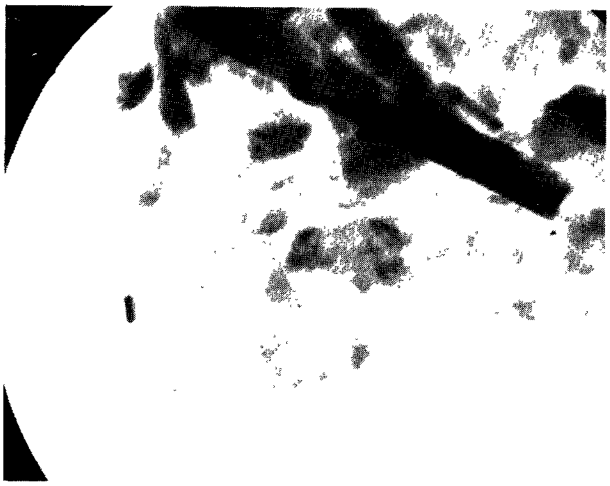

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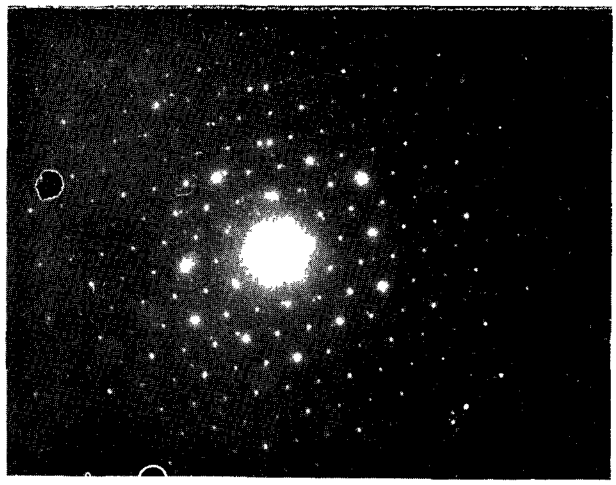


67

ANTHOPHYLLITE, UICC

1 μm

20,000X

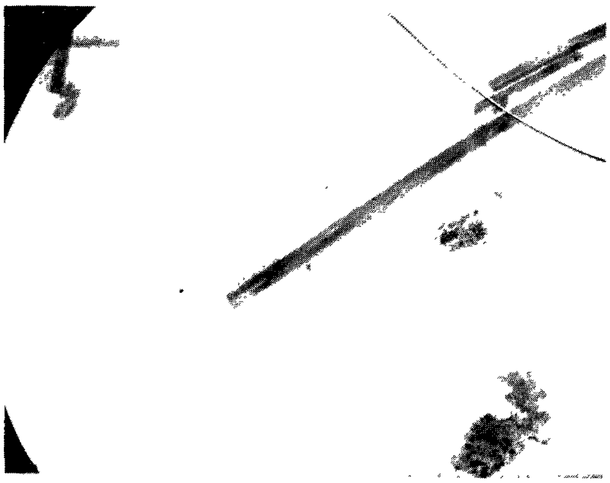
CROCIDOLITE (ASBESTOS)

Electron Micrographs

The morphology of crocidolite has some similarity to amosite (Figs. 68, 70, 72). The fibers are not made up of smaller diameter fibrils and do not have a regular internal structure like chrysotile. The diameter of the fibers is somewhat smaller than that of amosite. The fibers fall into a range of 33 to 205 nm diameter (Figs. 74, 75, 77). This range overlaps both chrysotile and amosite.

Electron Diffraction Patterns

The electron diffraction patterns of the fibers are somewhat like chrysotile but not as regular. Circular patterns are not seen (Figs. 69, 71, 73).



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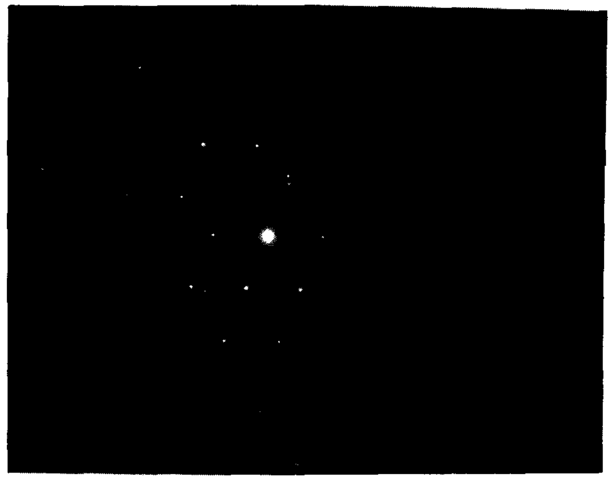

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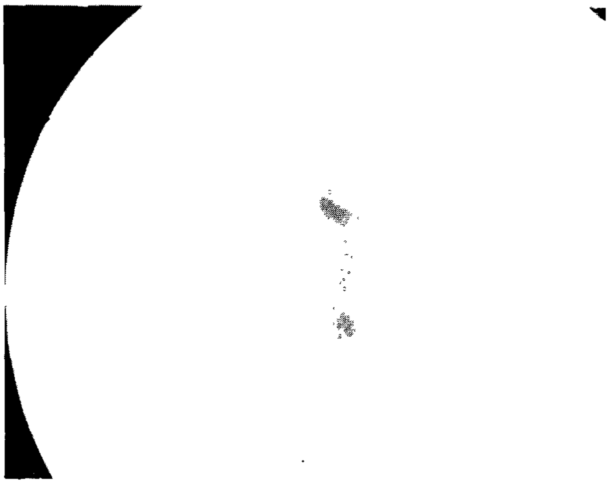

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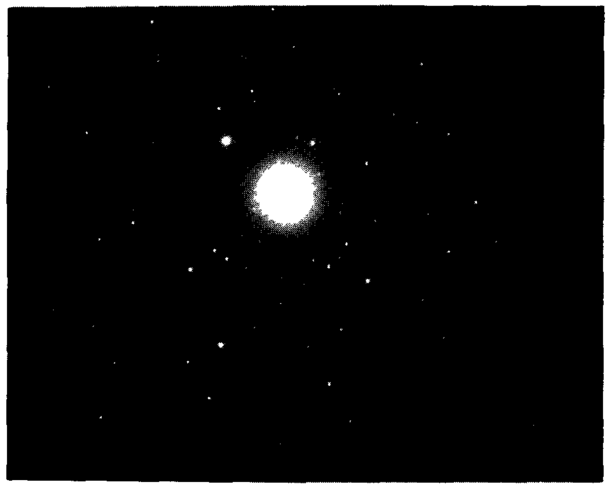


