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Bureau of Mines Information Circular/1977

Selected Silicate Minerals and Their Asbestiform Varieties

Mineralogical Definitions and Identification-Characterization

UNITED STATES DEPARTMENT OF THE INTERIOR

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Selected Silicate Minerals and Their Asbestiform Varieties

Mineralogical Definitions and Identification-Characterization

By W. J. Campbell, R. L. Blake, L. L. Brown, E. E. Cather, and J. J. Sjoberg

This current report on asbestos has been prepared by the Bureau of Mines, U.S. Department of the Interior to—

1. Provide precise nomenclature and information on selected silicate minerals and their asbestiform varieties.
2. Invite comment, revisions, or additional information on the subject.

Please direct communications to the author—
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UNITED STATES DEPARTMENT OF THE INTERIOR
Cecil D. Andrus, Secretary

BUREAU OF MINES

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SELECTED SILICATE MINERALS AND THEIR ASBESTIFORM VARIETIES

Mineralogical Definitions and Identification-Characterization

by

W. J. Campbell,[1] R. L. Blake,[2] L. L. Brown,[3] E. E. Cather,[4] and J. J. Sjoberg[5]

ABSTRACT

This report by the Federal Bureau of Mines Particulate Mineralogy Unit recommends mineralogical definitions and identification-characterization concepts for selected silicate minerals and their asbestiform varieties. Precise definitions acceptable to mineral analysts, regulatory personnel, and medical scientists are essential because of the present lack of conformity in terminology concerned with measuring and controlling asbestiform particulates and their related health effects. Because of the complexity and variability of crystal morphology in different mineral groups, the descriptive terms are generally explained by illustration rather than by numerical values. Applications and limitations of several analytical techniques for particulate identification and characterization are discussed.

INTRODUCTION

Concurrent with concerns within the Federal Government over future availability of minerals to meet our expanding needs is the requirement that minerals and mineral commodities be mined and processed with minimum environmental impact. Traditionally, matters related to "... inquiries and scientific and technologic investigations concerning mining, and the preparation, treatment, and utilization of mineral substances with a view to improving health conditions and increasing safety...." have been within the province of the Bureau of Mines as authorized in the amended Organic Act of 1913 (Public Law 62-386). Since its establishment by Congress, the Bureau of Mines has long been deeply involved in investigating the explosive characteristics of dusts in the mineral industries in its mining and metallurgy research centers, and has established analytical and mineralogical laboratories in seven metallurgy research centers. These laboratories are essential to solving the increasingly complex

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safety, health, and environmental problems posed by dusts within the mineral system.

Various legislative actions and public concerns within the past decade have had, and will continue to have, an impact upon the mineral industry. As a result, control of mineral particulates is becoming increasingly important, with much recent attention focused on asbestiform particulates in both air and water. Figure 1, from an Environmental Protection Agency report, shows the widespread occurrence of common amphibole and serpentine minerals that, according to existing regulatory definitions, may be classified as asbestiform minerals (16).[6] With such possibly overwhelming implications to both mineral producer and mineral consumer, it is essential that existing ambiguities regarding silicate minerals and their asbestiform varieties be resolved. Until recently, adverse health effects associated with asbestos were focused on occupational exposure in asbestos-related industries. Now there is international concern regarding the effect on health from long-term low-level, or short-term high-level, exposure to mineral particulates by the general public (5, 28, 36). These particulates may include both the common and the asbestiform varieties of certain silicate minerals. In many instances, cleavage fragments of common amphibole minerals have been mistakenly identified as

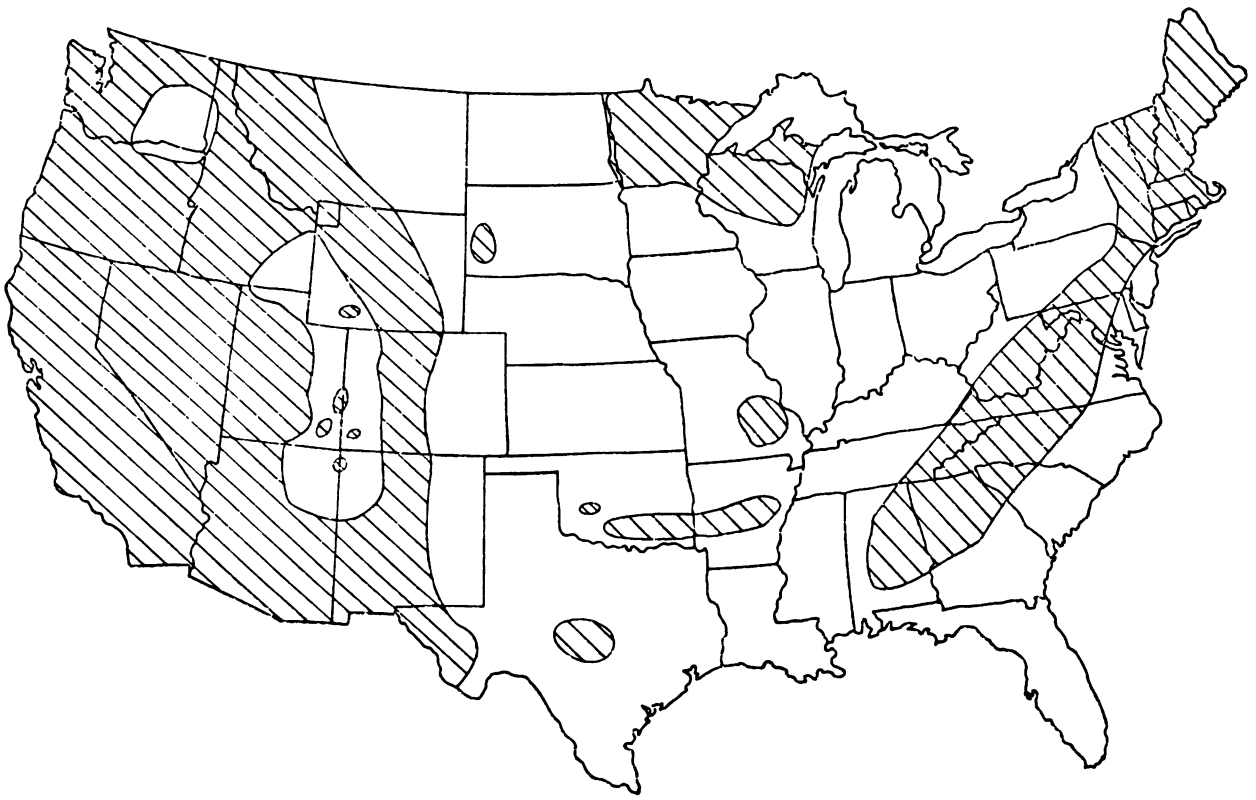


FIGURE 1. - Regions of the United States (shaded area) reported by the Environmental Protection Agency to contain asbestiform minerals in the bedrocks (16).

[6]Underlined numbers in parentheses refer to items in the list of references at the end of this report.

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microscopic fibers of the related asbestiform variety. Such lack of precision in identifying these particulates is a handicap to scientific decisionmaking by regulatory agencies and medical researchers. The Particulate Mineralogy Unit was created to work on problems such as this.

The Particulate Mineralogy Unit

The Bureau of Mines established the Particulate Mineralogy Unit in September 1976. The College Park Metallurgy Research Center in College Park, Md.,[7] is the focal point for this unit, but substantial support will be supplied by the other Bureau of Mines metallurgy and mining research centers, which are located throughout the United States. The unit is to assist local, State, and Federal agencies in establishing precise and workable mineral definitions and to improve or develop methods of particulate identification and quantitative measurement. The unit is also providing characterized serpentine and amphibole minerals for use by Federal health agencies in their asbestos-related research programs.

Scope of Report

This Bureau of Mines report is intended to clarify some of the terminology used in identification and characterization of asbestiform minerals, and to sharpen the distinction between common rock minerals and their asbestiform varieties. It defines certain mineral terms related to asbestiform minerals and discusses mineral-characterization techniques on a strictly mineralogical basis. The report then discusses the identification of silicate particulates and suggests how to apply this information to asbestos-related problems. Suggested areas for further research are summarized at the end of this report.

ACKNOWLEDGMENTS

The following College Park Metallurgy Research Center personnel are acknowledged for their invaluable contributions. Photographic assistance was provided by Garrett Hyde, research physicist, and Lawrence Johnson, geologist (mineralogy). Application data were provided by Raymond Brown, physical science technician; Charles W. Huggins, research chemist; and Eric Steel and Robert Virta, geologists (mineralogy).

[7]The research center is scheduled for relocation to Avondale, Md., in June 1978.

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NOMENCLATURE OF SELECTED SILICATE MINERALS AND THEIR ASBESTIFORM VARIETIES[8]

Background

In August 1976, a preliminary paper (1) was presented from which the Bureau of Mines hoped to elicit suggestions from the mineralogical community for critically defining silicate minerals and their asbestiform varieties. Significant terms such as "asbestos," "asbestiform," and "fibers" have different meanings to the various concerned groups--mineralogists, regulatory agencies, medical scientists, and industry. This has resulted in situations where erroneous conclusions have been drawn. Thus, precise definitions are required that will be uniformly accepted and applied by all personnel involved with silicate minerals that have asbestiform varieties (4, 11, 24-25, 31, 39).

Mineralogical nomenclature recognizes the historical origin of terms, and changes are made only when they are proven incorrect by new information (10, 32). The science of mineralogy is constantly being advanced with newly examined mineral occurrences and with new and improved instruments and techniques that provide more details on chemical composition, crystal structure, and morphology. To prevent constant revision, nomenclature has to have a certain flexibility, yet must be definite enough to be scientifically useful. The objective of this part of the report is to summarize mineralogically acceptable terms that relate to asbestos.

There is no "group" of asbestos minerals. "Asbestos" is a general term applied to certain minerals (that are themselves classified under crystal-structure-based groups) when these minerals crystallize as the asbestiform variety. Table 1 lists some common silicate minerals and their asbestiform varieties, together with their relationships and formulas. Although discussion in this report is limited to these minerals, appropriate terms and statements also apply to other silicate minerals that have rare fibrous varieties such as talc, some clay minerals such as attapulgite, and other amphiboles such as arfvedsonite, eckermannite, and richterite.

TABLE 1. - Selected silicate minerals and their asbestiform varieties

Mineral	Asbestiform variety
AMPHIBOLE GROUP	
Anthophyllite: $(Mg,Fe^{+2})_7\ Si_8O_{22}(OH,F)_2$...	Anthophyllite asbestos.
Cummingtonite-grunerite: $(Mg,Fe^{+2})_7\ Si_8O_{22}(OH)_2$.	Cummingtonite-grunerite asbestos.
Tremolite-actinolite: $Ca_2(Mg,Fe^{+2})_5\ Si_8O_{22}(OH,F)_2$.	Tremolite-actinolite asbestos.
Riebeckite: $Na_2Fe_3^{+2}\ Fe_2^{+3}\ Si_8O_{22}(OH,F)_2$....	Crocidolite.
SERPENTINE GROUP	
Serpentine: $Mg_6Si_4O_{10}(OH)_8$	Chrysotile.

[8]Tibor Zoltai, Professor, Department of Geology and Geophysics, University of Minnesota, made significant contributions to this section of the report.

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Amphibole minerals and, to a lesser degree, serpentine minerals occur widely distributed in the earth's crust in many igneous or metamorphic rocks. In some rare instances, the mineralogical occurrences contain sufficient quantities of usable asbestiform minerals to be economically minable for commercial asbestos.[9] The soft, silky fibers of asbestos (sometimes called mineral silk) are so flexible that they can be spun into threads from which cloth can be woven. The resulting material is fireproof, is a good thermal and electrical insulator, and has moderate to good resistance to acids. It has been used from Roman times, and is most familiar in daily use in brake linings for automobiles and as the "asbestos" siding used in residential construction.

Only a very small quantity of the amphibole and serpentine minerals under particular geologic circumstances occur as the asbestiform variety of the mineral. The asbestiform varieties occur in veins or small veinlets within rock containing or composed of the common (nonasbestiform) variety of the same mineral. Macrophotographs of the minerals of table 1, both asbestiform and nonasbestiform varieties, are shown in figures 2 through 7.

The serpentine group of minerals is limited to serpentine as the common variety and to chrysotile as the asbestiform variety. Antigorite and lizardite are not listed as separate varieties, but are understood to be included in the term "serpentine" because they represent 2 frequently named polytypes of about 10 recognized polytypes of serpentine (20). The polytypes differ only in minor structural stacking of components and are not sufficiently different to have a separate mineral status. Chrysotile generally occurs segregated as parallel fibers in veins or veinlets, although a recent study (6) has shown fine chrysotile intimately intergrown with the lizardite polytype.

The minerals and mineral series of the amphibole group in table 1 have variable compositions with extensive elemental substitutions and are found in forms ranging from massive to blocky to very fibrous. Crocidolite is the varietal name given to the fibrous habit of the mineral riebeckite (fig. 7), as shown by at least one study (35). It is retained here as a useful and correctly identified term.

Cummingtonite-grunerite asbestos is the mineralogically proper terminology for the commercial material commonly known as "amosite." Amosite, implied to be a mineral variety, is really an acronym--Asbestos Mines of South Africa--for a fibrous mixture of minerals; namely, cummingtonite-grunerite with variable tremolite-actinolite. Amosite has been discredited as a mineral species (22), and its use as a mineralogical term should be discontinued; however, it is still useful as a commercial term.

Mineral compositional series such as cummingtonite-grunerite involve replacing one cation for another in a crystal structure without significantly altering the structure. There may be a gradation in the structure in some series, and minor changes in physical characteristics may occur with elemental substitution. Usually a series involves two named compositional end members with intermediate substitutional compounds being separately named (if the members were recognized by early mineralogists), given a varietal name (for similar reasons), or just qualified by being referred to as members of the series. Members of the tremolite-actinolite-ferroactinolite series are hydroxylated calcium-magnesium, magnesium-iron, and iron silicates, respectively. Their series is named in table 1 for two of its members, and when its composition is known, it should be called by the specific name, such as

[9]Clifton, R. A. Asbestos. BuMines MCP-6, in preparation, 1977.

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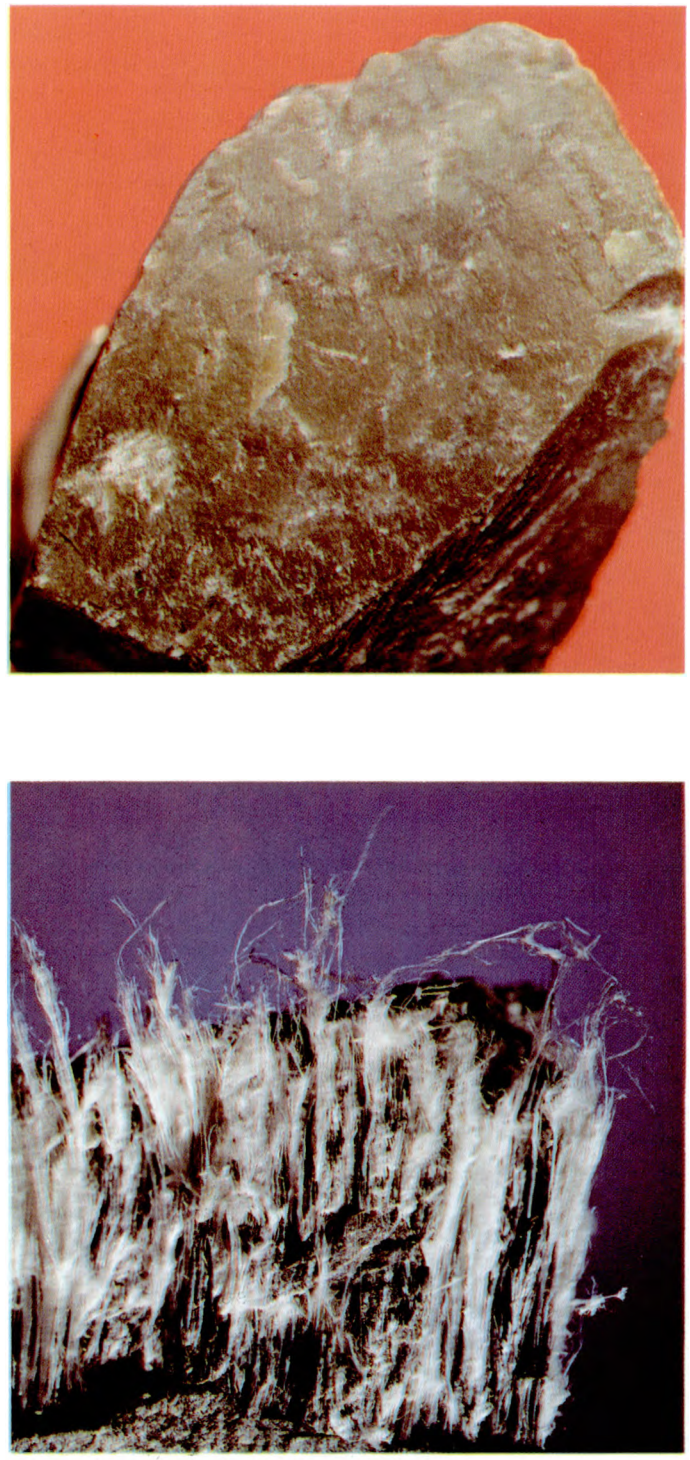


FIGURE 2. - Macrophotographs of serpentine (top, X 1) and chrysotile (bottom, X 3).