71

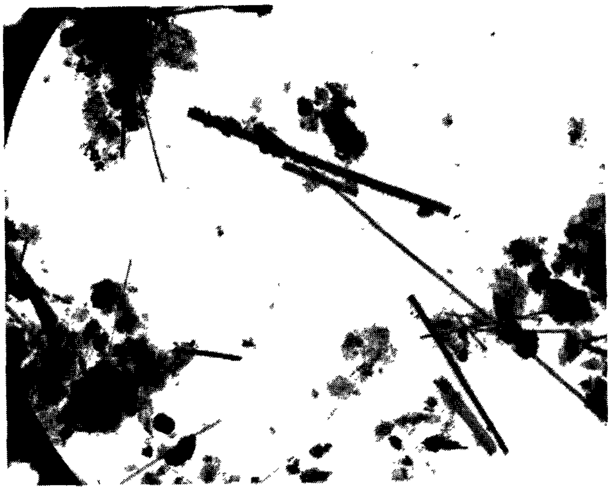
72

73

CROCIDOLITE, UICC

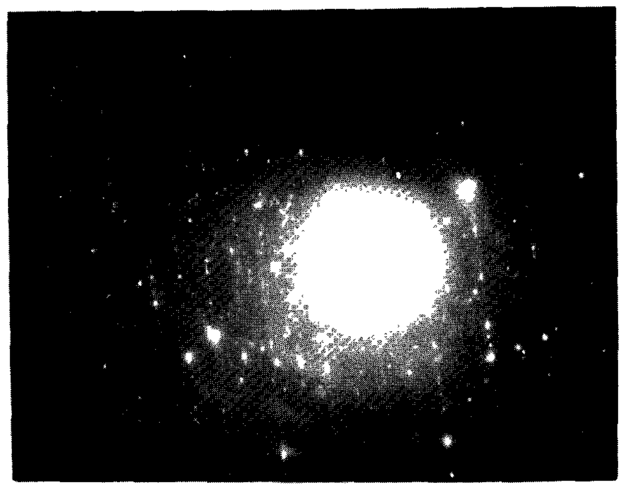
1 μm

20,000X

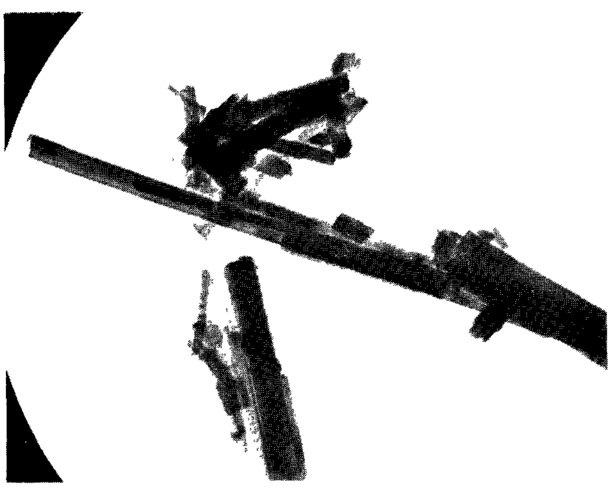


74

75



76



77

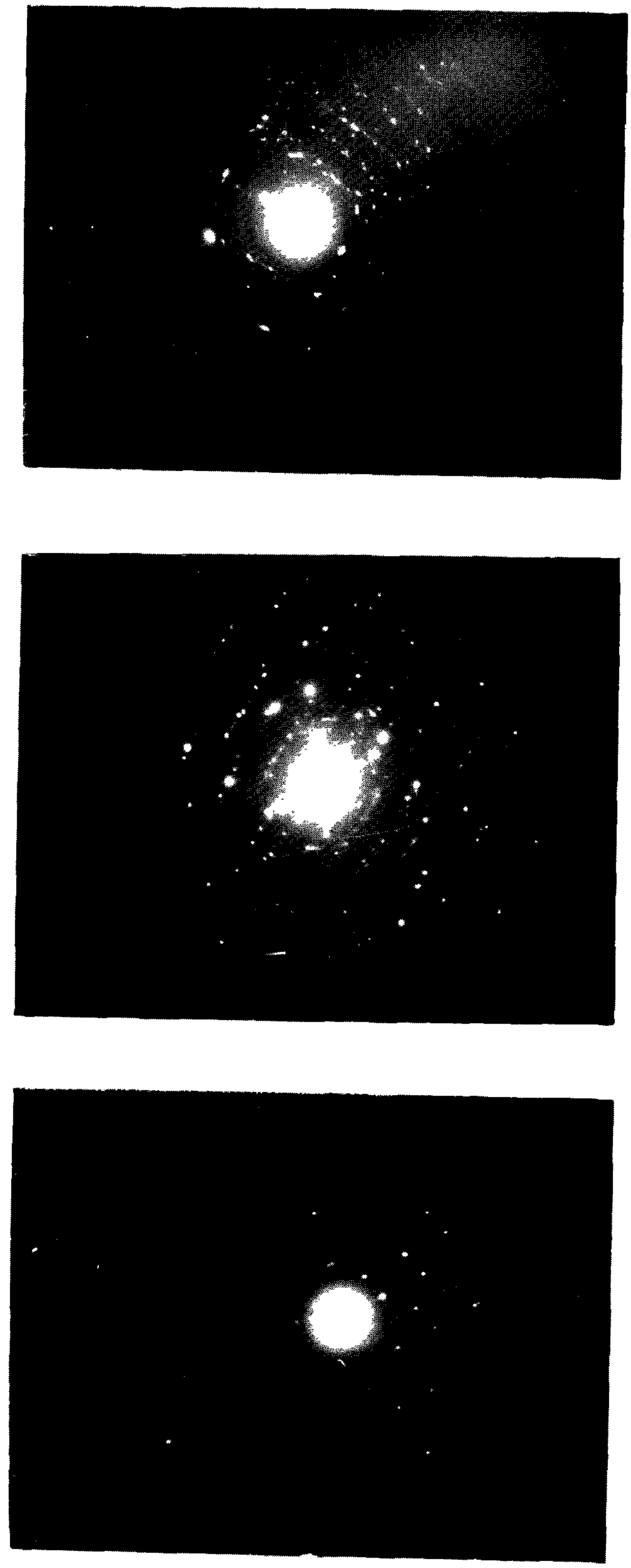
78

CROCIDOLITE, UICC

1 μm

20,000X

83



84

CROCIDOLITE, UICC
CROCIDOLITE, USPHS
CROCIDOLITE, S. AFRICAN, USPHS

1 μm

20,000X

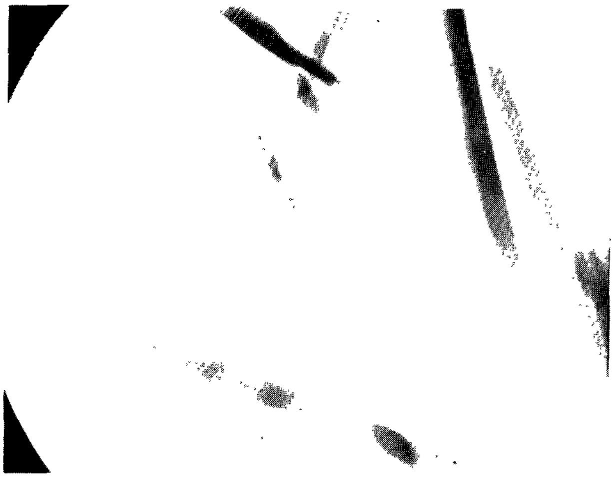
TREMOLITE (ASBESTOS)

Electron Micrographs

Tremolite fibers tend to have longitudinal striations (Fig. 97). The diameter of the fibers ranges from 100 (Fig. 69) to 1150 nm (Fig. 87). Most of the fibers are in the 200 to 400 nm diameter range. There are some bundles of fibers (Figs. 91, 95). It is difficult to distinguish tremolite fibers from other amphibole asbestos fibers.

Electron Diffraction Patterns

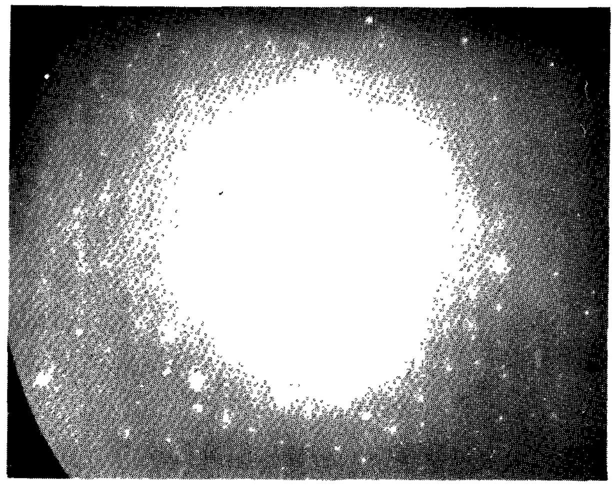
The pattern of a bundle is an array of parallel, rather thick lines made of dots (Fig. 92). Single fiber patterns have been obtained that have parallel arrays of spots (Figs. 88, 90). Multiple parallel arrays at various angles to each other (Fig. 96) and apparent random distributions of diffraction spots occur in fields where several fibers are irregularly oriented (Figs. 94, 98).



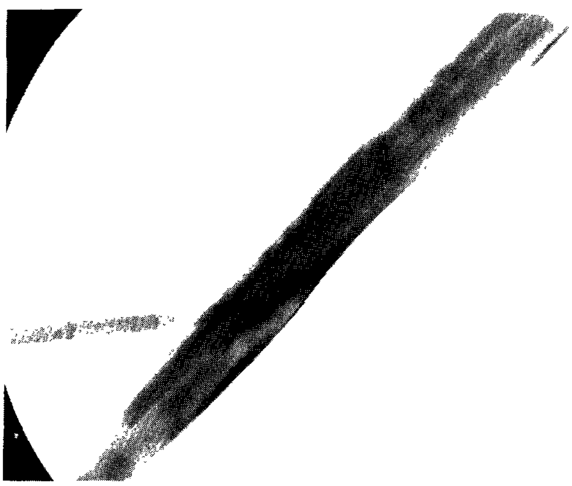
85

86

87



88



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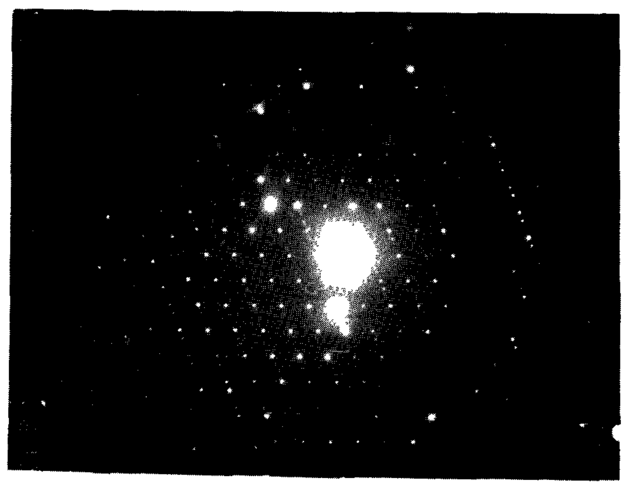
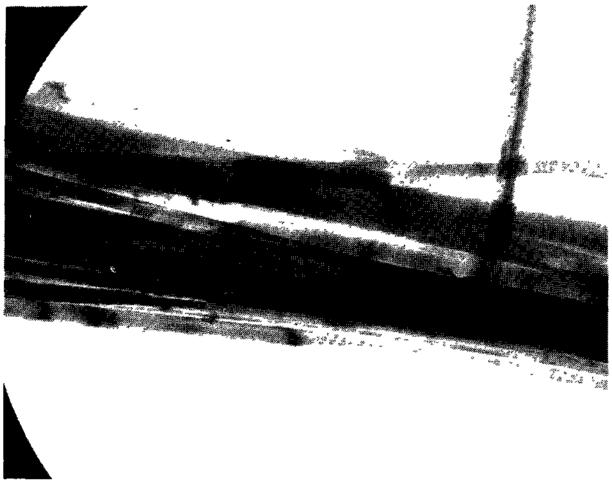


90

TREMOLITE

1 μm

20,000X



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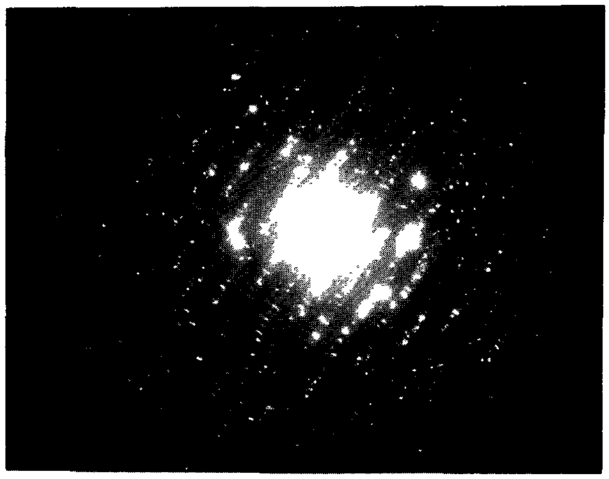

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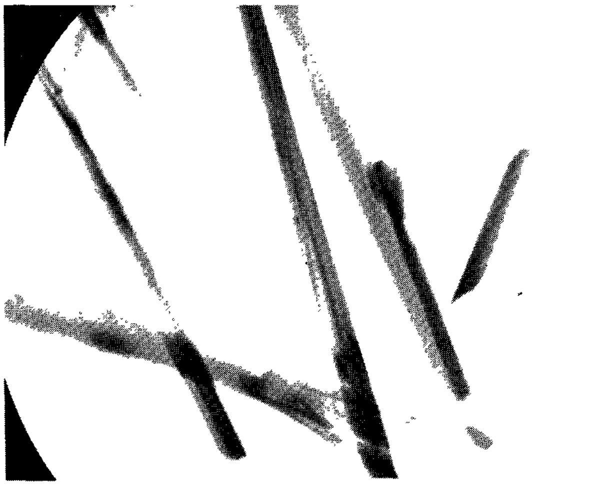