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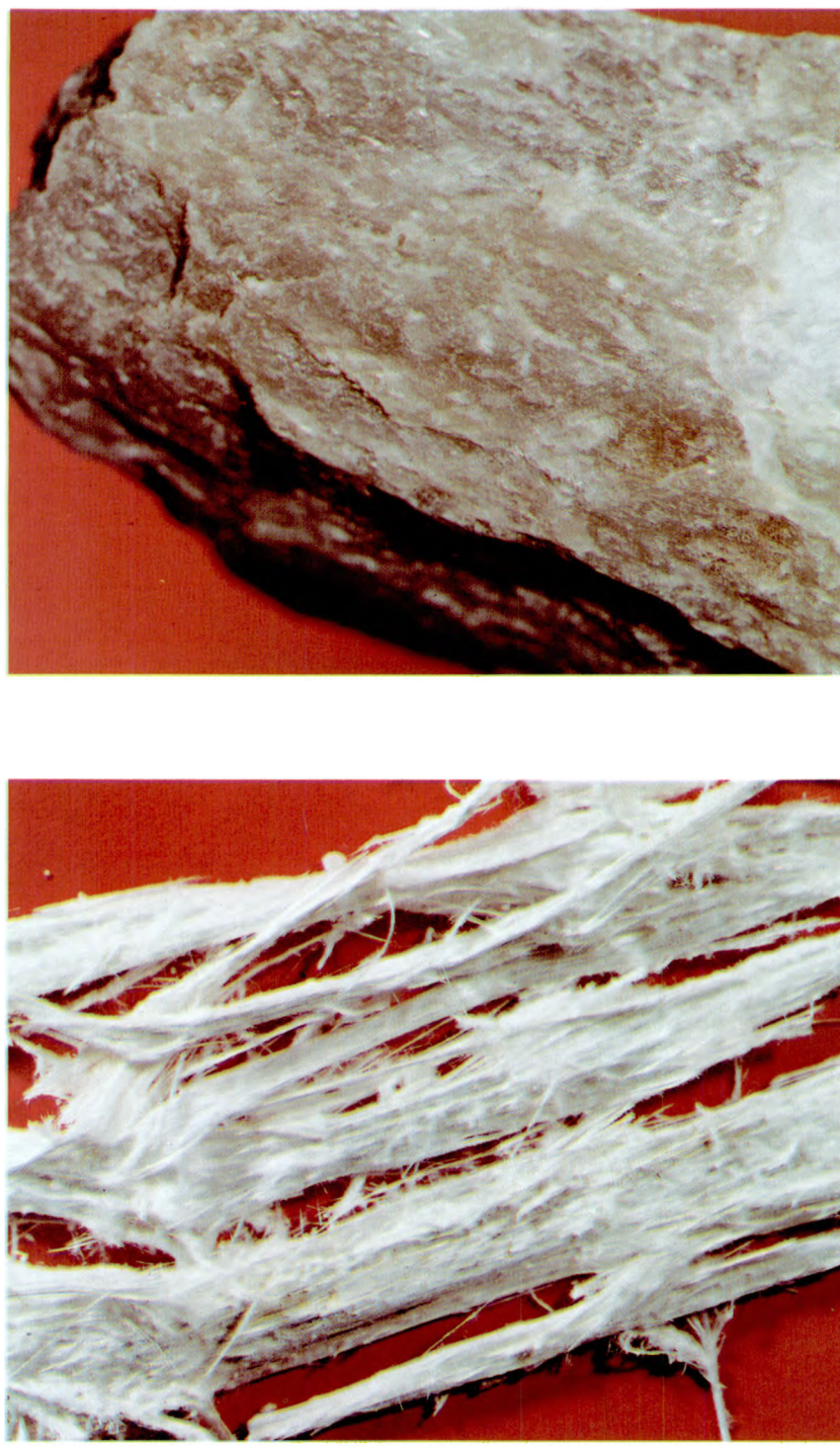


FIGURE 3. - Macrophotographs (X 3) of tremolite (top) and tremolite asbestos (bottom).

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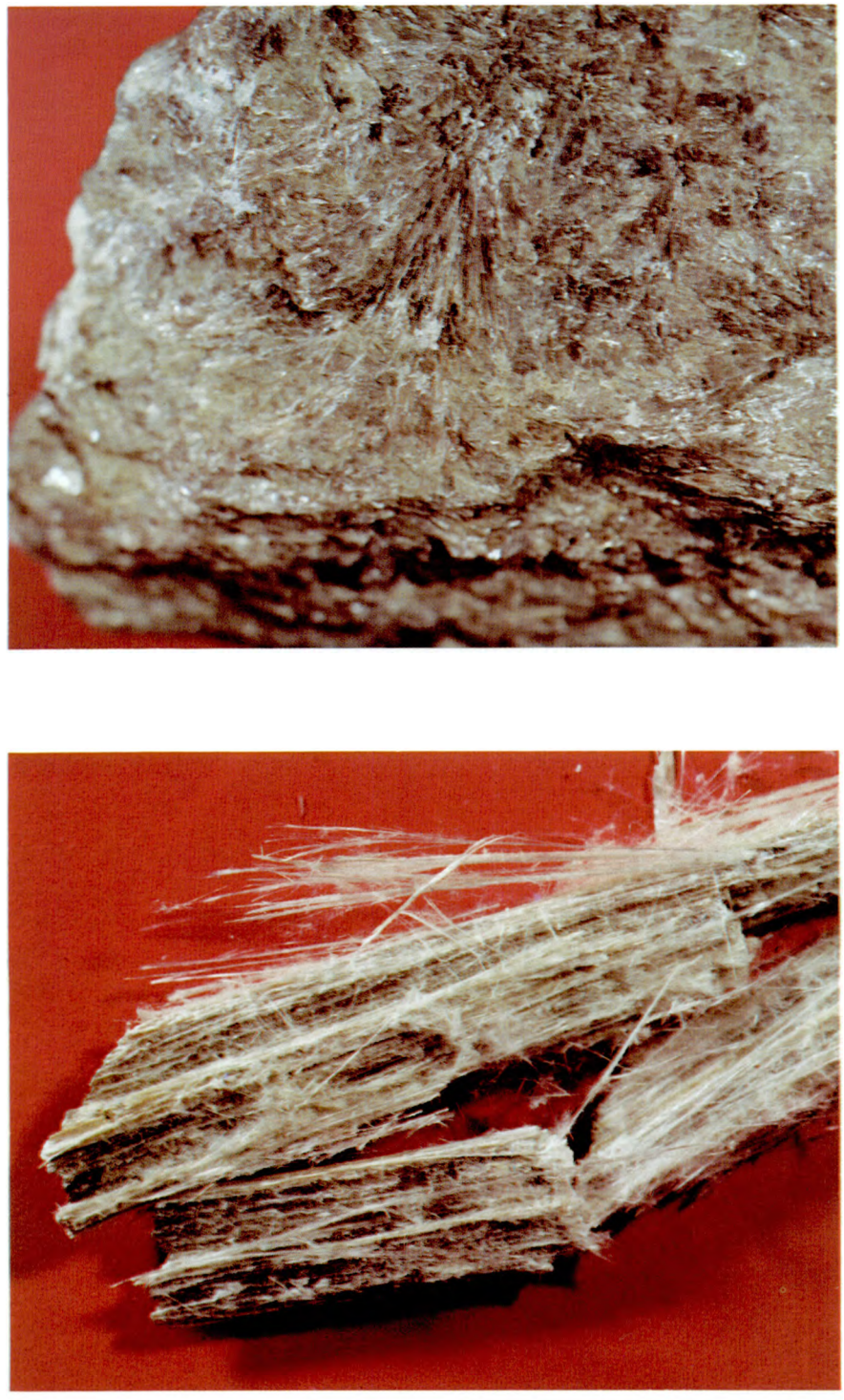


FIGURE 4. - Macrophotographs (X 3) of anthophyllite (top) and anthophyllite asbestos (bottom).

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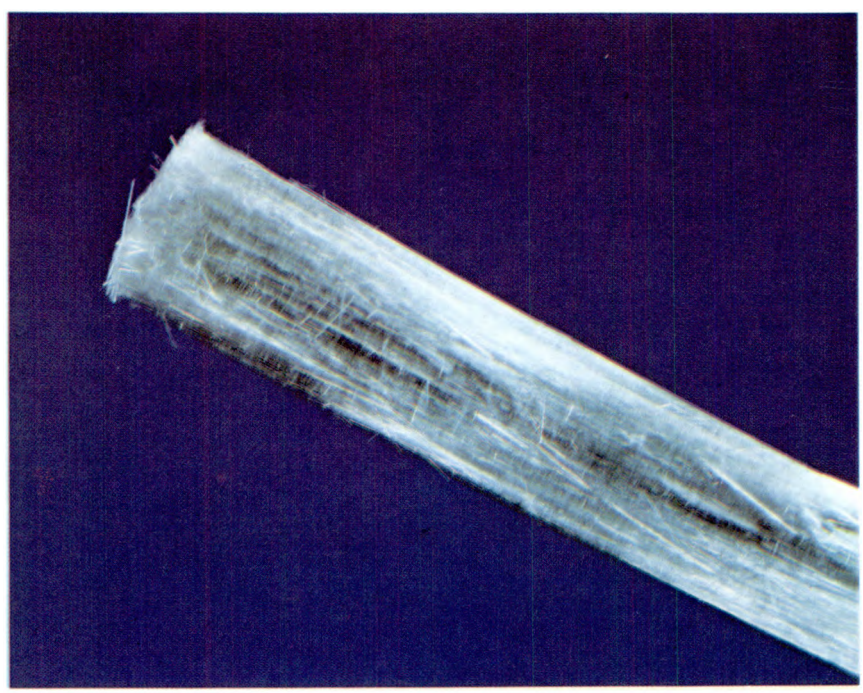


FIGURE 5. - Macrophotographs of actinolite (top, X 1) and actinolite asbestos (bottom, X 3).

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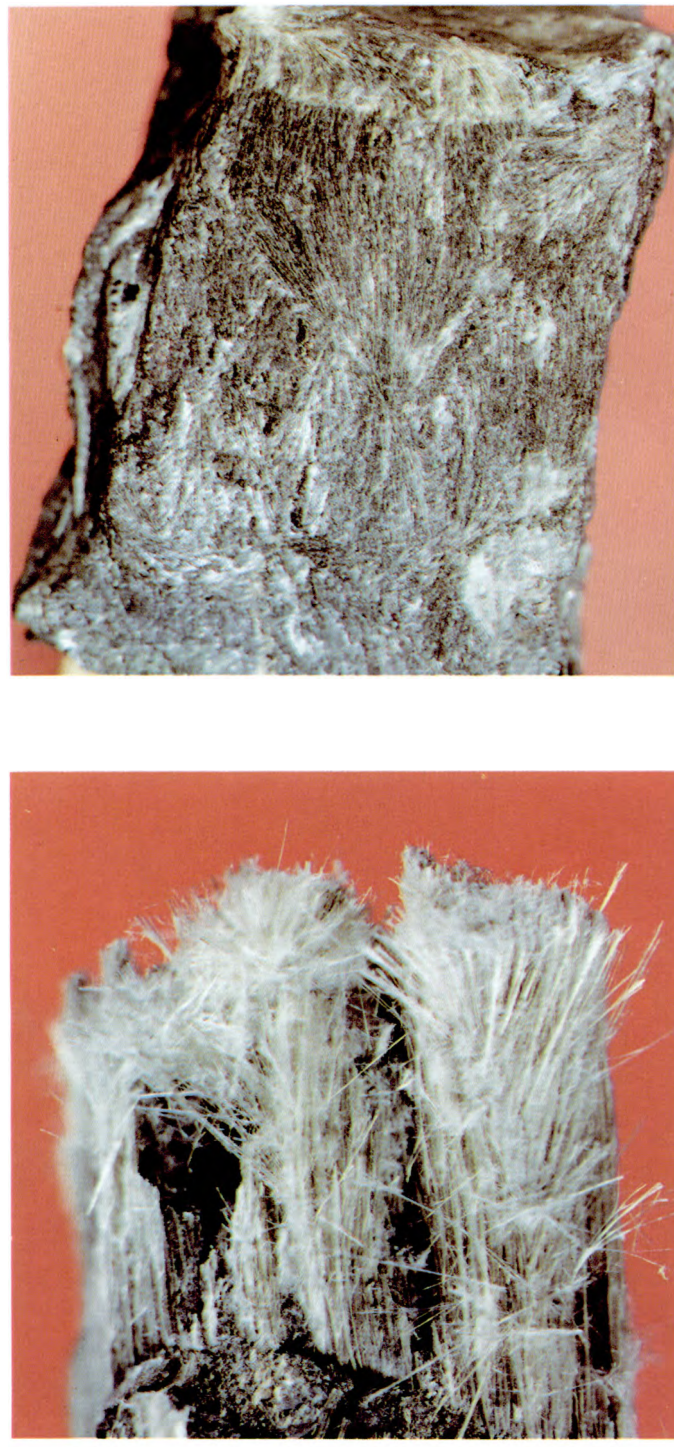


FIGURE 6. - Macrophotographs (X 3) of cummingtonite (top) and cummingtonite-grunerite asbestos (bottom).

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FIGURE 7. - Macrophotographs (X 3) of riebeckite (top) and crocidolite (bottom).

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tremolite. The following nomenclature for this series (7) is based on the mole ratio (in parentheses) of $\frac{Fe}{Fe + Mg}$ in the formula $Ca_2(Mg,Fe)_5Si_8O_{22}(OH)_2$: tremolite (0 to 0.2), actinolite (0.2 to 0.8), and ferroactinolite (0.8 to 1.0).

Definitions

Many mineralogical definitions apply to the entire mineral field, but the definitions in this report are restricted to those necessary to characterize the fibers and cleavage fragments related to asbestos identification-characterization. The terms to be defined are outlined in the following list:

- Mineral terms:
  - Mineral
  - Mineral groups
  - Mineral series
  - Mineral varieties
- Asbestos-related terms:
  - Asbestos
  - Fibrous
  - Mineral fiber
  - Fibril
  - Fibril structure
  - Asbestiform
- Crystal terms:
  - Crystal structure
  - Crystalline
  - Crystal
  - Single crystal
  - Twinned crystal
  - Polycrystalline
  - Polymorphs
  - Polytypes
  - Crystal form
  - Crystal morphology:
    - Single crystals:
      - Equant
      - Prismatic
      - Acicular
      - Fiber
      - Fibril
      - Filiform
      - Bladed
      - Platy
      - Lamellar
    - Crystal aggregates:
      - Asbestiform
      - Columnar
      - Fibrous
      - Lamellar
      - Massive
      - Radiating
      - Reticulated
- Breaking of minerals:
  - Cleavage:
    - Types:
      - Pinacoidal
      - Prismatic
      - Rhombohedral
      - Cubic
      - Octahedral
    - Quality:
      - Perfect
      - Good
      - Fair
      - Imperfect
    - Cleavage fragment
  - Fracture: Types:
    - Even
    - Uneven
    - Splintery
    - Conchoidal
    - Hackly
  - Parting

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Mineral Terms

Mineral.--A homogeneous, naturally occurring, usually inorganic and crystalline substance. Minerals with distinct crystal structure types (including lack of crystal structure) and limited variation in chemical composition are given individual names.

The early concept of a mineral as a natural-history species was gradually abandoned and changed into a chemical and structural definition around the middle of the 19th century, long before crystal structures were understood. The term "species" persisted for a long time with this newer definition, but is now seldom used in mineralogy.

Mineral Group.--Minerals that have essentially the same (or comparable) structures, but have different cations in secondary structural position (for example, pyrites, feldspars, and amphiboles).

In silicates, most of the mineral groups have the same linkage patterns of the silica tetrahedra, like the characteristic double chains of amphiboles. However, the secondary atomic sites may be occupied by a relatively wide variety of cations or some may even be vacant in the actual structures of the members. In some silicate-mineral groups, the identity of the silica-tetrahedral frame is less restricted and may be limited to the similarities of some basic characteristics. For example, the silica-tetrahedral frames of the zeolite minerals are variable, but they are all characterized by large open channels.

All mineral groups have names. In some instances, this name is the universal name of a common or important member of the group (for example, serpentine group).

Mineral Series.--Two or more members of a mineral group in which the cations in secondary structural position are similar in properties and can be present in variable, although frequently limited, ratios (for example, cummingtonite-grunerite). Also known as an isomorphic series.

Some mineral series such as the plagioclases have unique names, but most are identified by the combined names of the end-member minerals, such as tremolite-actinolite. The current trend is to simplify long series names by using the mineral name of only one (end or intermediate) member.

Individual minerals in the series are either identified by the names given to compositional ranges (for example, bytownite or oligoclase in the plagioclases), or by the name of the series followed by a symbol expressing the mineral's position in the series or the ratios of the variable cations ($Ab_{x\%}An_{100-x\%}$ for plagioclases, where Ab and An designate the two end members, albite and anorthite).

Mineral Variety.--Minerals that are conspicuously different from those considered normal or common in crystallization habits, polytypes, and other structural variants, or other physical properties such as color. Varieties are named by mineralogists, miners, gemologists, manufacturers of industrial products, and mineral collectors.

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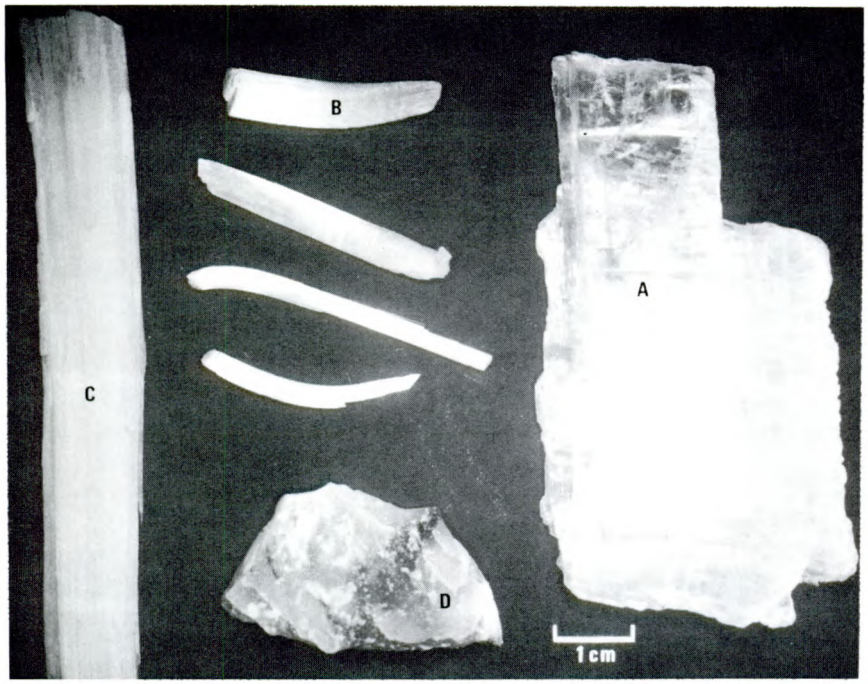


FIGURE 8. - Four varieties of gypsum: (A) Selenite; (B) satin spar, fine fibrous; (C) satin spar, coarse fibrous; and (D) alabaster.