93

94

95

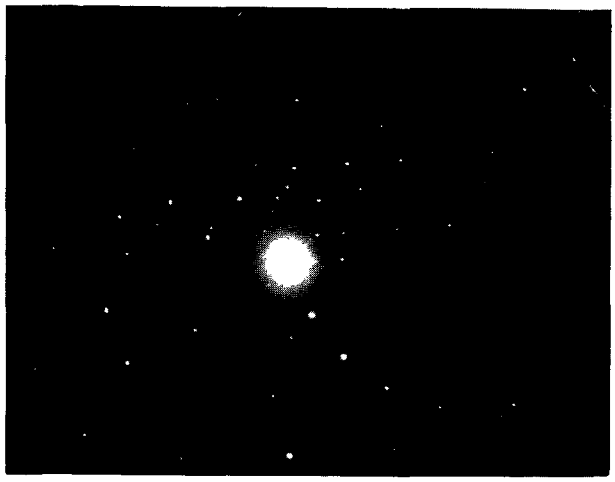
96

TREMOLITE

1 µm

20,000X

97



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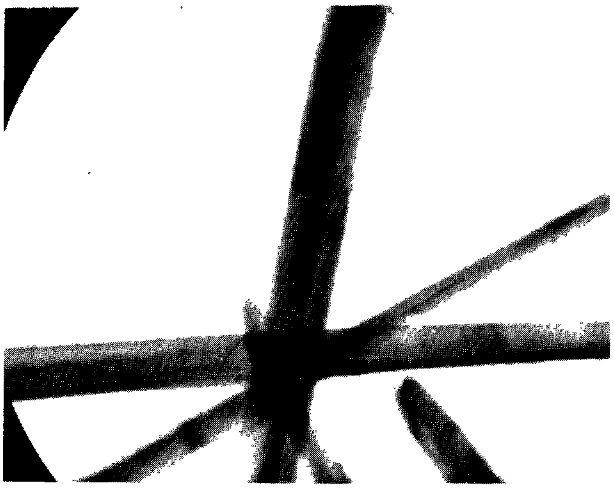
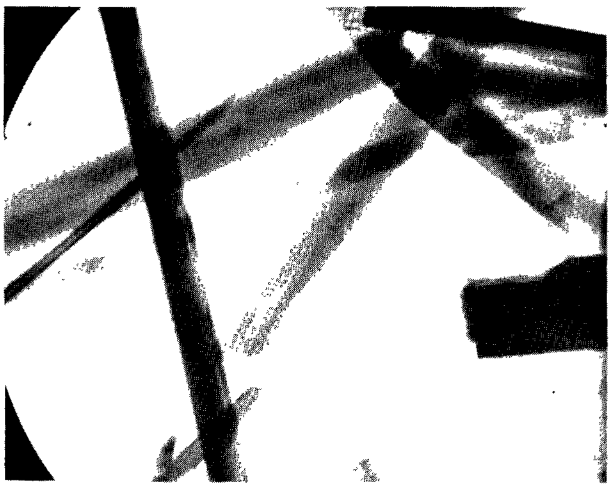
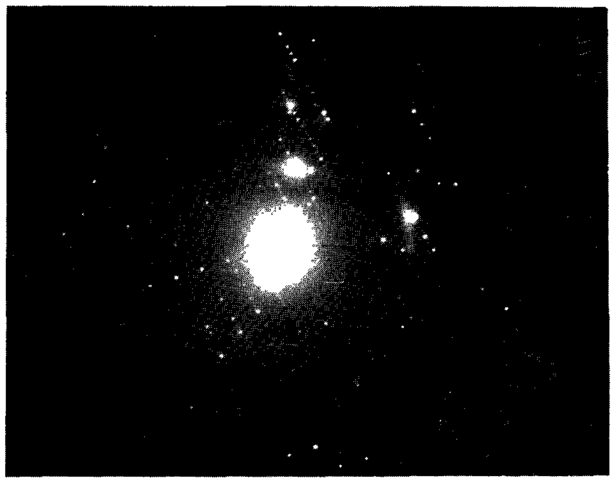


99

100



101



102

TREMOLITE

1 μm

20,000X

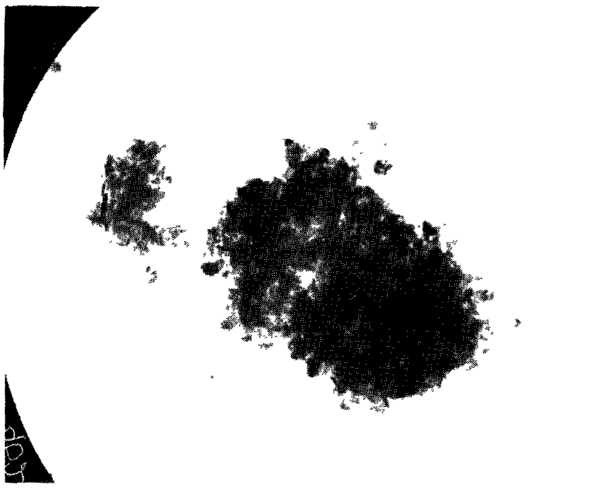
BRUCITE

Electron Micrographs

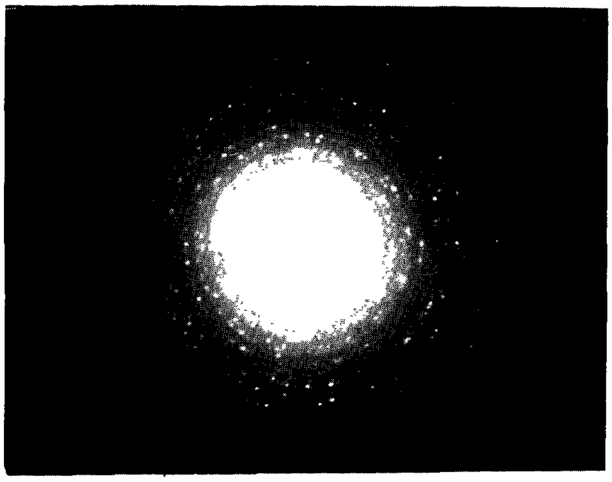
The particles seen in the electron micrographs are made up of aggregates of small rods and plates somewhat circular in form (Figs. 103, 105).

Electron Diffraction Patterns

The electron diffraction patterns are circular in pattern. No streaks or hexagonal arrays are observed (Fig. 104, 106, 108).

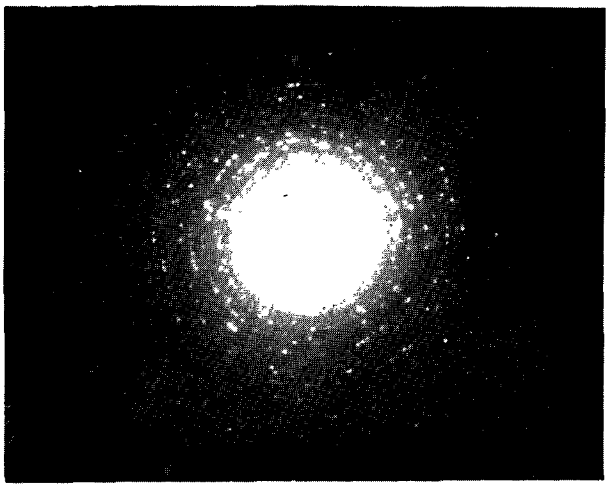


103



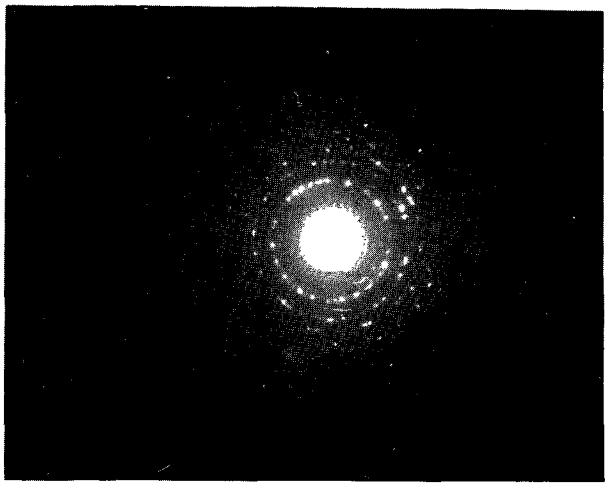
104

105



106

107

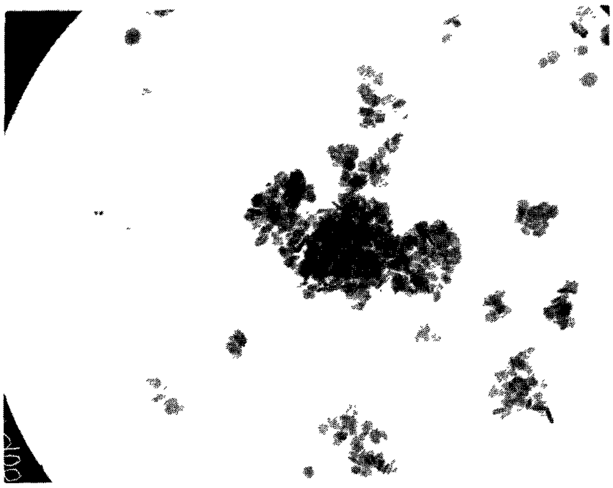


108

BRUCITE, $Mg(OH)_2$

1 μm

20,000X



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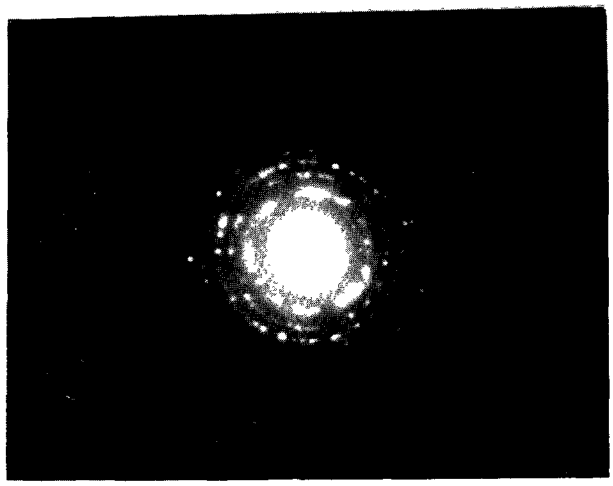