Although mineral names are controlled by national and international mineralogical organizations, variety names are not. In practice, any variety name that becomes sufficiently popular is eventually recognized by these organizations as distinct enough to be used as a mineral-variety name. Typical of these are the various varieties of gypsum shown in figure 8. Figures 2 through 7 illustrate the massive and asbestiform varieties of serpentine and amphiboles.

Asbestos-Related Terms

In the following discussion, asbestiform refers only to asbestos. The other terms, "fibrous," "mineral fiber," "fibril," and "fibril structure," apply to both asbestiform and nonasbestiform varieties.

Asbestos.--(1) A collective mineralogical term encompassing the asbestiform varieties of various minerals; (2) an industrial product obtained by mining and processing primarily asbestiform minerals.

The quality of asbestos depends on the mineralogy of the asbestiform variety, the degree of asbestiform development of the fibers, the ratio of asbestiform fibers to acicular crystals or other impurities, and the length and flexibility of the fibers. The major asbestiform varieties of minerals used for asbestos are chrysotile, tremolite-actinolite asbestos, cummingtonite-grunerite asbestos, anthophyllite asbestos, and crocidolite. Asbestos may be marketed by its mineral name such as anthophyllite asbestos, its variety name such as chrysotile or crocidolite, or a trade name such as Amosite or Montasite. Two types of commercial asbestos are shown in figures 9 and 10.

The term "asbestos" was first introduced by Plinius Secundus in 77 A.D. The term "amiant" was previously used for the same mineral by Dioscorides in 50 A.D., and this term was the more common one until the middle of the 17th century. After that until the 20th century, "asbestos" was more common, and "amiant" was reserved for the more silky and flexible asbestos. In the 18th century, asbestos was classified into five different species. Currently, all

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FIGURE 9. - Macrophotograph of tremolite asbestos (X 3).



FIGURE 10. - Macrophotograph of anthophyllite asbestos (X 3).

asbestos is recognized as varieties of several individual minerals. Thus, in mineralogy, "asbestos" became a collective term, somewhat like "clays" or "gems." During the 20th century, asbestos developed into an important industrial material. Some asbestos products contain nonasbestiform minerals (for example, asbestos-cement and asbestos-magnesia); consequently, the mineralogical and the industrial definitions of asbestos do not always coincide.

Fibrous.--The occurrence of a mineral in bundles of fibers, resembling organic fibers in texture, from which the fibers can usually be separated (for example, satin-spar and chrysotile).

The term "fibrous" has been used during the last 200 years to describe all kinds of minerals that crystallized in habits resembling organic fibers, including asbestos minerals. However, the related term "asbestiform" was never used for fibrous mineral habits other than asbestos. Accordingly, "fibrous" is the more general term, and asbestiform is a specific type of fibrosity. Figures 11, 12, and 13 show various types of fibrous mineral habits. Examples of fibrous minerals, both silicates and nonsilicates, that are not classified as asbestiform are shown in figure 13.

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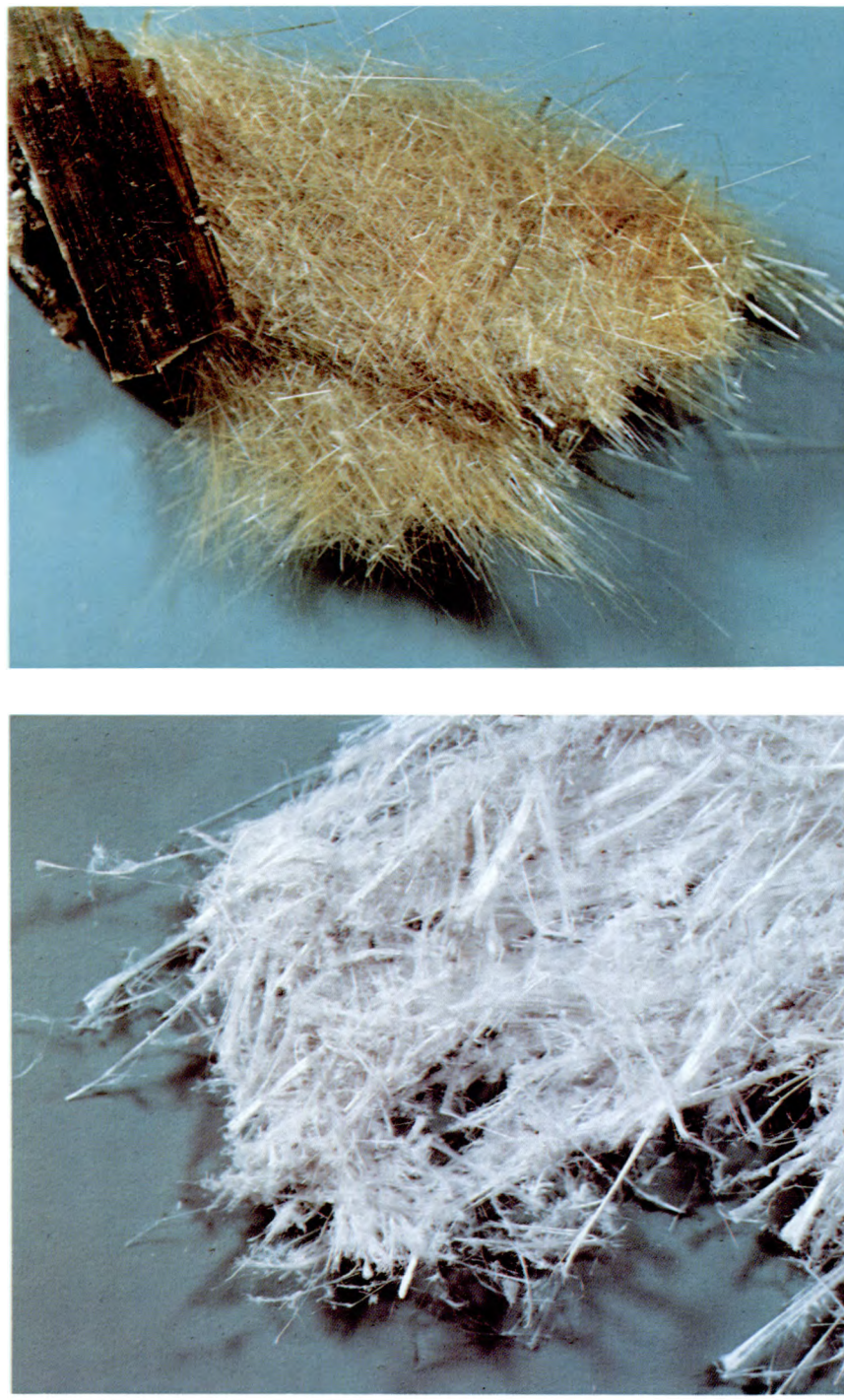


FIGURE 11. - Macrophotographs (X 3) of two fibrous amphiboles showing asbestiform habit: Byssolite (top) and richterite (bottom).

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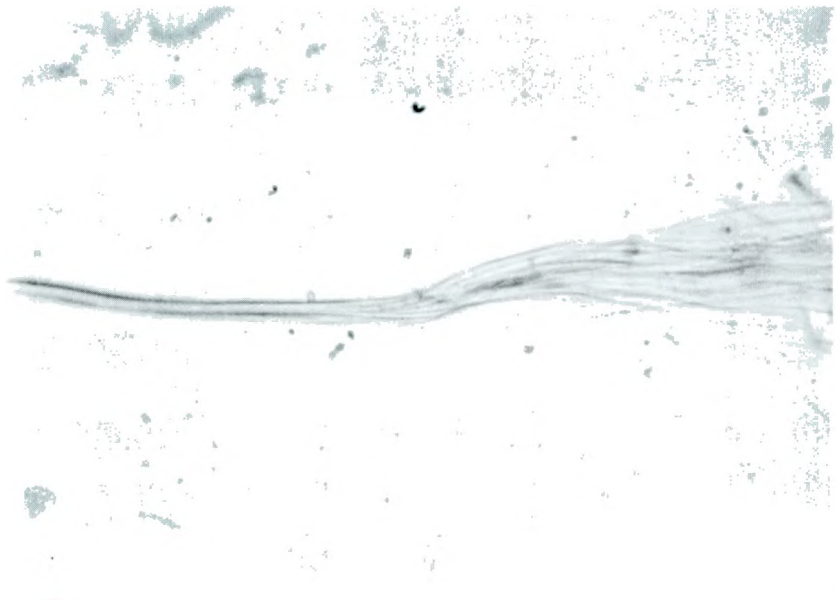


FIGURE 12. - Photomicrograph illustrating the separation of a crocidolite fiber bundle into fibers (X 600).

Mineral Fiber.--The smallest elongated crystalline unit that can be separated from a bundle or appears to have grown individually in that shape, and that exhibits a resemblance to organic fibers. (Examples: fiber bundles, chrysotile and crocidolite; individual fibers, epsomite and millerite.)

The term "fiber" is not limited to asbestos. However, it is distinct from "acicular" because it requires the resemblance to organic fibers. Figures 14 and 15 illustrate mineral fiber habits. Excellent photomicrographs of organic fibers are illustrated in The Particle Atlas (18).

Fibril.--A single fiber, which cannot be separated into smaller components without losing its fibrous properties or appearances.

Most fibers are single structural entities, such as millerite and nickel sulfide, and some may be called fibrils. However, some fibers are composed of two or more fibrils that are less readily separable from each other than fibers are from bundles (for example, chrysotile and crocidolite). Figure 16 shows the high magnification necessary to resolve a fibril.

Fibril Structure.--A systematically deformed and/or defective crystal structure of a fibril. A defect structure would involve various types of dislocation. The fibril structure may be exhibited by a single crystal, a group of single crystals, or a twinned single crystal.

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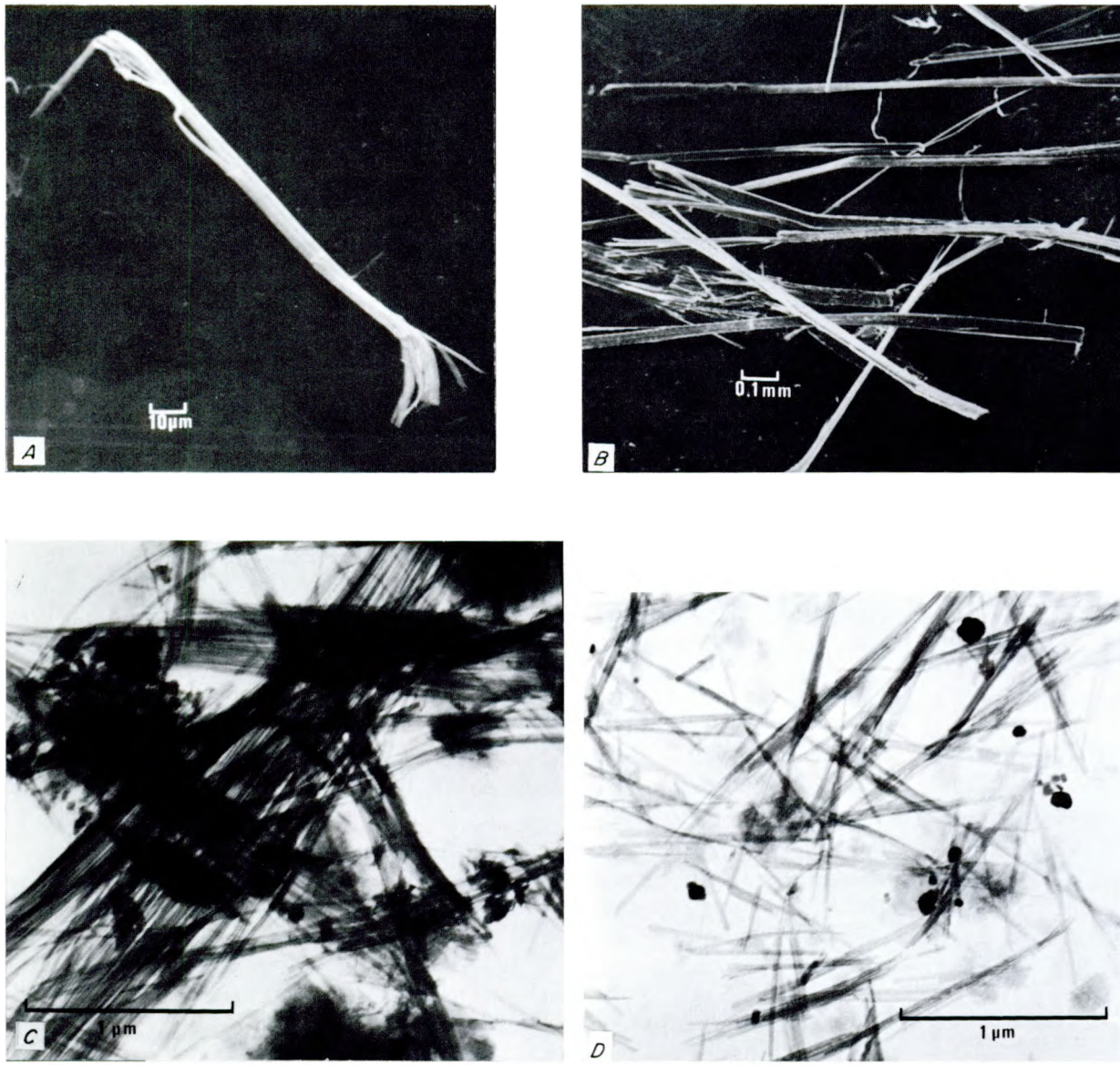


FIGURE 13. - Four fibrous nonasbestiform mineral varieties: *A*, Fibrous talc (X 500); *B*, fibrous brucite (X 50); *C*, palygorskite (X 30,000); and *D*, attapulgite (X 30,000).

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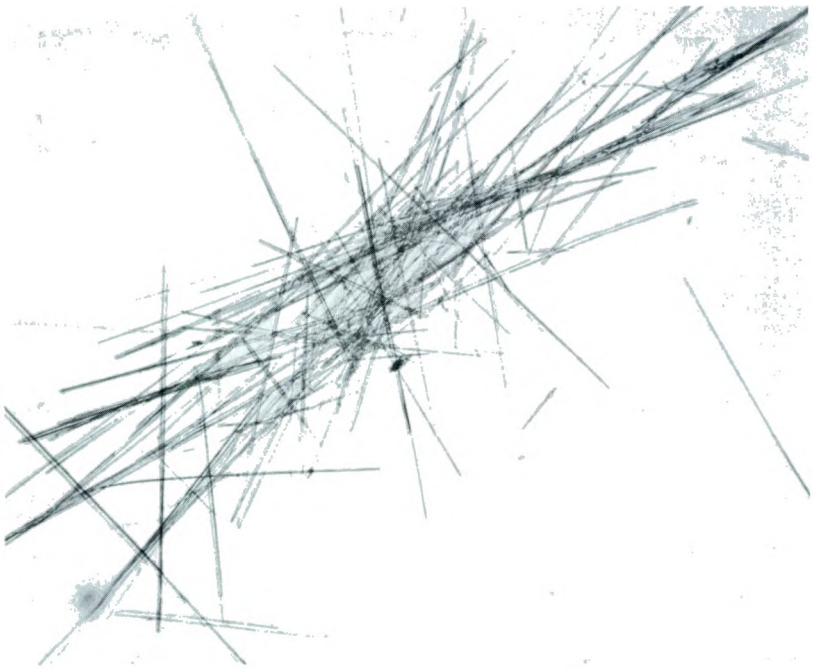


FIGURE 14. - Light optical photomicrograph of fibers from tremolite asbestos (X 115). A few particles are seen to be bundles of fibers.

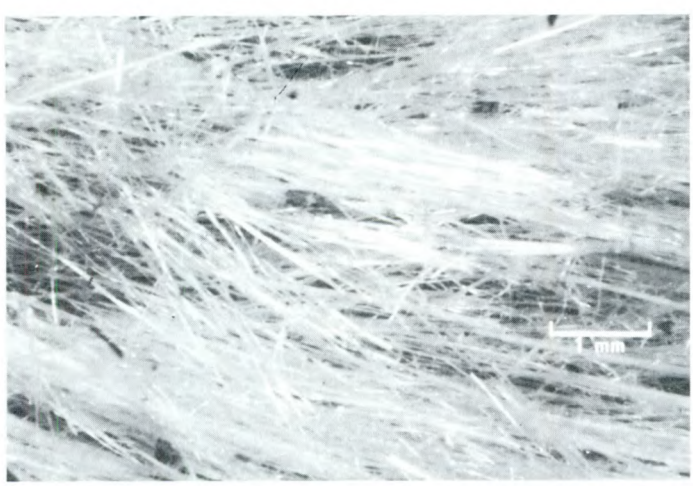


FIGURE 15. - Fibers of epsomite (magnesium sulfate hydrate) (X 13).

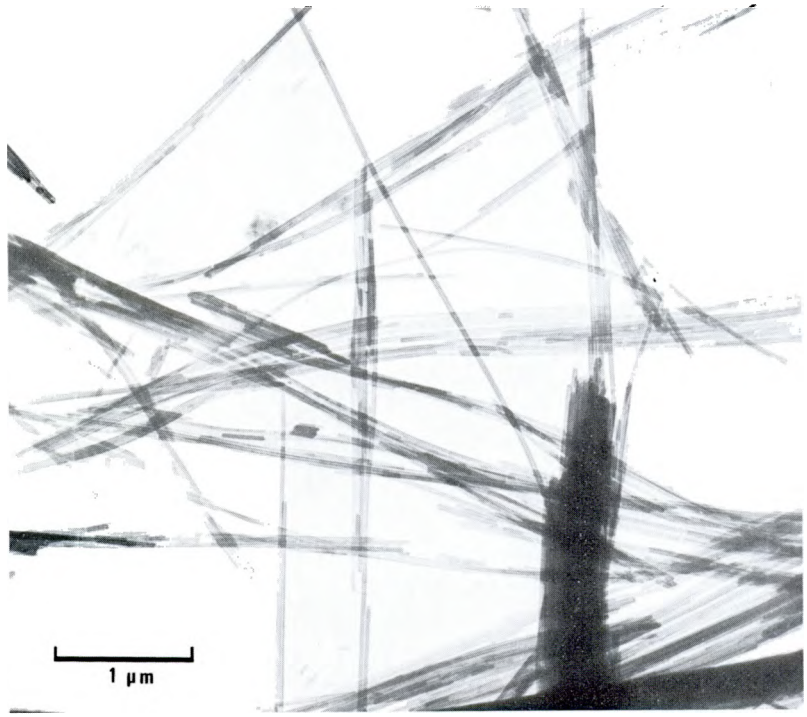
The scroll-like fibril structure of chrysotile (38), the twinned single-crystal fibrils of chrysotile (6), and the incompletely resolved fibril structure of an amphibole (9) are all examples illustrated in the literature.

Some acicular single crystals may have the appearance of fibers and fibrils, yet there is nothing unusual about their crystal structures. Other acicular single crystals may have significant structural deviations in addition to appearance that result in the display of certain properties usually found in fibers such as high tensile strength along the fiber axis. Thus, fibril structure is not limited to asbestiform structures, but may occur in a minor form in non-asbestiform structures.

Asbestiform.--A specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength and flexibility.

"Asbestiform" and "asbestos" are essentially synonymous in current usage. Some special properties of asbestiform varieties, including optical extinction and surface charge, are either not fully understood or are not uniformly applicable to all asbestiform fibers; consequently, they cannot be considered fundamental characteristics at this time. The prototype of the expression "asbestiform" was introduced by Werner in 1774. He recognized three subspecies of actinolite and of tremolite. One of these subspecies had the prefix "asbestartiger." Thus, the restriction of "asbestiform" to certain mineral varieties appears to

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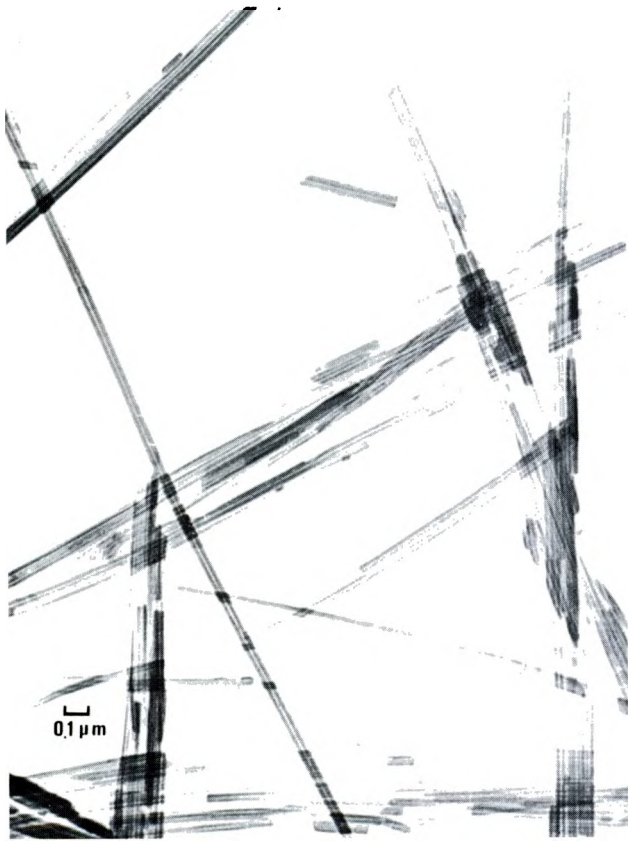


FIGURE 16. - Chrysotile, showing individual fibrils, at two magnifications: X 18,000 (top) and X 35,000 (bottom). The hollow-tube structure is visible at the higher magnification. (TEM micrographs.)

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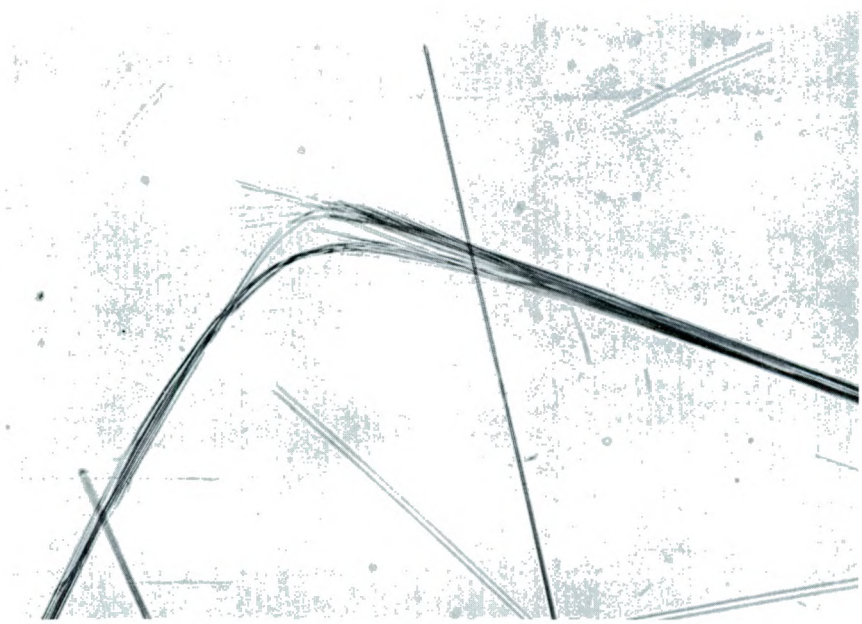


FIGURE 17. - Crocidolite, showing a fiber bundle and fibers (X 300).

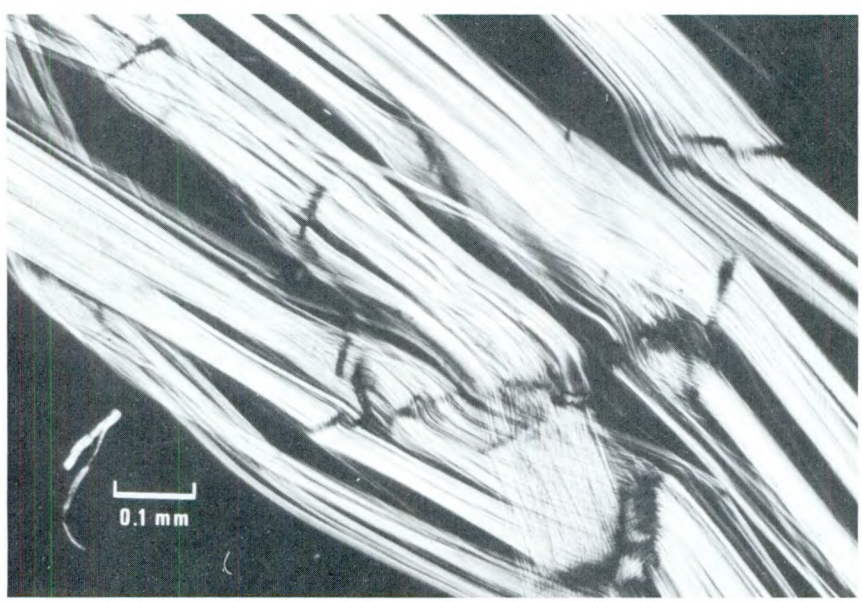


FIGURE 18. - Chrysotile by polarized light (X 100).

be justified on a historical as well as physical basis. Figures 17 to 20 show several asbestiform varieties of minerals.

Crystal Terms

Crystal Structure.--The pattern of the regular arrangement of atoms and ions in space. Usually refers to the basic (or average) structure of solids (and the ordered portions of liquids) without reference to minor, localized deviations.

Crystalline.--A substance possessing a reasonably well developed and long-range ordered crystal structure.

Crystal.--(1) Any single crystal; (2) a single crystal terminated by planar or nearly planar surfaces called crystal faces.

Single Crystal.--A crystal containing an uninterrrupted crystal structure in a single orientation.

Twinned Crystal.--A crystal composed of two or more single crystals where adjacent crystals share a plane that is an integral part of both orientations of the crystal structure.

Polycrystalline.--A substance composed of two or more single crystals.

Polymorphs.--Two crystals with identical chemical composition but different crystal structures.

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FIGURE 19. - Chrysotile (X 300).

Polytypes.--Polymorphs that contain essentially identical structural components, like layers, which are arranged in various patterns, like stacking of layers.

Crystal Form.--A set of symmetrically equivalent faces in a single crystal. A crystal may display several crystal forms.

Crystal Habit.--The actual shape assumed by a crystal or group of crystals as a result of the growth of dominant crystal forms (faces). Also known as crystal morphology.

Crystal Aggregate.--A cohesive mass of individual crystals or grains.

Single crystals exhibit growth shapes, while crystal aggregates exhibit growth patterns or arrangements (fig. 21). Some habits imply minor deviation of the crystal structure (deformation of the structure, presence of defects, inclusions, or impurities). Most habits are limited to the appearance of single crystals (crystal forms, irregular terminations, dimensional development, texture) or the pattern of aggregation of single crystals (dimension, orientation of the crystals, and cohesion between them). Under the following heading of "Single Crystals," the growth shapes are arranged first as equant, followed by other shapes that can be thought of as being derived from equant by suppressing or extending one or two of the three space dimensions, thus conveying the gradational nature of various defined crystal shapes.

Single Crystals

Equant.--The shape of a single crystal or grain with three approximately equal space dimensions.

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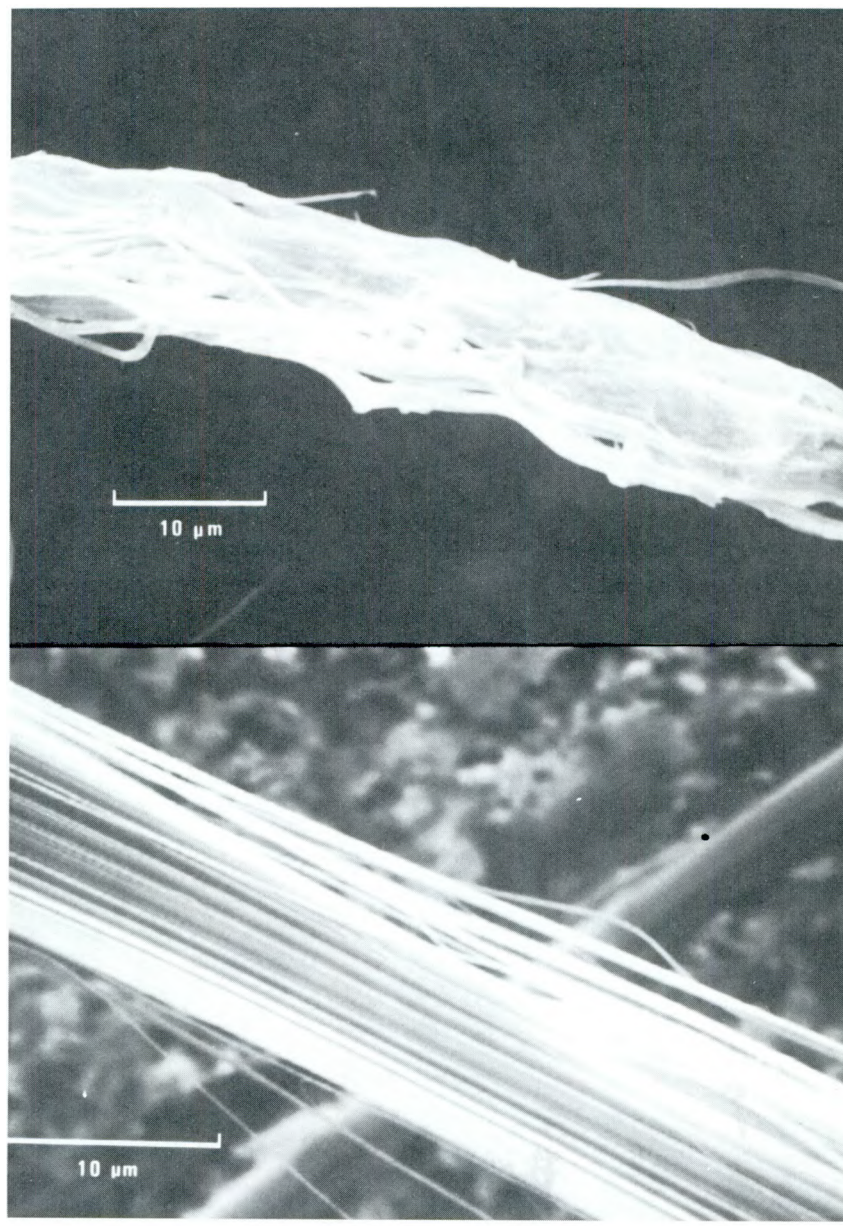


FIGURE 20. - Chrysotile at two magnifications: X 1,960 (top) and X 2,800 (bottom).

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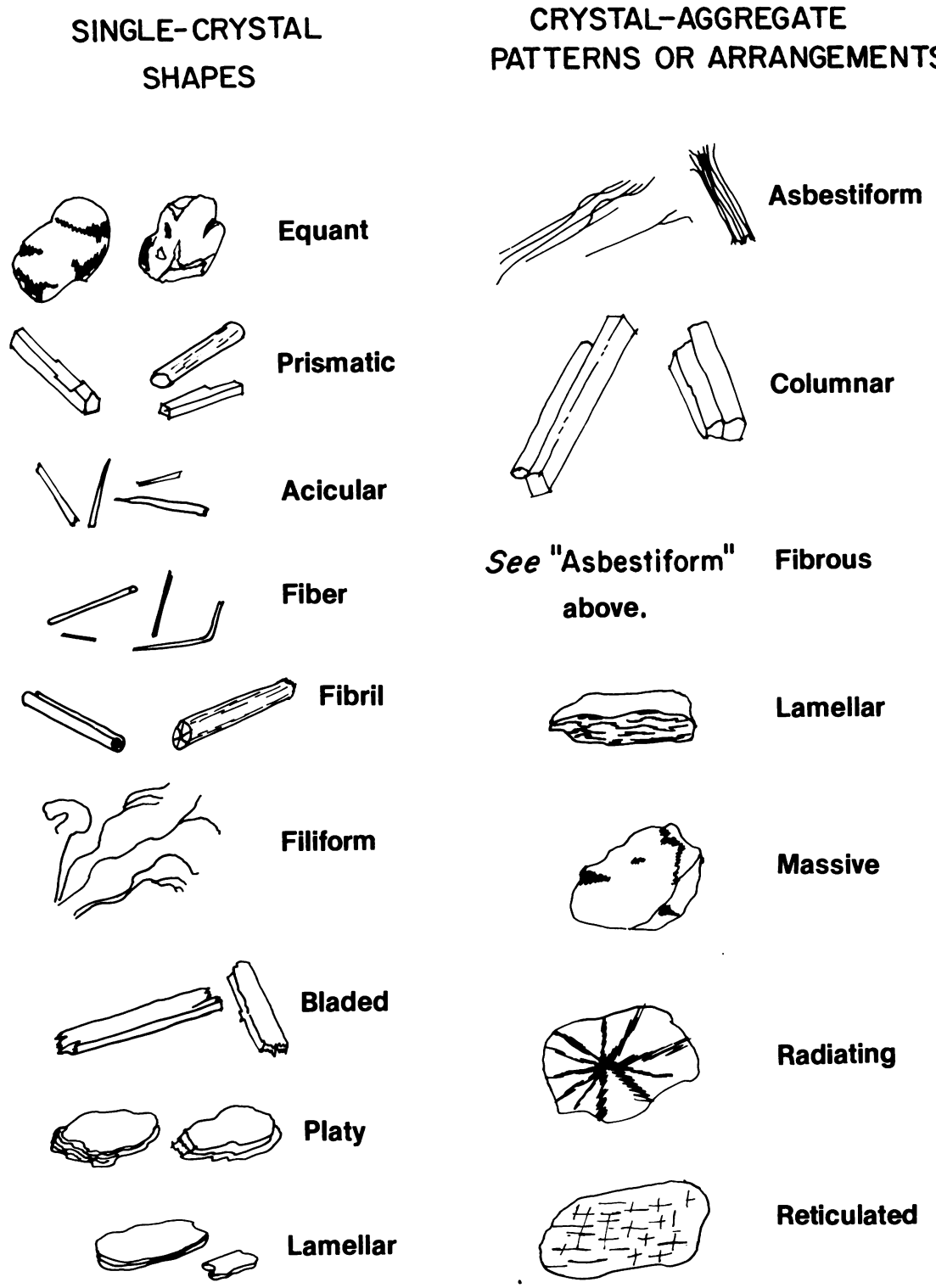


FIGURE 21. - Various shapes of single crystals, and patterns or arrangements of crystal aggregates.

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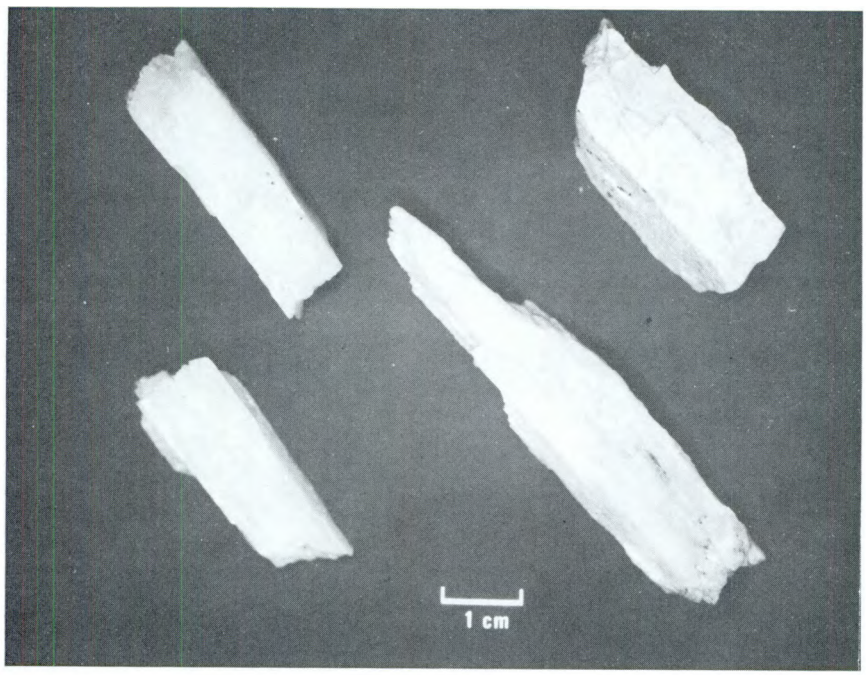


FIGURE 22. - Macrophotograph of spodumene ($LiAlSi_2O_6$) showing prismatic shape (X 1).