110

BRUCITE, $Mg(OH)_2$

1 µm

20,000X

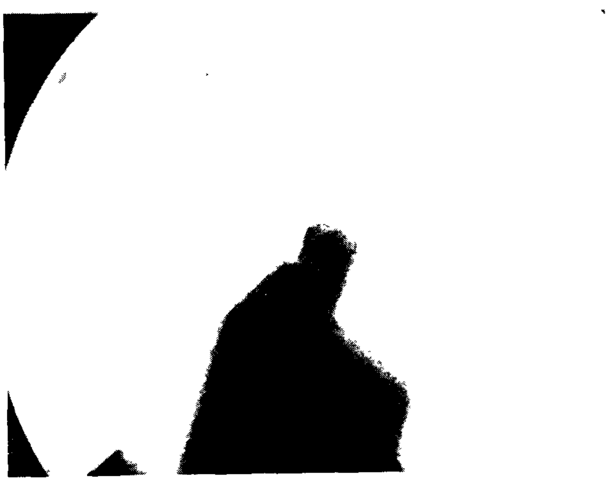
CALCITE

Electron Micrographs

The particles are made up of aggregates of very small particles of irregular shape (Fig. 113).

Electron Diffraction Patterns

The electron diffraction patterns range from a circular pattern to a pattern of widely spaced streaks. One pattern exhibits an ill-defined group of comet-like Kikuchi lines (Fig. 114).



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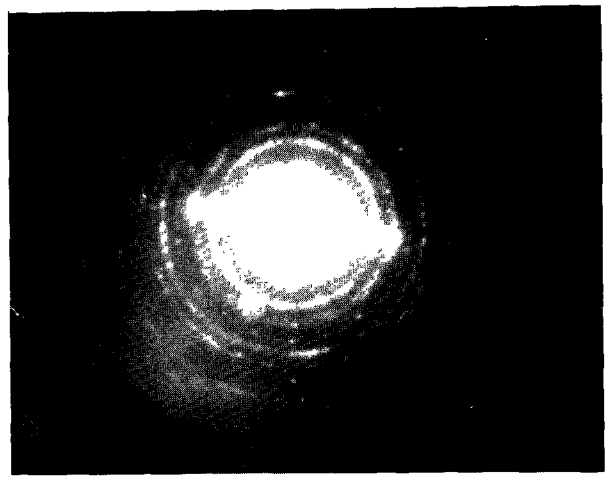
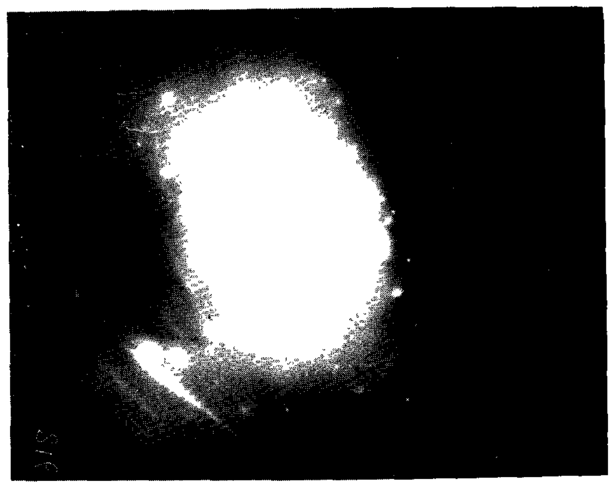


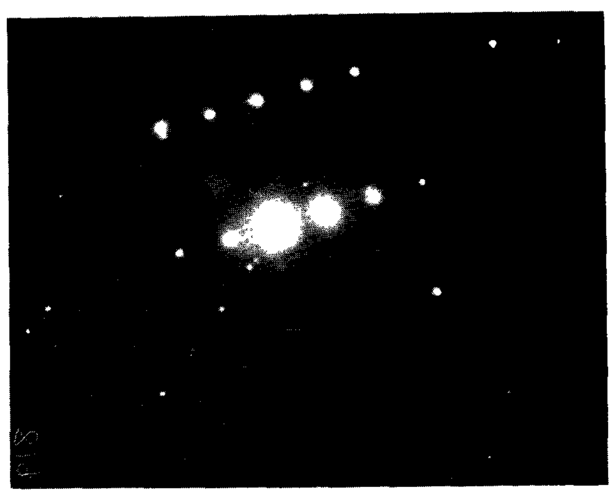
112

113



114

115



116

CALCITE, $CaCO_3$

1 μm

20,000X

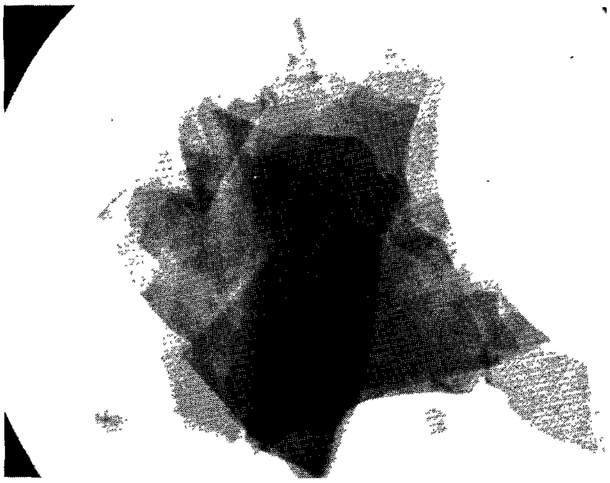
MAGNESITE

Electron Micrographs

The morphology of magnesium carbonate is primarily plate-like with the occurrence of a few irregular fibers. The plates tend to be stacked together in an irregular pattern.

Electron Diffraction Patterns

The electron diffraction pattern for a thick mass (Fig. 120) consists of concentric circles with a background of faint diffraction spots. A thinner stack of plates gives a well-defined streak pattern somewhat like amosite (Fig. 122) but with visibly narrower spacings. An irregular fiber gives a streak pattern with relatively widely spaced streaks. The plates of $MgCO_3$ give a quite different pattern from antigorite or lizardite.