FIGURE 23. - Macrophotograph of tremolite showing prismatic and acicular crystal shapes (X 10).

Prismatic.--The shape of a single crystal with one elongated dimension and two shorter, approximately equal, dimensions.

Prismatic shapes of various single crystals are shown in figures 22 to 25.

Acicular.--The shape shown by an extremely slender crystal with small cross-sectional dimensions (a special case of prismatic form). Acicular crystals may be blunt-ended or pointed. The term "needlelike" refers to an acicular crystal with pointed termination at one or both ends.

Figures 23 and 26 show acicular crystals or crystal shapes.

Fiber.--See definition under "Asbestos-Related Terms."

Fibril.--See definition under "Asbestos-Related Terms."

Filiform.--The shape of threadlike mineral fibers.

Bladed.--The bladelike shape of a crystal with one longer dimension and two unequal, much shorter, dimensions.

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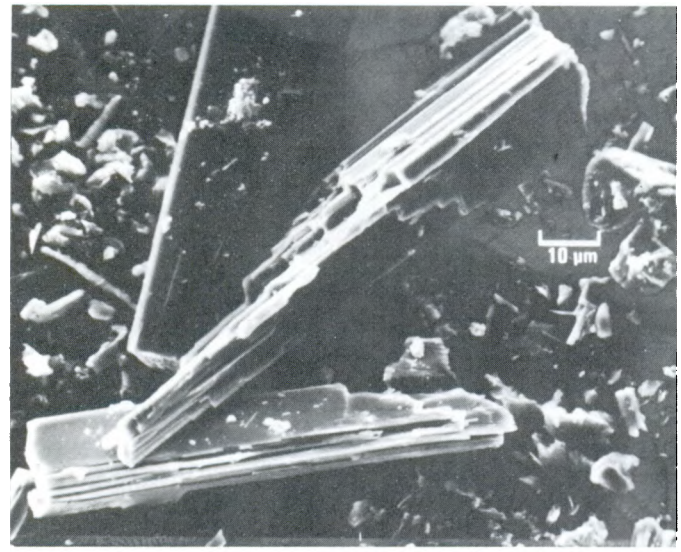


FIGURE 24. - Riebeckite, showing prismatic shape (SEM photomicrograph at X 800).

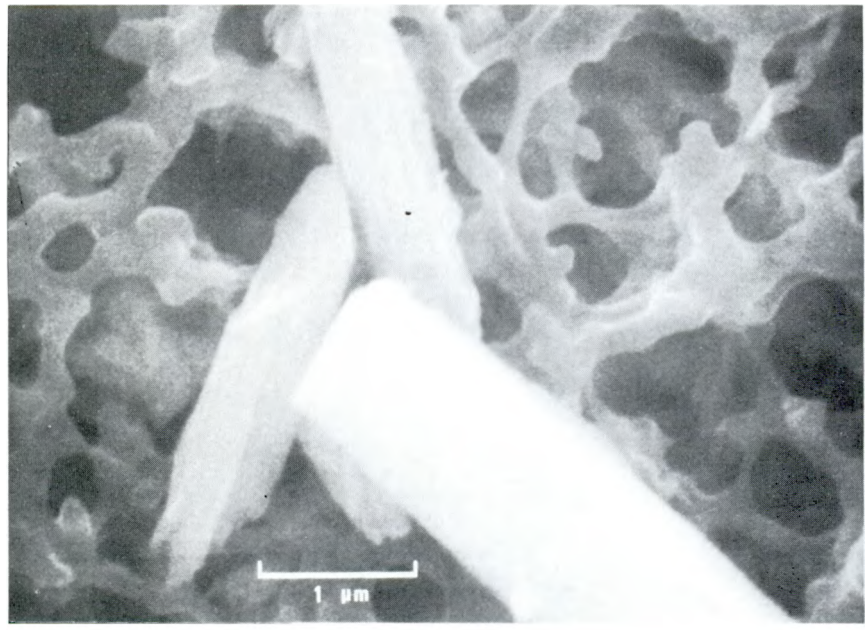


FIGURE 25. - Actinolite, showing prismatic shape (SEM photomicrograph at X 20,000).

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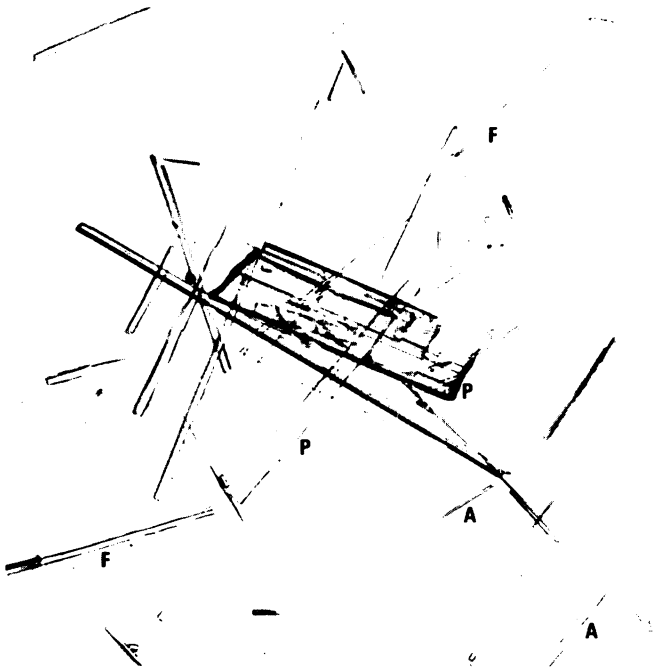


FIGURE 26. - Tremolite cleavage fragments (X 130), showing acicular (A), fibrous (F), and prismatic (P) shapes.

Figure 25 shows the bladed prismatic habit of actinolite.

Platy.--The shape of a crystal with one short dimension and two longer, approximately equal, dimensions. Chlorite, micas, and talc usually crystallize into platy shapes.

The serpentines, although possessing layered structure similar to micas and talc, rarely grow in platy shapes. Talc is of interest here because some talc deposits have associated asbestiform and nonasbestiform minerals. Platy talc, when seen in thin sections or as fragments in oil, may occur in various orientations. Plates lying flat look like plates but, if standing on edge, could appear fibrous.

Lamellar.--The shape of a very thin platy crystal.

Crystal Aggregates

Asbestiform.--See definition under "Asbestos-Related Terms."

Columnar.--The arrangement of a group of approximately parallel, prismatic, acicular, or bladed crystals.

Figure 27 shows columnar aggregates of coarse anthophyllite.

Fibrous.--See definition under "Asbestos-Related Terms."

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FIGURE 27. - Macrophotograph of columnar aggregates of coarse anthophyllite (X 1).

Lamellar.--The pattern exhibited by aggregates of very thin platy minerals.

Massive.--Homogeneous structure without stratification, flow-banding, foliation, or schistosity. Also, crystals or crystalline grains that are tightly packed and scarcely distinguishable.

Figure 2 (top) is a good example of massive serpentine.

Radiating.--An arrangement of prismatic, acicular, or bladed crystals that appear to be diverging from a common center.

Figure 28 shows radiating aggregates of pyrophyllite.

Reticulated.--The pattern of a crisscross network of acicular, prismatic, or bladed crystals.

Breaking of Minerals

When a mineral crystal or grain is strained beyond its elastic and plastic limit, it will break in one of several characteristic modes described as cleavage, fracture, or parting.

Cleavage.--The tendency of a crystal to break in definite directions that are related to the crystal structure and are always parallel to possible crystal faces.

Cleavage Types

Pinacoidal Cleavage.--A crystal with only one cleavage plane that yields platy or lamellar fragments (for example, talc and the mica minerals). Also called platy, basal, or lamellar cleavage.

Prismatic Cleavage.--A crystal with two distinct cleavage planes that yield prismatic fragments (for example, the amphiboles and pyroxenes).

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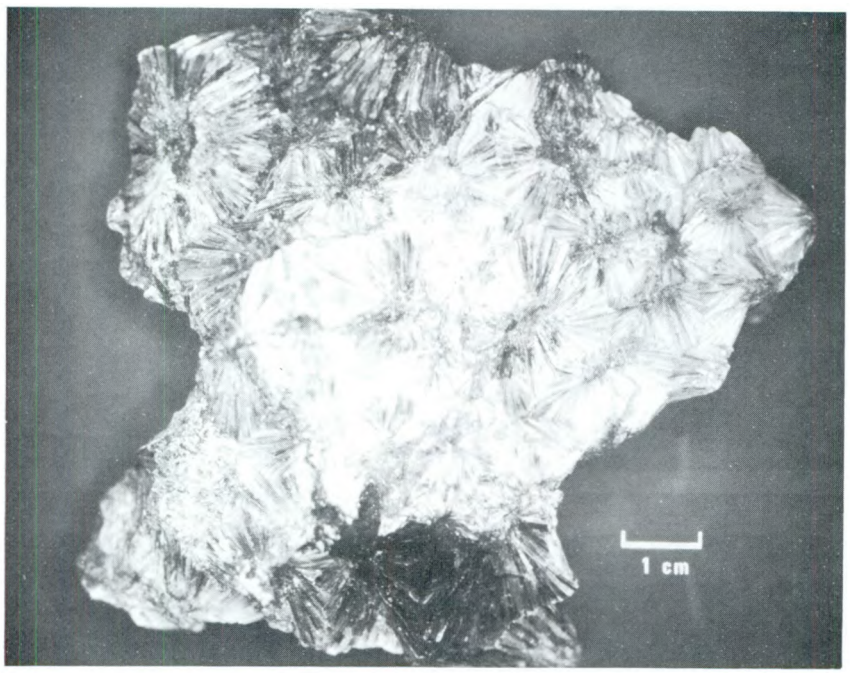


FIGURE 28. - Macrophotograph of radiating aggregates of acicular pyrophyllite (X 1).

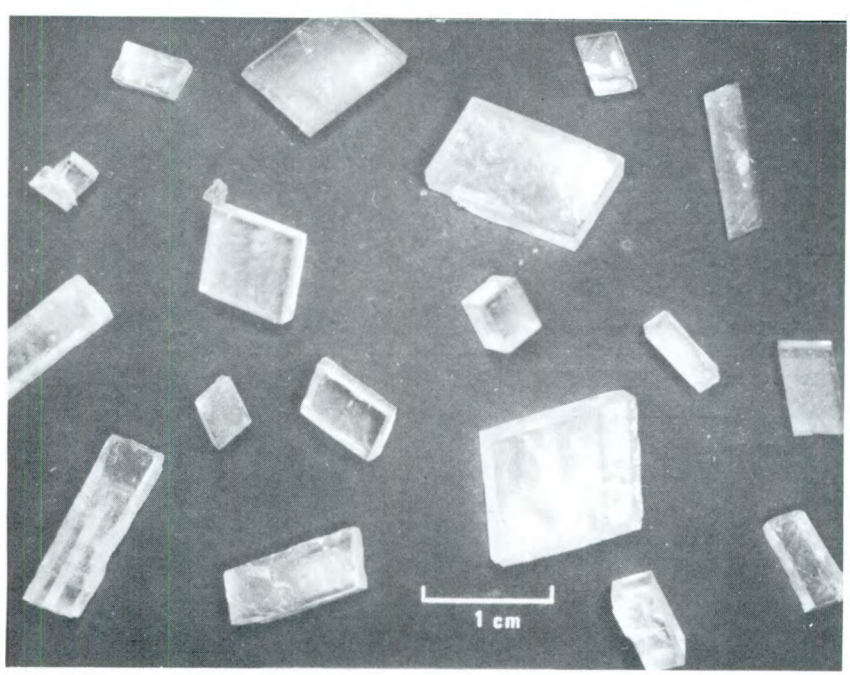


FIGURE 29. - Macrophotograph of calcite rhombohedral cleavage fragments (X 1.7).

Rhombohedral Cleavage.--A hexagonal crystal with three distinct cleavage planes that yield rhombohedral fragments (such as calcite).

Figure 29 shows the excellent rhombohedral cleavage of calcite.

Cubic Cleavage.--An isometric crystal with three distinct cleavage planes that yield cubic fragments (for example, halite).

Octrahedral Cleavage.--An isometric crystal with four distinct cleavage planes that yield octahedral cleavage fragments (for example, magnetite and fluorite).

Cleavage Quality

The quality or persistence of a cleavage is described as follows:

Perfect.--Difficult to break in any other direction; cleavage surfaces are extensive and smooth.

The cleavage of calcite in figure 29 is perfect.

Good.--Breaks readily along one direction, but can be broken in other directions; cleavage

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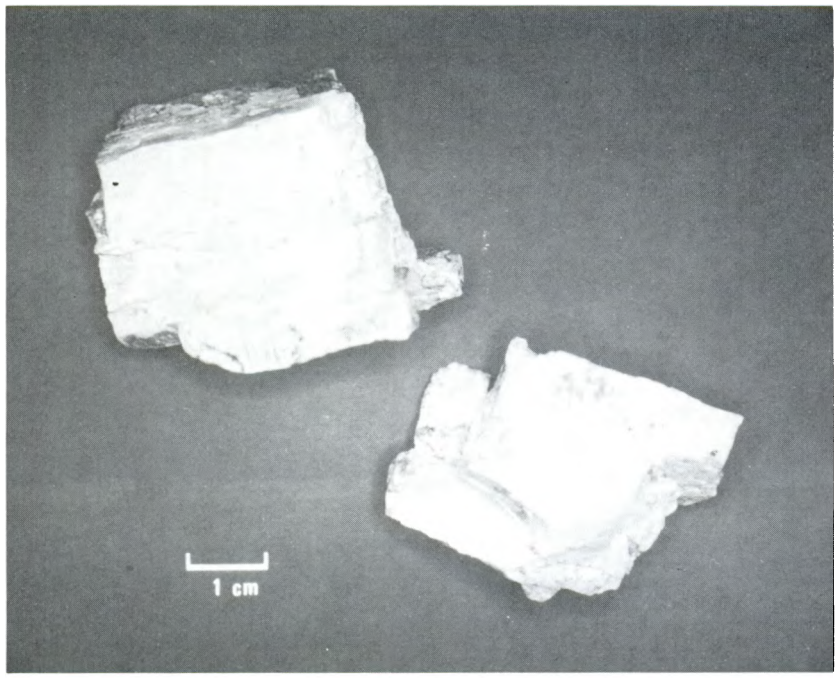


FIGURE 30. - Macrophotograph of pyroxene showing good cleavage interrupted by uneven fracture (X 1).



FIGURE 31. - Tremolite, showing good prismatic cleavage (SEM photomicrograph at X 560).

surfaces are smooth but interrupted by other fractures.

Figures 30 and 31 show good cleavage of pyroxene and tremolite.

Fair.--Breaks most readily along cleavage but also fractures easily in other directions; cleavage surfaces are seldom large.

Imperfect.--Breaks about as easily by fracture as by cleavage; cleavage surfaces tend to be small and frequently interrupted.

Cleavage Fragment

Cleavage Fragment.--A fragment produced by the breaking of crystals in directions that are related to the crystal structure and are always parallel to possible crystal faces.

Minerals with perfect cleavage can produce perfect regular fragments. Amphiboles with prismatic cleavage will produce prismatic fragments (fig. 31). These fragments can be elongated and on superficial observation may resemble fibers (figs. 26 and 32). However, because they did not grow as fibers, they cannot have the characteristics of fibers. Consequently,

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FIGURE 32. - Cleavage fragments of riebeckite (X 500). Particle F has a fibrous appearance.

cleavage fragments cannot be called fibers. Minerals do not always break into the same shapes as their growth habits. For example, calcite has many growth habits, but usually breaks into rhombohedral cleavage fragments (fig. 29).

Fracture.--The tendency of a crystal or grain to break in an irregular manner apparently unrelated to crystallographic directions.

Fracture types are as follows:

Even.--Breaking along relatively smooth planes.

Uneven.--Breaking along irregular planes.

Splintery.--Breaking into irregular, elongated fragments.

Conchoidal.--Breaking along spherical or conical surfaces.

Hackly.--Breaking with a jagged irregular surface.

Parting.--The tendency of a crystal or grain to break along crystallographic planes weakened by inclusions or structural defects. Different specimens of the same mineral may or may not exhibit parting. Twinned crystals often part along composition planes, which are lattice planes and, therefore, potential crystal faces. Parting is similar to cleavage.

MINERAL IDENTIFICATION AND CHARACTERIZATION

Until recently, emphasis in the United States was placed on occupational exposure of employees manufacturing or using asbestos products for insulation and other applications (29, 33). Regulatory procedures were adopted from those used in Great Britain. The industrial-hygiene identification procedures were acceptable to industry, health, and regulatory organizations because the concern was restricted to several mineral products known collectively as asbestos. Although light optical microscopic procedures counted only the larger particles collected on the air filters, the procedure was adequate for

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correlating health effects to the number of fibers observed. Exact definitions for asbestos-related mineralogical terms were not essential since all three groups (industry, health, and regulatory) clearly understood what was being counted and regulated.

The light optical microscopic procedures used by industrial hygienists were designed for control of asbestos-processing operations in which the chrysotile and asbestiform amphiboles are present as bundles of fibers as well as individual fibers (15). These bundles may have an average diameter of 0.75 to 1.5 μm for chrysotile and 1.5 to 4.0 μm for the amphibole asbestos (3). Particulates of these sizes can be readily observed at a magnification of X 450 to X 500. In contrast, samples from ambient air and personnel air monitors may consist of individual fibrils or small bundles of chrysotile 0.02 to 0.1 μm in diameter, and/or amphiboles 0.1 to 0.2 μm in diameter (3). Fibrils and small fibers in this size range are not visible using the conventional light optical microscopic procedures (13, 34). Therefore, the identification procedures currently used for regulating the U.S. mineral producing and consuming industries must be reexamined to insure that they are both mineralogically correct and applicable to the size range of the particles being regulated.