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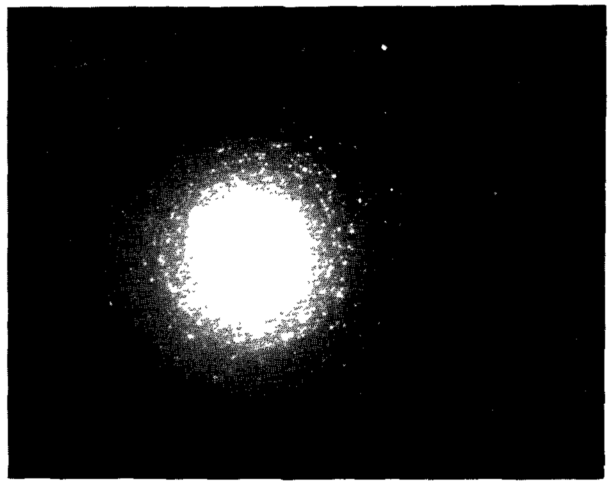

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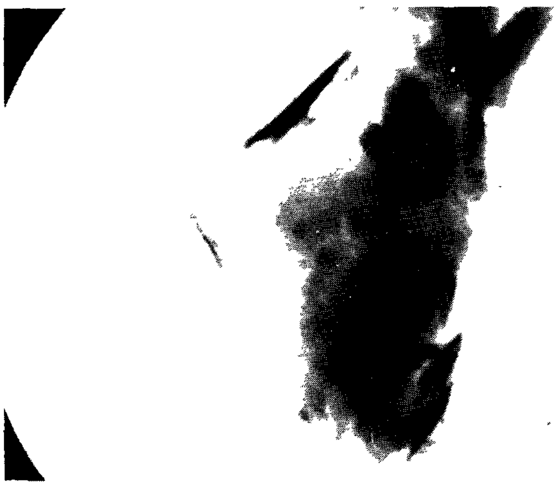

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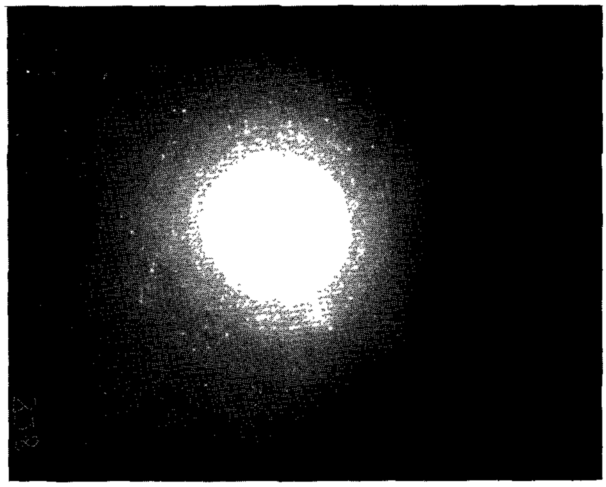

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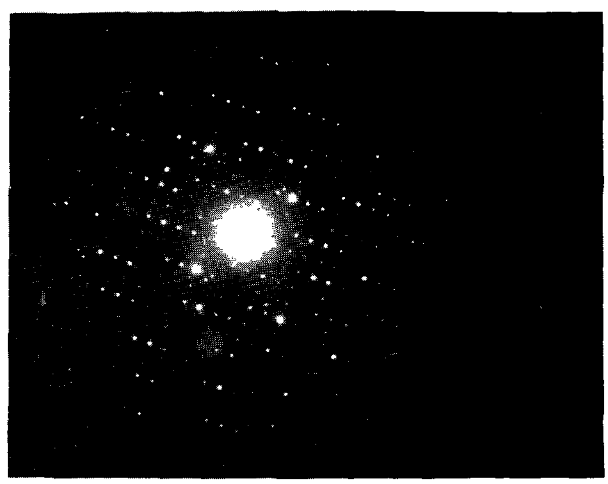
121

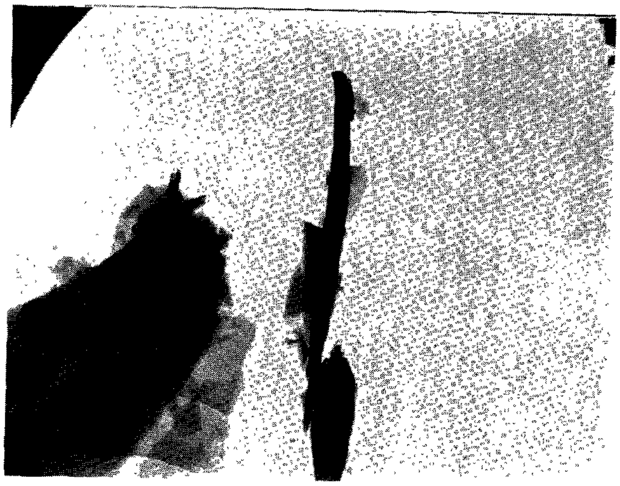


122

MAGNESITE, $MgCO_3$

1 μm

20,000X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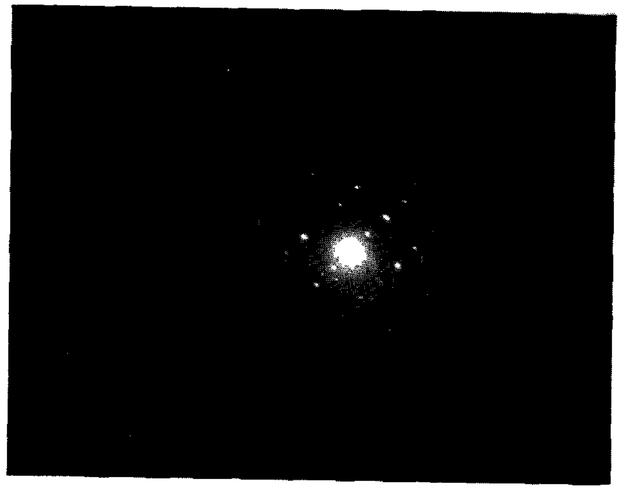

124

MAGNESITE, $MgCO_3$

1 μm

20,000X

QUARTZ

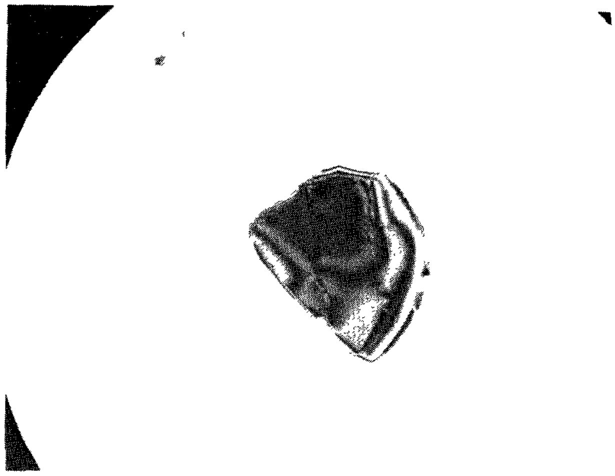
Electron Micrographs

Plates. Occurs as sharply angular chips. (Figs. 125, 127, 129).

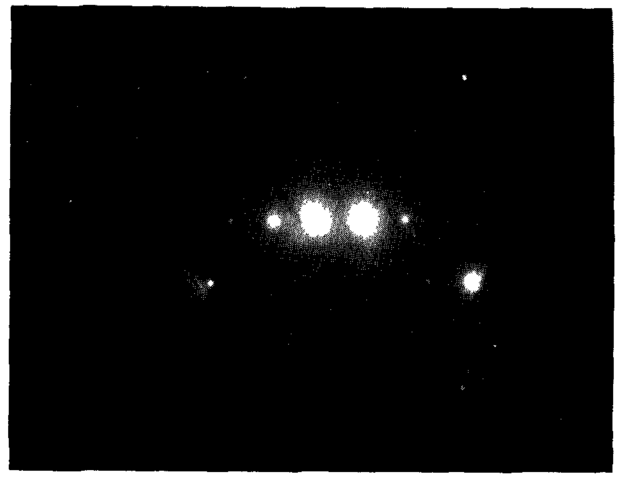
Electron Diffraction Patterns

Some patterns are circular arrays of dots, tending towards a hexagonal pattern. (Fig. 128).