This discussion will be limited to the selected silicate minerals and their asbestiform varieties listed in table 1. The objective is to point out the particle size at which the minerals can be identified and characterized by various analytical techniques (17). Detailed descriptions of the various analytical and characterization techniques are available in numerous publications and textbooks and are therefore not included in this report.

A crystalline mineral is defined primarily by its crystal structure and by its definite composition or range of compositions. Therefore, any system of mineral identification should be based principally on crystal structure and chemical criteria. Additional characteristics have to be determined to distinguish varieties. These varieties have similar basic crystal structures and composition, but are usually differentiated macroscopically by the characteristic habits and/or other specific features of the varieties. The objective for this section of the report is to summarize the methodology for identifying the mineral first by mineral group (such as serpentine and amphibole), then by mineral (actinolite, anthophyllite, or chrysotile), and finally by mineral variety.

Macroscopic Samples

At the macroscopic level (easily visible by the unaided eye), the obvious feature of the asbestiform varieties is the presence of fibers that can be easily separated, while the nonasbestiform varieties have a massive, blocky, bladed, or columnar appearance (figs. 2-7). Although chrysotile does occur very rarely in a nonasbestiform habit, in general the distinction between chrysotile and serpentine can be based on the presence or absence of separable fibers. In some serpentine samples where an obvious asbestos texture is not displayed, the distinction between serpentine varieties may require more specialized techniques (6, 19). The distinction between serpentine and

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amphibole minerals at the macroscopic level can be made by optical microscopy, elemental analysis, differential thermal analysis, and X-ray diffraction techniques. For essentially pure samples, these techniques should also be sufficient to identify the individual amphibole minerals based on the elemental composition corresponding to the various members of the solid solution series.

Many macroscopic samples of interest to the occupational and environmental health personnel may contain low percentages of asbestiform minerals (for example, chrysotile in serpentine and tremolite asbestos in talc). As a supplement to optical microscopy, the presence or absence of serpentine or amphibole minerals can be determined in 10- to 100-mg samples by instrumental techniques such as X-ray diffraction, differential thermal analysis, or infrared spectrophotometry. In general, the sensitivity of these instrumental methods is approximately 1.0 weight-percent. Sensitivity is significantly affected by the presence of other minerals that give a response at or near the response peak of the serpentine and amphibole minerals. It is important to note that these methods usually only distinguish between mineral groups; light optical or electron optical microscopy is required to obtain morphological characteristics necessary to identify varieties of the same material.

Chemical characterization is generally necessary to assign a specific mineral name to an amphibole whose structure is known. The amphiboles have been described (8) using the structural formula $W_{0-1}X_2Y_5Z_8O_{22}(OH,O,F)_2$. Generally, W = Na, K; X = Na, Ca, Mg, Fe^{+2}, Mn; Y = Al, Fe^{+3}, Ti; and Z = Si, Al. In addition to the variation implied by the structural formula, a chemical analysis must take into account inclusions of other minerals that may be present. In contrast to the more formidable task of amphibole chemical characterization, the serpentine minerals generally show little deviation from the formula $Mg_3Si_2O_5(OH)_4$. For either structural or chemical characterization of a macroscopic sample, sufficient time must be spent in sample preparation to insure that relatively pure minerals are being examined.

Microscopic Samples

The petrographic microscope provides a general method by which particles larger than 5 μm can be characterized. By observing the optical properties characteristic of the structure and chemistry of a mineral, an experienced microscopist can distinguish amphiboles from serpentines and, in some cases, distinguish individual minerals within these groups (7). The refractive indices are sufficiently different for the serpentine and amphibole groups to make a distinction between groups by using the appropriate index oil (table 2). There is significant overlap in the range of the three refractive indices among the amphiboles, but a specific index (for example, α, β, or γ) can be determined to aid in identifying the amphibole species. Optical relationships can be confused, however, if the particle consists of fiber bundles or is some other form of crystalline aggregate. The well-known parallel extinction of the commercial asbestos known as Amosite can be used to distinguish that variety from the nonasbestiform varieties of cummingtonite and actinolite. A method of using extinction angles and cleavage directions to distinguish specific asbestiform and nonasbestiform amphiboles has been described (37); however, this technique is limited to particles with diameters greater than about

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5 μm and cannot be universally applied to all amphiboles. There are many other optical parameters such as pleochroism, sign of the elongation, and color that are easy to obtain. Other parameters such as optic axial angle, optical orientation, and optic sign are relatively more difficult to obtain.

TABLE 2. - Refractive indices for the serpentine group and selected amphibole minerals

	Refractive index	Range of values
Chrysotile.....	α	1.493-1.560
	β	1.504-1.550
	γ	1.517-1.562
Antigorite-lizardite.....	α	1.538-1.564
	γ	1.546-1.573
Anthophyllite.....	α	1.596-1.652
	β	1.605-1.662
	γ	1.615-1.676
Actinolite-tremolite.....	α	1.599-1.668
	β	1.612-1.680
	γ	1.622-1.688
Cummingtonite-grunerite.....	α	1.635-1.696
	β	1.644-1.709
	γ	1.655-1.729
Riebeckite.....	α	1.654-1.701
	β	1.662-1.711
	γ	1.668-1.717

Except for the asbestiform variety, serpentines are usually massive, while amphiboles range from fine-grained massive to columnar or radiating aggregates of prismatic or acicular crystals. Amphiboles in acicular habit may appear to grade into the asbestiform varieties. The characteristic features of this habit may still be seen by electron microscopy. Terms such as "acicular" or "prismatic" may still be applied when seen, but the term "asbestiform" begins to lose its usefulness. For example, how may flexibility be demonstrated in a 2-μm bundle of fibers? As particle size decreases, the inability to manipulate the mineral grains restricts the use of the term "asbestiform" without altering the original sense of the word. High magnification necessitates the use of strictly dimensional terms such as size and aspect ratios to accurately describe the morphology of the amphiboles and serpentines. The degree of morphologic characterization possibly will depend on the magnification being used. An asbestos particle being described as a single fiber at low magnification may be seen to be a bundle of fibers at some high magnification. Therefore, the magnification must be stated in the description. Morphologic characterization using light microscopy can be accomplished on particles as small as a few micrometers. Electron optics can

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be used to characterize a wide range of sizes extending down to a few angstroms. Morphologic characterization alone will not identify a mineral without supplemental structural or chemical data.

Structural information on individual particulates can be obtained by use of a transmission electron microscope (TEM) in the selective area electron diffraction mode (SAED). The inclination of the single crystal fragments to the electron beam is very critical since a slight tilt of the crystal may change a relatively simple reciprocal lattice pattern into a very complex one. Consequently, a special goniometer or tilting stage is necessary to obtain easily interpretable diffraction patterns. For the identification of the mineral, a goniometer or tilting stage is even more essential since dependable conclusions cannot be made from measurements on one reciprocal lattice plane. The quality of the SAED pattern is a function of fiber diameter (fig. 33). The larger diameter fibers (>0.5 μm) strongly absorb the 60- to 100-kev electrons used in a conventional TEM, while the very small-diameter fibers (<0.2 μm) do not give sufficient electron-diffraction intensity. A second problem with small-diameter fibers is the degradation of the single-crystal pattern by diffraction lines from nearby particles. A higher energy TEM, with the resultant greater penetration of the electron beam, can be utilized for large-diameter particles. However, these costly instruments are not widely available.

Although the magnitude of the characteristic C, the distance between the conspicuous layer lines for chrysotile and the amphiboles, is similar in direct space ($d_{001} \sim 5.3A$), the chrysotile pattern has very prominent streaks on these layer lines compared with the spot pattern for the amphiboles (27). Researchers indicate the ability to distinguish between the fibrous and nonfibrous variety of amphiboles by SAED is still to be resolved.

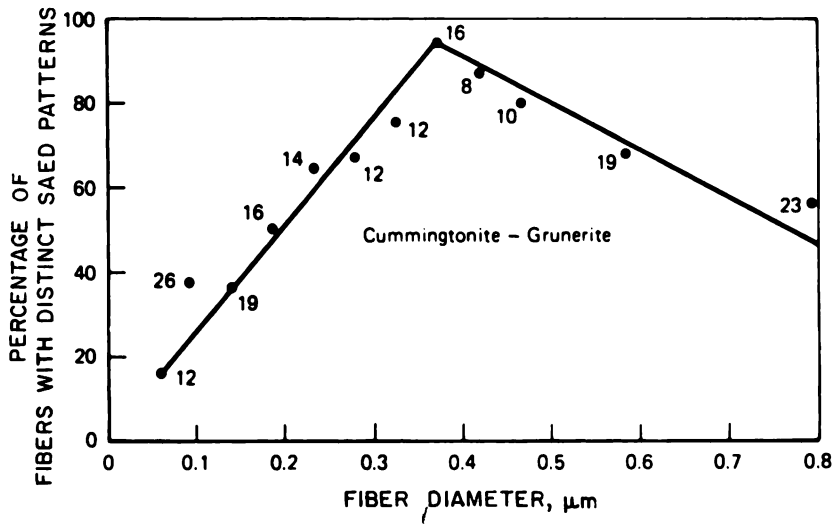


FIGURE 33. - Quality of SAED pattern as a function of amphibole fiber diameter. The values indicated on the graph are the number of fibers examined at each diameter. (Reprinted with permission of D. R. Beaman and D. M. File from Analytical Chemistry (2). Copyright by the American Chemical Society.)

At the very high magnification available with a TEM, chrysotile's hollow-tube (scroll-like) structure, approximately 50 A in diameter, is visible (fig. 16). This hollow-tube structure, together with chemical and structural data regarding the sample, is sufficient to identify the mineral variety. However, the hollow-tube structure is only visible for individual fibrils; fibers (composed of

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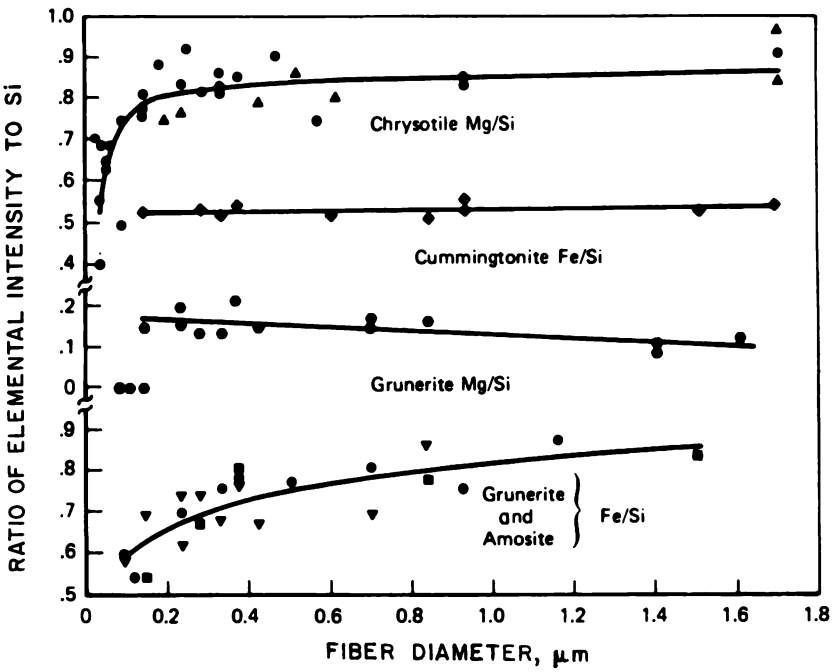


FIGURE 34. - Intensity ratio of FeKα, MgKα, or CaKα relative to SiKα as a function of fiber diameter. (Reprinted with permission of D. R. Beaman and D. M. File from Analytical Chemistry (2). Copyright by the American Chemical Society.)

several fibrils) will not display this characteristic because of stacking of the fibrils.

The elemental composition of microscopic grains is determined by either wavelength or energy-dispersive X-ray spectrography in conjunction with scanning or transmission electron microscopy. Extreme care must be taken in the calculation of elemental concentrations from X-ray spectral intensities because the spectral line intensities (FeKα, MgKα, CaKα, relative to SiKα) are dependent on particle diameter for small fibers (2). The particle diameter becomes a significant variable in the spectral intensity-composition relationship below 0.2 μm (fig. 34). Carbon contamination from diffusion pump oils must also be considered when analyzing small particles because the longer measuring times required to count sufficient numbers of X-ray photons allows time to build up a contamination layer. This carbon layer preferentially absorbs the lower energy X-ray photons.

Energy-dispersive X-ray spectral calibration data for each scanning or transmission electron microscope must be made using relatively pure standard minerals analyzed by accepted chemical-instrumental techniques. The analyst should be aware that other nearby grains may be contributing to the characteristic X-ray lines because of either penetration of the electron beam through the particles or secondary excitation of nearby particles from primary X-rays generated in the particle being measured. Modern electron optical instruments have electron beams diameters of approximately 0.1 μm; however, the sphere of excitation can be several micrometers in diameter as a result of scattered electrons and primary X-rays generated in this particle (26). Conversion of intensity into concentration using accepted computer programs such as "MAGIC" is limited in accuracy because these programs are designed for use with grains or particles several micrometers in diameter or larger, whereas the average mineral fiber diameter is less than 0.5 μm for amphiboles and less than 0.1 μm for chrysotile. A good example is the diameter size distribution of

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chrysotile fibers in ambient air samples (table 3). The important point to note is that approximately 95 percent of these chrysotile fibers are 0.12 μm or less in diameter. Therefore, quantitative correction procedures applicable to large particles will be of limited value in mineral-fiber identification because the relative X-ray spectral intensities are dependent on fiber diameter below 0.2 μm.

TABLE 3. - Frequency distribution of the width of chrysotile fibers in ambient-air samples,[1] percent

Diameter of chrysotile fibers, μm	Sample					
	1	2	3	4	5	6
0.02-<0.04	10	70	57	17	15	17
.04- <.06	47	24	28	29	33	49
.06- <.08	24	5	8	28	20	15
.08- <.10	14	1	2	12	26	6
.10- <.12	2	0	1	7	3	6
.12- <.14	0	0	2	3	1	1
.14- <.16	1	0	1	2	1	1
.16- <.18	0	0	0	1	0	1
.18- <.20	0	0	0	0	1	1
.20- <.22	1	0	0	0	0	1
.22- .24	0	0	1	0	0	1
>.24	1	0	0	1	0	1

[1]Samples were collected 1-2 miles from a serpentine rock quarry.

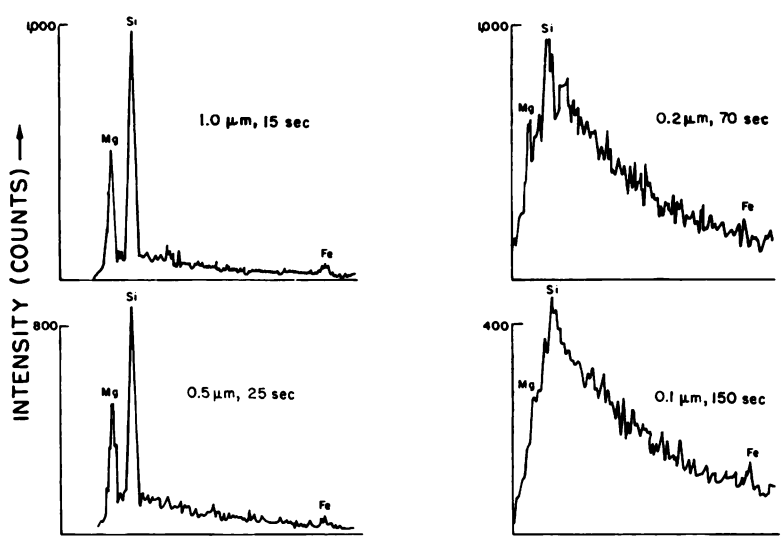


FIGURE 35. - Energy-dispersive X-ray spectra of chrysotile as a function of fiber diameter, BeO substrate (21).

Another problem with the elemental characterization of very small particles is the poor signal-to-background ratio. Longer counting times will help to improve the reliability of the measurement, but the best approach is to minimize the continuum background resulting from the interaction of the electron beam and the same substrate. Figures 35 and 36 show the energy-dispersive X-ray spectra from chrysotile fibers mounted on beryllium oxide (BeO) and beryllium (Be) substrates, respectively. The lower effective atomic number of Be compared with that of BeO results in a reduced continuum, therefore

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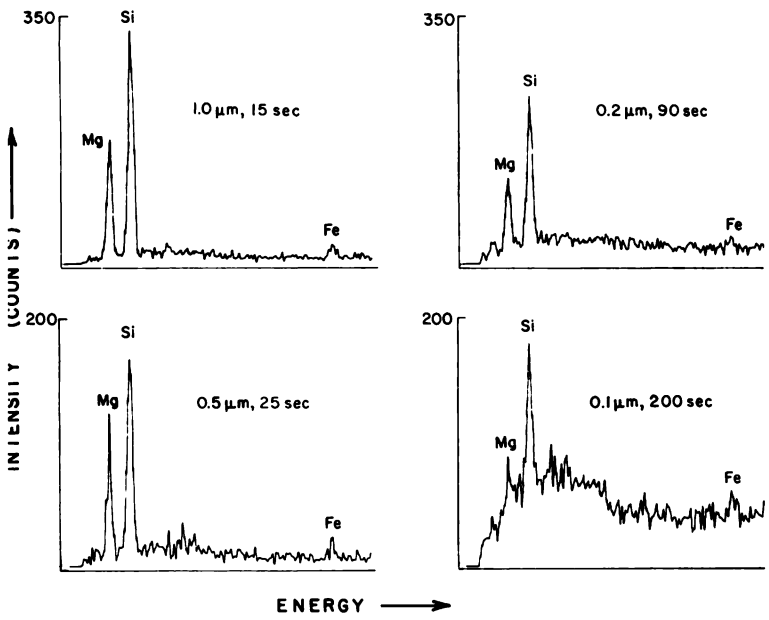


FIGURE 36. - Energy-dispersive X-ray spectra of chrysotile as a function of fiber diameter, Be substrate (21).

giving an improved signal-to-background ratio.

APPLYING MINERAL TERMINOLOGY TO THE IDENTIFICATION AND CHARACTERIZATION OF PARTICULATES

This section addresses the practical considerations and limitations encountered when applying nomenclature and identification-characterization procedures to regulatory and environmental samples.

Applying Morphological Terminology

One of the obvious features of minerals and their particulates is their morphology or shape. The need for precise definitions of terms such as "asbestiform," "fiber," "cleavage fragment," and "fibril" was explained earlier. These definitions were carefully structured to eliminate ambiguity and to be technically correct. Applying the definitions to samples requires careful thought as to what limits must be placed on interpretations resulting from the use of these terms and other mineralogical concepts. The underlying problem, recognized by both medical and regulatory personnel, is classifying the mineral particle as the asbestiform or nonasbestiform variety. The classification should withstand the test of mineralogical logic and proof. In a mineralogical sense, the source of the mineral particulates must be considered, as explained in the following discussion.

Particulates From a Known Asbestiform Serpentine or Amphibole Source

The definition of asbestiform minerals includes three aspects: morphology, structure, and chemistry. Morphologically, asbestiform mineral varieties separate into flexible fibers or flexible bundles of fibers. Flexible fibers bend readily and only break across the fibers into distinct pieces with some difficulty. Structurally, the asbestiform minerals are limited, in this report and in common practice, to the serpentine and amphibole mineral groups. Chemically, these minerals are all hydroxylated silicates; the term "hydroxylated" is preferred over "hydrated" because these minerals contain OH ions rather than water of crystallization. The serpentines contain approximately 13 weight-percent water; the amphiboles, approximately 2.5 weight-percent water.

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For the purpose of this discussion, assume that a hand specimen meeting these requirements is correctly identified as an asbestiform mineral. If this sample is crushed and its fragments examined at various magnifications, its fibrous nature would be apparent, as in figures 14 and 17 to 19. These elongated fragments would be termed "fibers" and "bundles of fibers," and with the other available information would be called asbestiform. As these asbestiform particles are examined at increasing magnification, smaller particles become visible, while the image of large fibers and fiber bundles may exceed the field of the microscope. At increasingly smaller sizes, while fibers or bundles of fibers are still the predominant shape, a few of the fibers are observed to have broken into shorter and shorter segments. (Several short fiber segments are visible in figures 14 and 16.) These very short fiber segments are no longer described as fibers, but would be classified as fragments of fibers, or cleavage fragments if one or more cleavage planes govern their shape. Therefore, a known asbestiform sample would show an increase in the ratio of fiber fragments to fibers with a decrease in particle size.

Particulates From a Known Nonasbestiform Serpentine or Amphibole Source

If the hand specimen discussed previously does not separate into flexible fibers or bundles of fibers, the mineral would not be considered asbestiform. However, the specimen would be classified as serpentine or amphibole if the specific mineral is identified on the basis of optical properties, chemistry, and structure.

If crushed fragments of this known nonasbestiform mineral are examined at various magnifications, the particles would be primarily cleavage fragments, or irregularly broken fragments if cleavage does not govern breakage. However, a few elongated particles may resemble a fiber in appearance to the degree that they may be indistinguishable morphologically from fibers derived from an asbestiform mineral sample. Figures 26 and 32 for tremolite and riebeckite, respectively, show cleavage fragments with fibrous shapes that could be incorrectly identified as fibers.

What can be stated morphologically about particles derived from crushing a known nonasbestiform mineral is that most of the particles are cleavage fragments with nonasbestiform texture; a few are fibrous in appearance, particularly at low magnification; and all of the particles are known to be derived from a nonasbestiform source.

Comparison of Particulates From Known Serpentine and Amphibole Minerals and Their Asbestiform Varieties

The appearance of particles generated by milling known serpentine and amphibole minerals and their asbestiform varieties is shown in figures 37 to 40. The samples shown in figures 37 to 39 were photographed using light optical microscopy at three magnifications to show that, at decreasing size (depicted by increasing magnification), the original habit generally persists. For the nonasbestiform amphibole minerals, there were a few elongated particles from the riebeckite and tremolite. Elongated particles of this type

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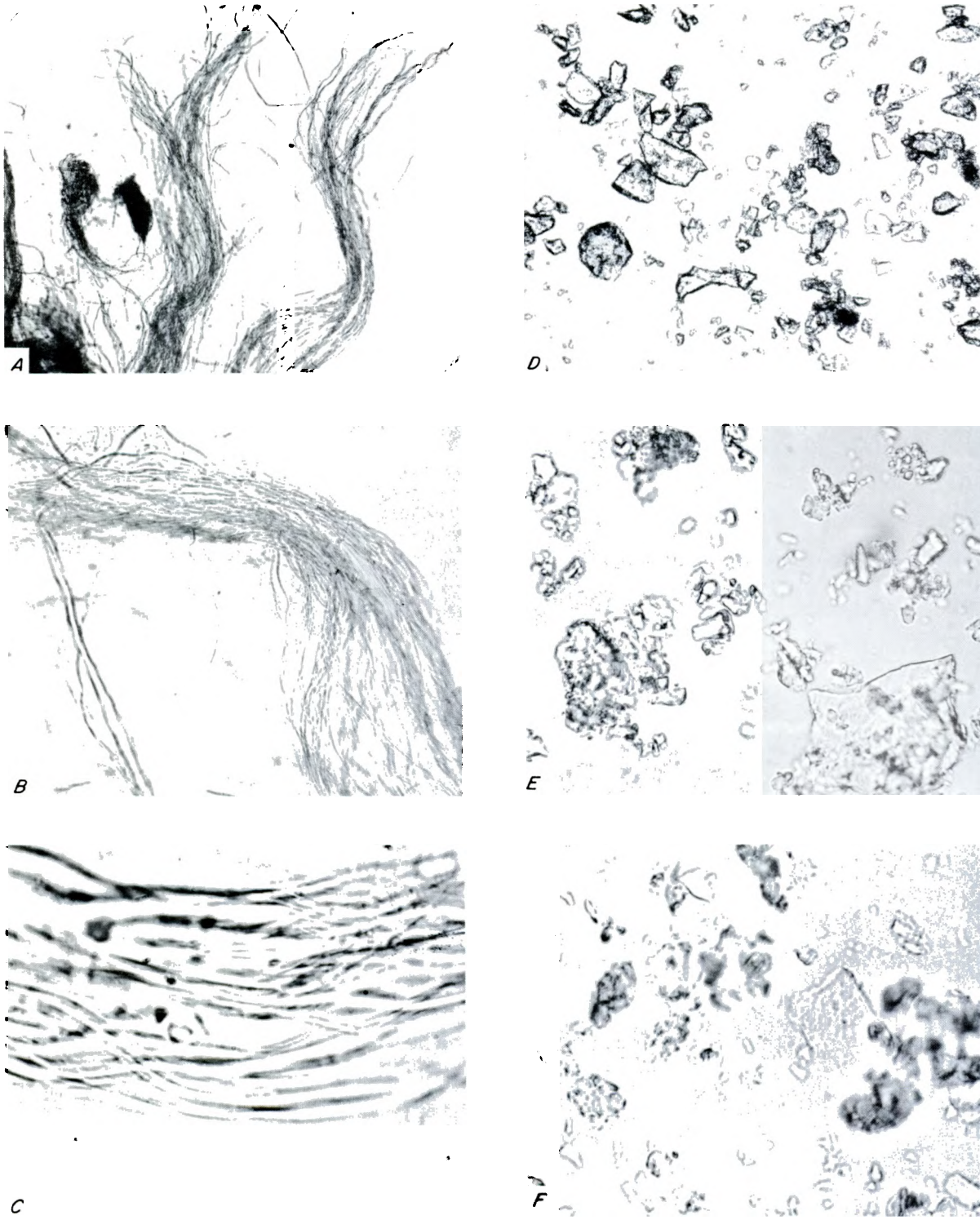


FIGURE 37. - Light optical photomicrographs of chrysotile and antigorite-lizardite at three magnifications. Chrysotile (left) at *A*, X 100; *B*, X 500; and *C*, X 950. Antigorite-lizardite (right) at *D*, X 100; *E*, X 500; and *F*, X 950.

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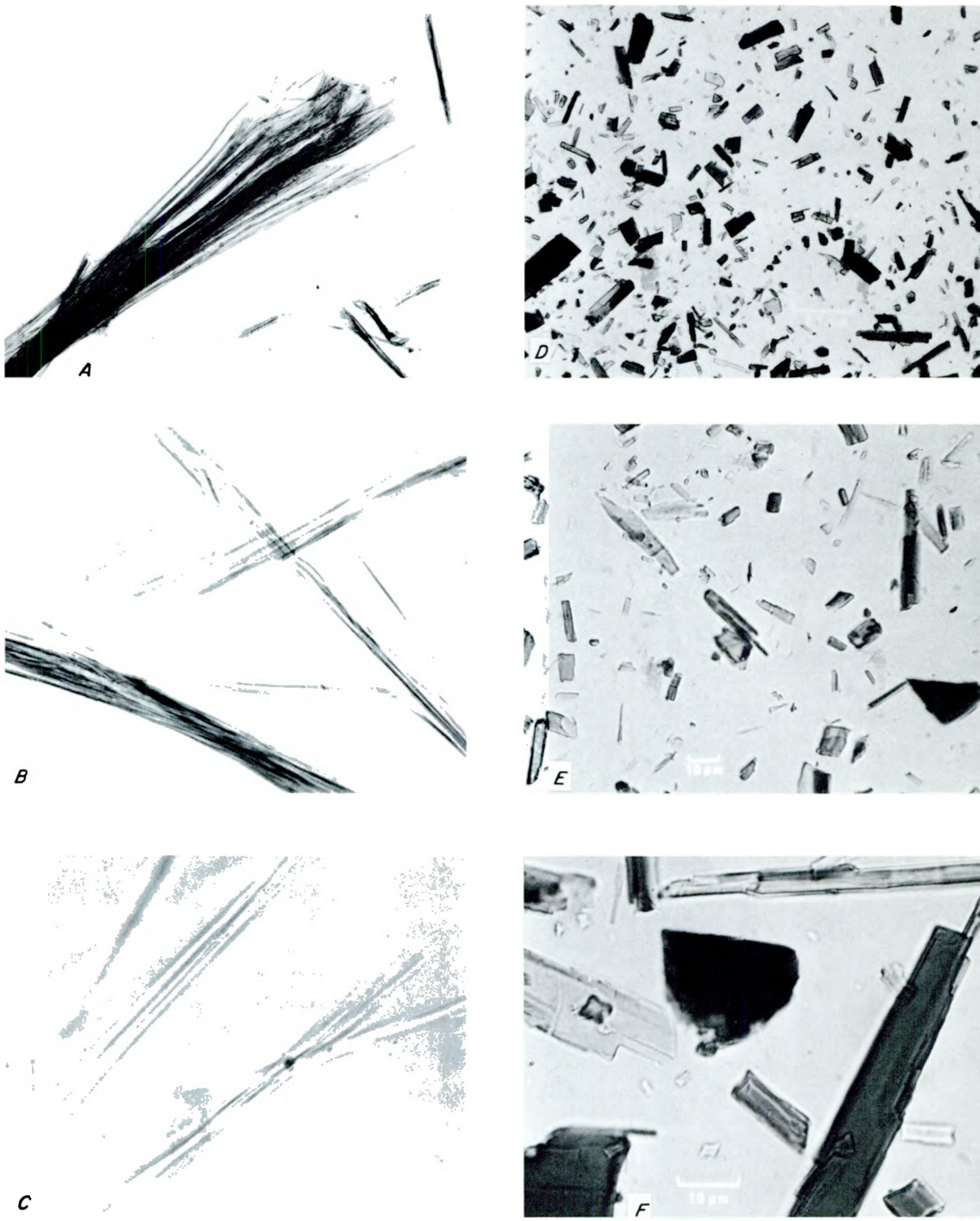


FIGURE 38. - Light optical photomicrographs of crocidolite and riebeckite at three magnifications; Crocidolite (left) at *A*, X 100; *B*, X 500; and *C*, X 950. Riebeckite (right) at *D*, X 100; *E*, X 500; and *F*, X 950.

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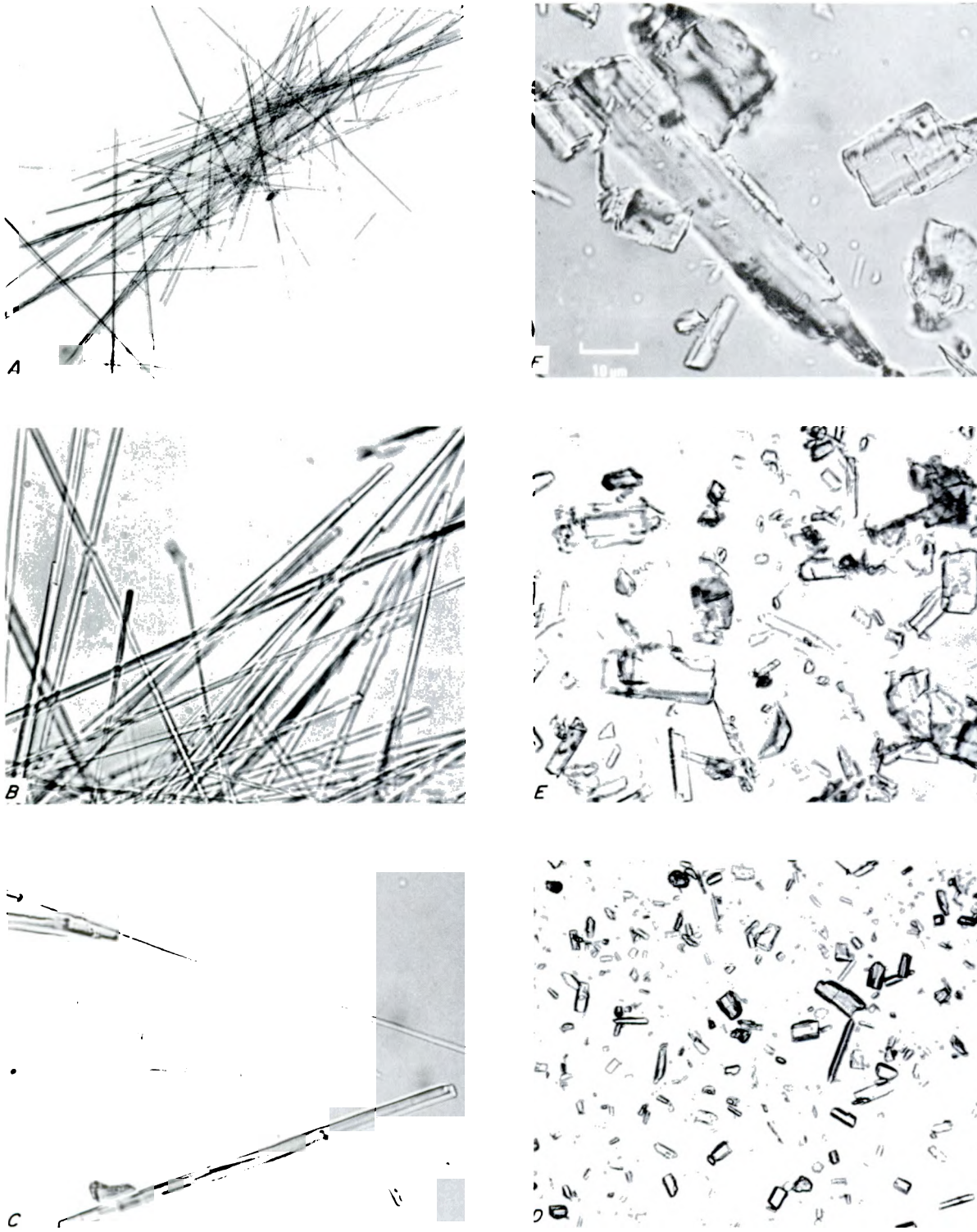


FIGURE 39. - Light optical photomicrographs of tremolite asbestos and tremolite at three magnifications. Tremolite asbestos (left) at *A*, X 100; *B*, X 500; and *C*, X 950. Tremolite (right) at *D*, X 100; *E*, X 500; and *F*, X 950.