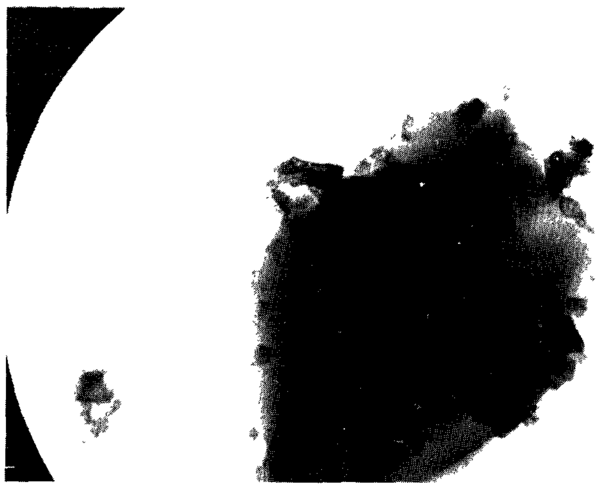
Overall hexagonal patterns, somewhat irregular, are also found (Fig. 126). A few comet-like Kikuchi line patterns occur, including black and white lines (Fig. 130).



125

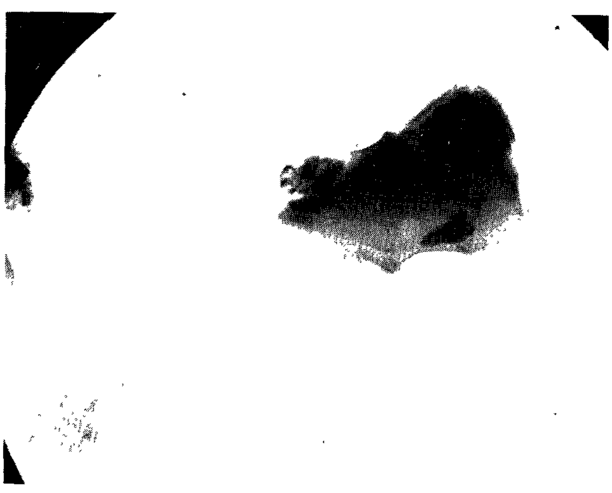


126



127

128



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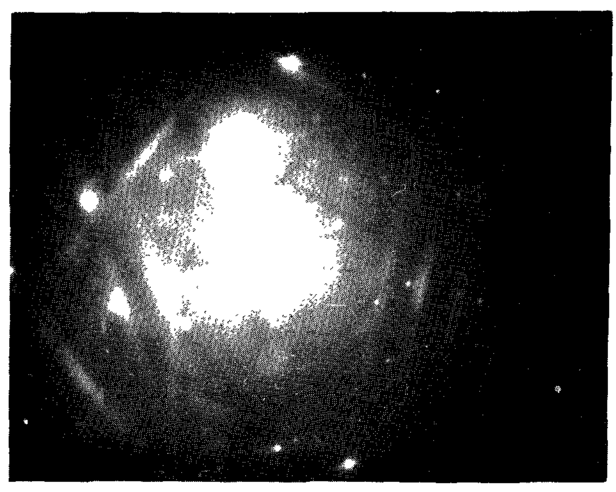


130

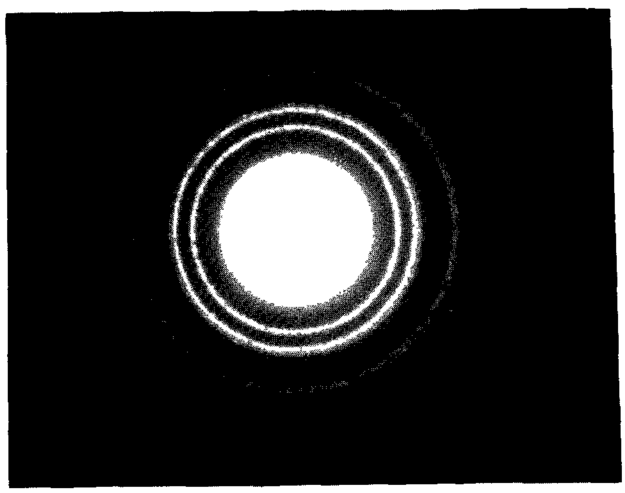
α-QUARTZ

1 μm

20,000X

GOLD DIFFRACTION PATTERN

High purity gold wire was used to form a thin deposit on 300-mesh stainless steel collodion-coated grids at vertical incidence in a vacuum evaporator. The diffraction (Fig. 131) was performed with the identical microscope parameter settings that were used to obtain the diffraction patterns for the other specimens in the atlas.



131

GOLD DIFFRACTION PATTERN

1 μm

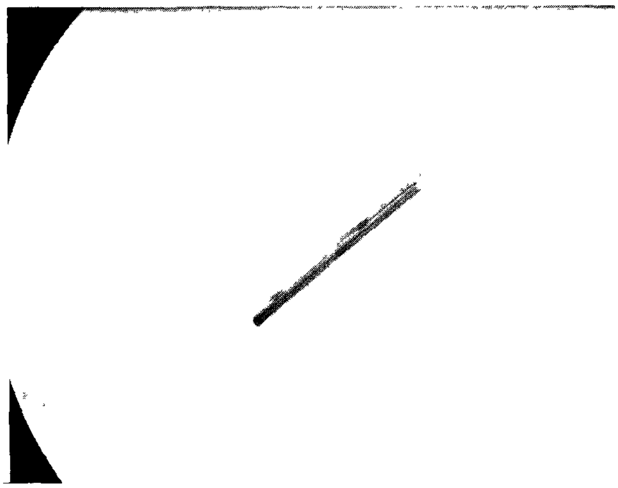
20,000X

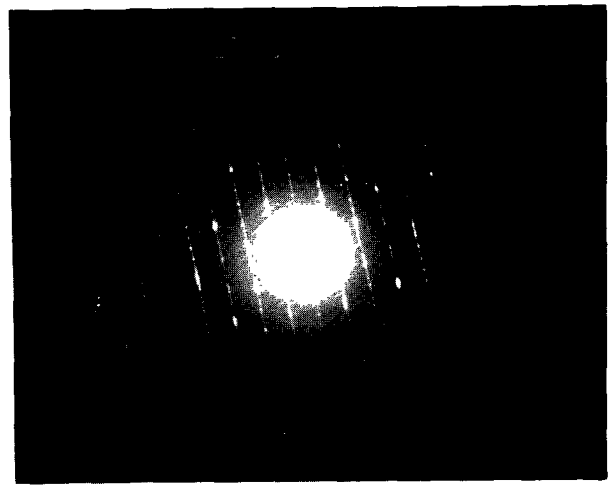
ASBESTOS ISOLATED FROM AIR SAMPLES

Figures 132, 134 and 136 are micrographs of asbestos isolated from air samples. Figures 133, 135, and 137 are the corresponding diffraction patterns. Figure 132 shows chrysotile fibers collected from about 50 m^3 of air drawn through a nitrocellulose membrane (Millipore) filter in Berkeley, California, downwind from an office building under construction in April 1970. Figure 134 shows chrysotile isolated from 1500 m^3 of air filtered through a polystyrene (Microsorban) filter in San Diego, California, near ship insulation work in July 1969. Figure 136 shows chrysotile found in about 2000 m^3 of air filtered through a polystyrene filter in the rural community of San Martin, California, in September 1969.

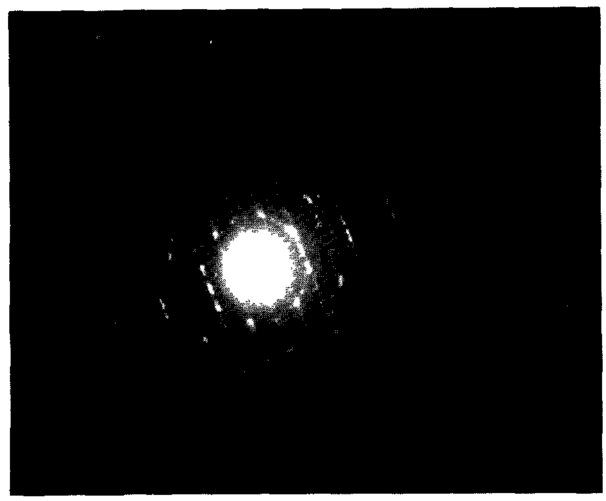
A typical bundle of chrysotile fibrils was isolated from a sample taken in San Francisco (Figs. 138, 139) in 1969.

Figures 140 and 141 show amosite and its diffraction pattern isolated from a sample of ambient air from Newark, New Jersey, filtered through a Nylon microweb filter (Millipore) in 1969.

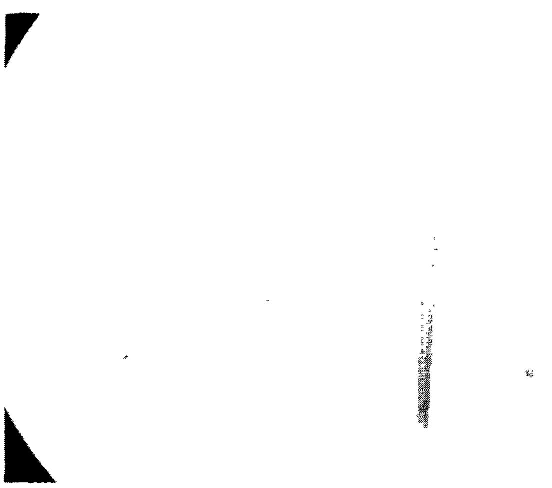


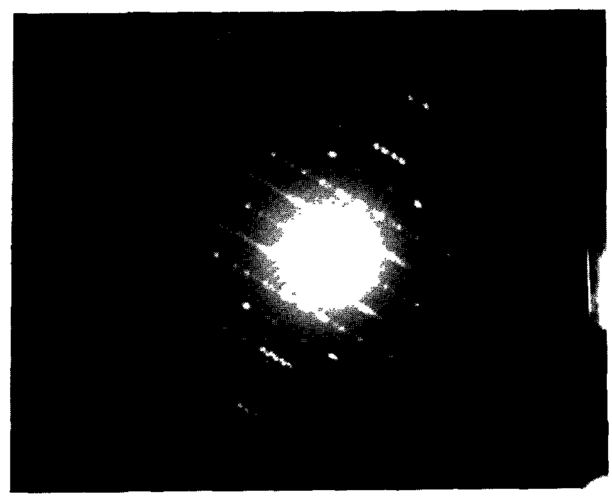


132 Berkeley 133



134 San Diego 135

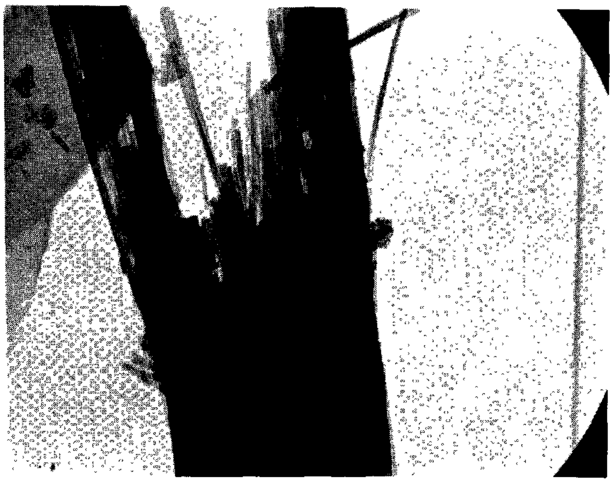




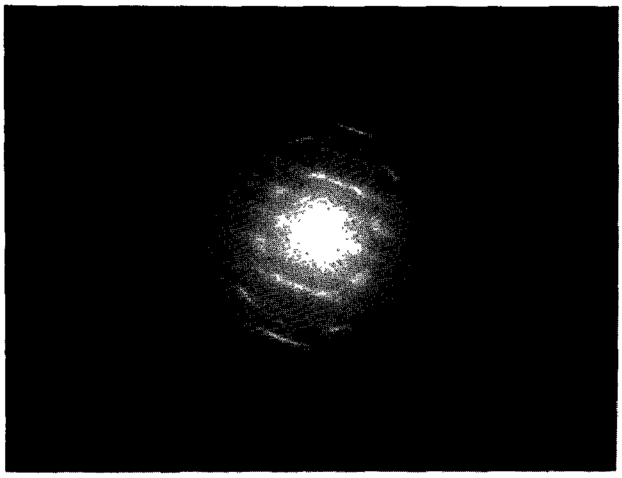
136 San Martin 137

CHRYSOTILE FOUND IN CALIFORNIA AIR SAMPLES
1969-1970

1 μm 20,000X



138



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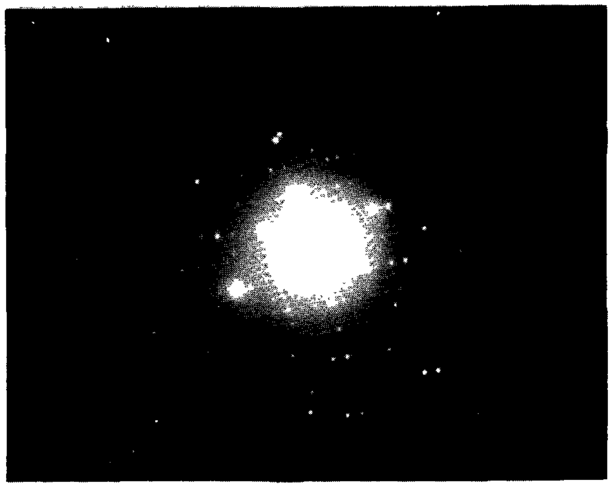
CHRYSOTILE FROM AIR SAMPLE
TAKEN IN SAN FRANCISCO, CALIF., 1969

1 μm

20,000X

140



141

AMOSITE FROM AIR SAMPLE
TAKEN IN NEWARK, N.J., 1969

1 μm

20,000X

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TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO. EPA-650/2-75-036	2.	3. RECIPIENT'S ACCESSION NO.
4. TITLE AND SUBTITLE Asbestos Fiber Atlas		5. REPORT DATE April 1975
		6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S) Peter K. Mueller, Arthur E. Alcocer Ronald L. Stanley, and Glenn R. Smith		8. PERFORMING ORGANIZATION REPORT NO. AIHL Report No. 98
9. PERFORMING ORGANIZATION NAME AND ADDRESS Air and Industrial Hygiene Laboratory Laboratory Services Program State of California Department of Health 2151 Berkeley Way, Berkeley, CA 94704		10. PROGRAM ELEMENT NO. 1AA010 (26AAN)
		11. CONTRACT/GRANT NO. 801336
12. SPONSORING AGENCY NAME AND ADDRESS Chemistry and Physics Laboratory National Environmental Research Center Research Triangle Park, N.C. 27711		13. TYPE OF REPORT AND PERIOD COVERED FINAL
		14. SPONSORING AGENCY CODE
15. SUPPLEMENTARY NOTES		

16. ABSTRACT

Transmission electron micrographs and corresponding selected area electron diffraction patterns of standard specimens of serpentine and amphibole asbestos are presented for use by analysts as an aid in identification. Micrographs and diffraction patterns of typical ambient air samples and of certain minerals that often occur with airborne asbestos are also included. Specimens were uniformly prepared and examined in a single electron microscope.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Asbestos Electron microscopy Electron diffraction		
18. DISTRIBUTION STATEMENT Release unlimited	19. SECURITY CLASS *(This Report)* unclassified	21. NO. OF PAGES 58
	20. SECURITY CLASS *(This page)* unclassified	22. PRICE

EPA Form 2220-1 (9-73)

☆ U.S. GOVERNMENT PRINTING OFFICE· 1975-640-884