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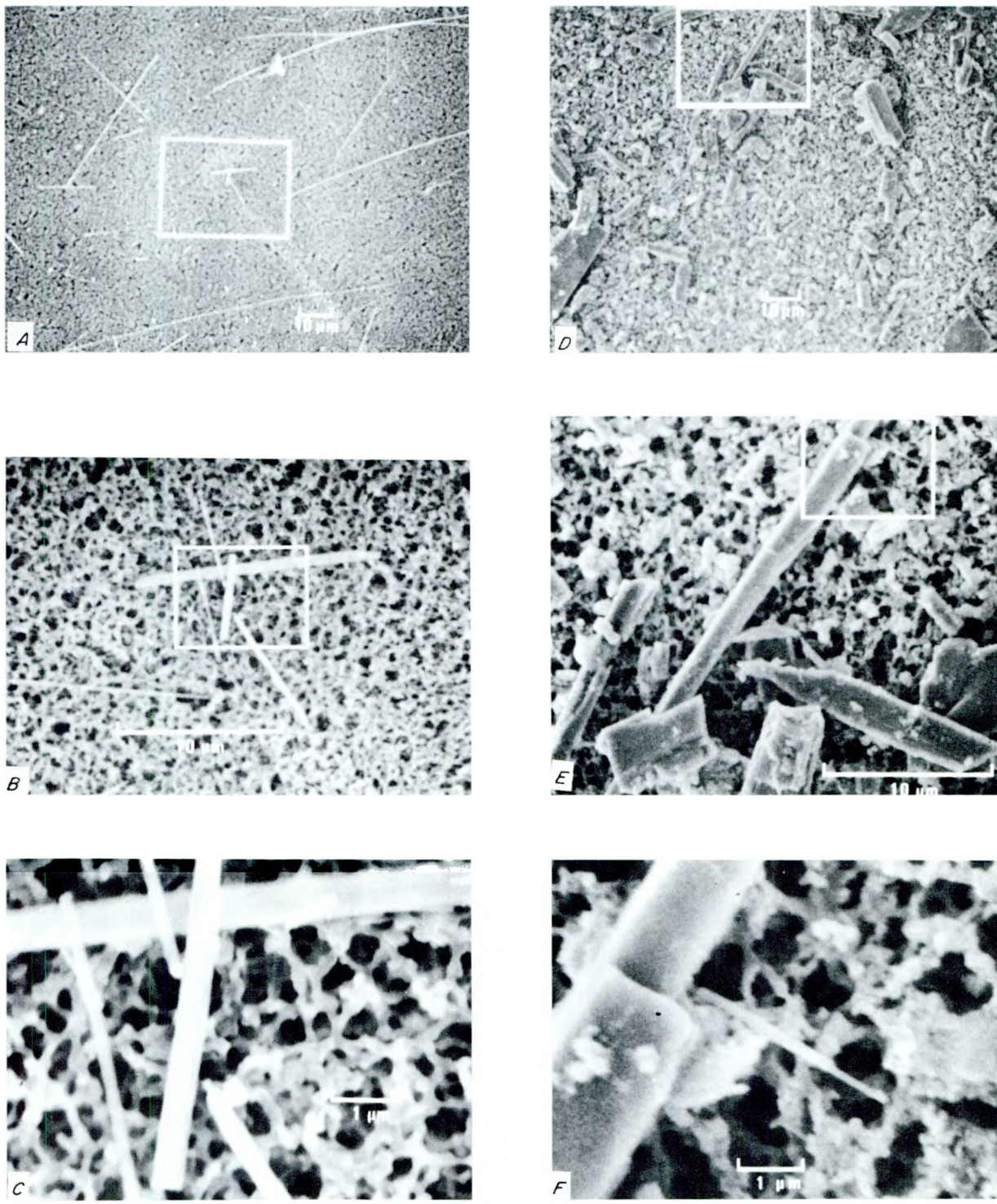


FIGURE 40. - SEM photomicrographs of crocidolite and riebeckite at three magnifications: Crocidolite (left) at *A*, X 500; *B*, X 2,500; and *C*, X 10,000. Riebeckite (right) at *D*, X 500; *E*, X 2,500; and *F*, X 10,000. Rectangles indicate the area shown at the next higher magnification.

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are typical of the prismatic cleavage of amphiboles. To increase optical contrast, the serpentine group samples were dispersed in an immersion oil considerably below the refractive indices for the serpentine.

Riebeckite and crocidolite particles are compared at higher magnifications in figure 40. The outlined areas in the scanning electron micrographs indicate the area displayed at the next higher magnification. Again, note the presence of a few elongated cleavage fragments of riebeckite visible at the higher magnification. In contrast, the aspect ratio of the crocidolite will decrease with decreasing particle size because the individual fibers cannot cleave further along the fiber axis; they can only break into shorter segments.

Aspect Ratio

Existing regulatory standards are based on counting specific mineral particulates with aspect ratios of 3 to 1 or greater. This report emphasizes that the aspect ratio has little mineralogical significance for individual particulates but is applicable to a large number of particles. A few relatively long thin particles are produced as cleavage fragments from the crushing and grinding of many nonasbestiform minerals. Conversely, similar milling treatment will result in a few short segments of true fibers from the asbestiform varieties. However, statistically, the length-to-width characteristics of the milled amphiboles and serpentine and their asbestiform varieties are significantly distinct, as shown by the data in figures 41-44.

Figures 41, 42, and 43 show the frequency polygons of the aspect ratio distribution for milled samples of the normal nonasbestiform variety of three amphiboles--anthophyllite, tremolite, and hornblende, respectively. Note that in all three examples, approximately 70 percent of the particles have an

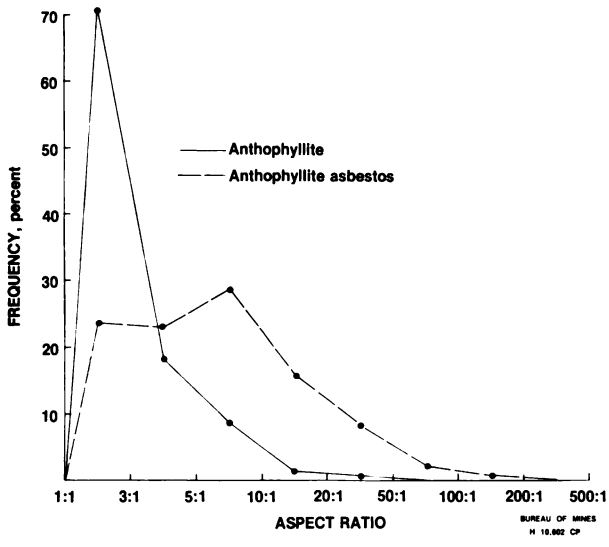


FIGURE 41. - Frequency polygons for the aspect ratios of anthophyllite and anthophyllite asbestos.

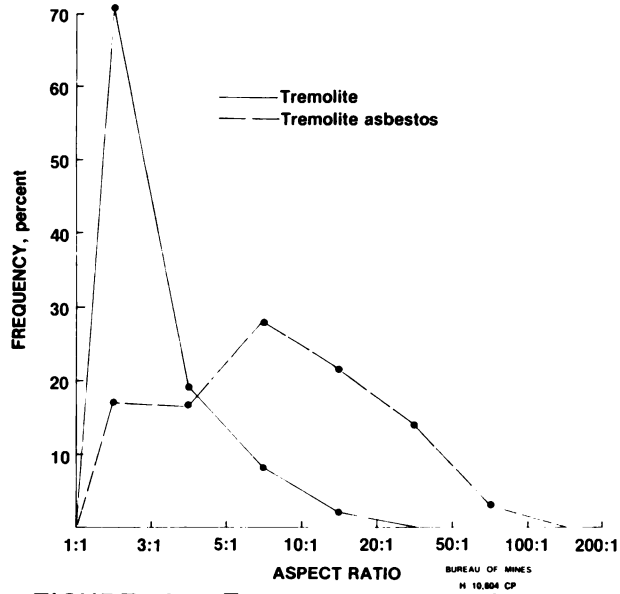


FIGURE 42. - Frequency polygons for the aspect ratios of tremolite and tremolite asbestos.

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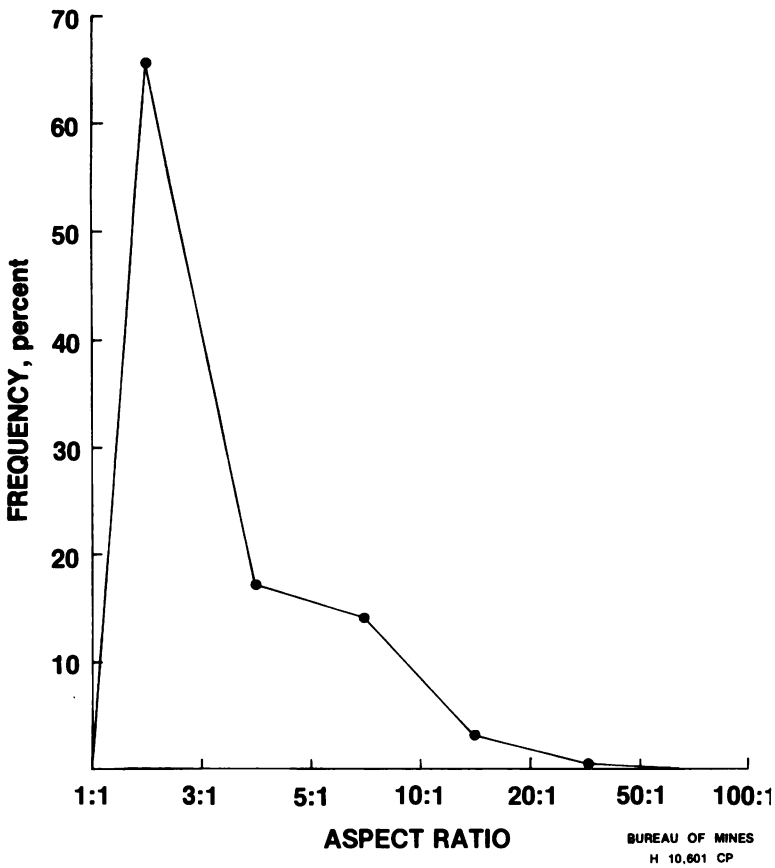


FIGURE 43. - Frequency polygons for the aspect ratio of hornblende.

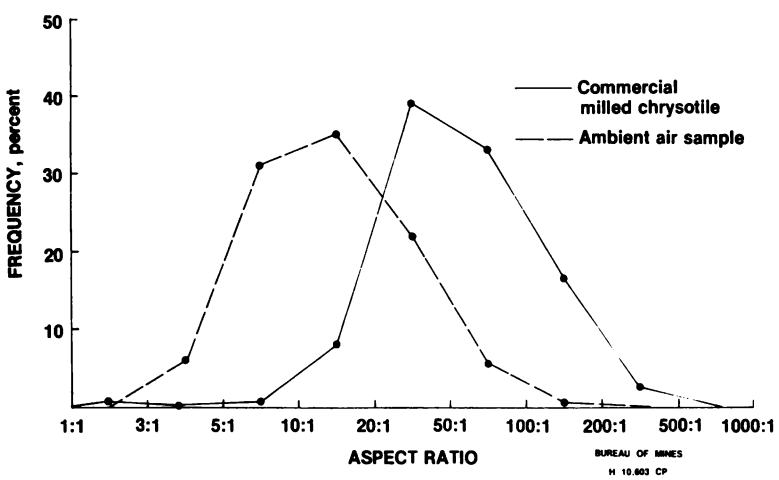


FIGURE 44. - Frequency polygons for the aspect ratios of commercial-grade chrysotile and chrysotile in ambient air.

aspect ratio of less than 3 to 1, and 95 percent of the particles have a length-to-width ratio of less than 10 to 1. The frequency distribution maximums of the aspect ratios for milled anthophyllite asbestos and tremolite asbestos are significantly higher than those for the normal, nonasbestiform variety. Thirty to forty percent of the asbestiform particulates are in the 10-to-1-or-longer class, with a significant number of particles having an aspect ratio greater than 20 to 1.

Figure 44 shows the distribution frequencies for a milled commercial grade of chrysotile asbestos and for chrysotile particulates collected on ambient air filters in the vicinity of a serpentine rock quarry. For the commercial-grade chrysotile, over 50 percent of the particles have an aspect ratio greater than 50 to 1, whereas the frequency distribution for the ambient air sample has a maximum between 10 to 1 and 20 to 1. These results are anticipated because the higher aspect ratios for the commercial-grade chrysotile are characteristic of the significantly longer starting material.

All of the aforementioned samples except the ambient air were milled, then dispersed in water for collection on a suitable substrate. The samples were then measured using electron microscopy at magnifications

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of 5,000 to 10,000. The ambient air sample, collected near a serpentine rock quarry, was measured using a TEM with magnifications of X 5,000 to X 32,000.

Based on these data, one test for distinguishing the presence or absence of the asbestiform variety of a mineral could be an examination of the frequency distribution of the aspect ratio for that mineral. Assuming positive identification of the mineral type, then the designation of variety would be based both on particle morphology and the frequency maximum of the aspect ratio. Cleavage fragments will generally have a frequency maximum less than 3 to 1, whereas the asbestiform varieties will fall between 10 to 1 and 20 to 1 or higher, depending on the characteristics of the mineral and the history of the sample, particularly the type and degree of milling. If any shape or size limits are placed on characterizing mineral particulates, such limits should be based on medical evidence or on some limitation of the characterizing technique and so stated.

Particulates From Unknown Sources

Samples such as environmental airborne or waterborne mineral particulates collected at a considerable distance from a possible source are examples of particulates from an unknown source. The samples could have been collected at a location so distant from a known source that other mineral particulates originating from other sources compose most of the sample.

The source of the particulates in an environmental sample may be located by taking additional samples at selected intervals in the direction of, and closer to, the suspected source. However, several factors must be considered: The direction of air and water currents with respect to the suspected source, and the proximity to and direction of other sources with regard to the suspected source. One study found very low concentration of airborne chrysotile upwind from a source compared with a concentration two orders of magnitude greater downwind (14). Another important consideration is the level of natural or human disturbances of particulates; for example, strong versus weak winds, or heavy versus light vehicle traffic. In some instances, it may be possible to identify the source if the mineral particulates of interest have unique trace elements or combinations of elements that are specific to the probable mining or milling operation emitting the particulates. Detailed elemental analysis using the X-ray spectral capabilities of an SEM or TEM is required on both the suspected source and the particulates.

APPLICATIONS

The following examples illustrate the application of mineral terminology and identification-characterization procedures to three types of problems: (1) chrysotile determination in ambient-air samples collected near a serpentine rock quarry, (2) identification of asbestiform minerals in ceilings and walls of public buildings, and (3) characterization of a mineral product. These examples illustrate, in order, the need for higher magnification than available with the light optical microscope, the use of various characterization techniques to screen and identify asbestiform minerals, and the judgment of the analyst in distinguishing cleavage fragments and asbestiform particles.

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FIGURE 45. - Macrophotograph showing chrysotile veins in serpentine rock (X 1).

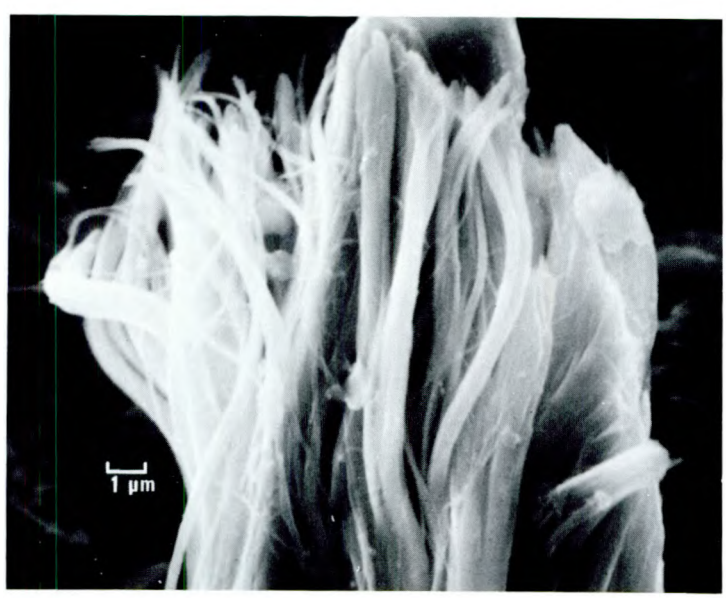


FIGURE 46. - Chrysotile bundle (SEM photomicrograph at X 5,000).

Ambient-Air Samples Collected Near Serpentinite Rock Quarry

The Bureau of Mines is working with State and Federal officials to measure mineral particulates in ambient-air samples collected in the vicinity of a serpentine rock quarry. Optical microscopic procedures at about X 500 are limited to the identification of mineral particulates longer than 5 µm with an aspect ratio of 3 to 1 or larger (criteria set by the Mining Enforcement and Safety Administration and the Occupational Safety and Health Administration). The mineralogist can further identify the particles as belonging to the serpentine, amphibole, or other mineral group with index oils (table 2).

The serpentine rock in the quarry is interlaced with small veins of chrysotile (fig. 45). Optical microscopic procedures used for industrial hygiene are adequate for the detection of large chrysotile fiber bundles. These fiber bundles of commercial-grade chrysotile can be several micrometers or larger in diameter (fig. 46). In contrast, the mining and crushing operations in the quarry plus transport of particulates over a distance breaks bundles of fibers down to fibers or fibrils with diameters of 250 to 1,000 A (table 3).

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Figure 47 is a series of SEM photomicrographs of a mixture of chrysotile and nonasbestiform serpentine handpicked from a small vein in the serpentine rock quarry. Note that at X 450 (corresponding to the optical microscope magnification), only one or two bundles of chrysotile are faintly visible; the predominant particles are the nonasbestiform serpentine. As the magnification is increased, the high concentration of chrysotile fibers becomes readily visible. The fiber diameter size data in table 3 indicate that more than 95 percent of the chrysotile fibers in these ambient air samples are below the limit of resolution of the optical microscope. Although many other scientists have pointed out the limitation of the optical procedures for chrysotile in ambient air, there is need for continuous emphasis that higher magnification techniques are necessary for environmental and regulatory samples.

Asbestos in Ceiling and Wall Materials

A possible environmental hazard is the release of asbestos from ceilings and walls in homes, churches, schools, and various other public and commercial buildings. Because of the very high number of potential samples to be examined by various State or Federal agencies, a rapid and reliable screening procedure is necessary to identify those samples that warrant further tests. Three complementary analytical methods for screening, identification, and semiquantitative estimate of the asbestiform mineral concentration are X-ray diffractometry, differential thermal analysis, and microscopy (light optical and scanning electron).

The screening identification procedures can be relatively simple because chrysotile is the principal asbestos mineral used for building insulation materials, with Amosite used to a much lesser extent. In 18 samples from a midwestern municipal health department, chrysotile was a major constituent (>50 weight-percent) in 2 samples, a minor constituent (1 to 10 weight-percent) in 12 samples, and not detected in 4 samples. Other minerals present in various concentrations in these samples were calcite, quartz, gypsum, and mica. Amosite was found as a major constituent in the ceiling of an older building located on a university campus.

The presence of either serpentine or amphibole minerals in the insulation materials can be used as a probable indication of asbestos. Therefore, screening tests are based on the presence or absence of characteristic differential thermal analysis or X-ray diffraction peaks of either serpentine or amphibole minerals. For the positive samples, confirmation of the presence of the asbestiform variety requires some type of microscopic examination because the thermal and X-ray diffraction methods do not identify the mineral variety.

Differential thermal analysis provides a detectable signal from chrysotile at 0.5 to 1.0 weight-percent, as indicated by the curves shown in figure 48. Note the increase in the endothermic (A) and exothermic peaks (B) upon addition of about 5 weight-percent chrysotile to a sample taken from a school ceiling. The sensitivity of differential thermal analysis for the amphibole minerals is significantly poorer because the H_2O content of amphiboles is approximately 2.5 percent compared with about 13 percent for chrysotile. The sensitivity of the X-ray diffraction method also ranges from 0.5 to

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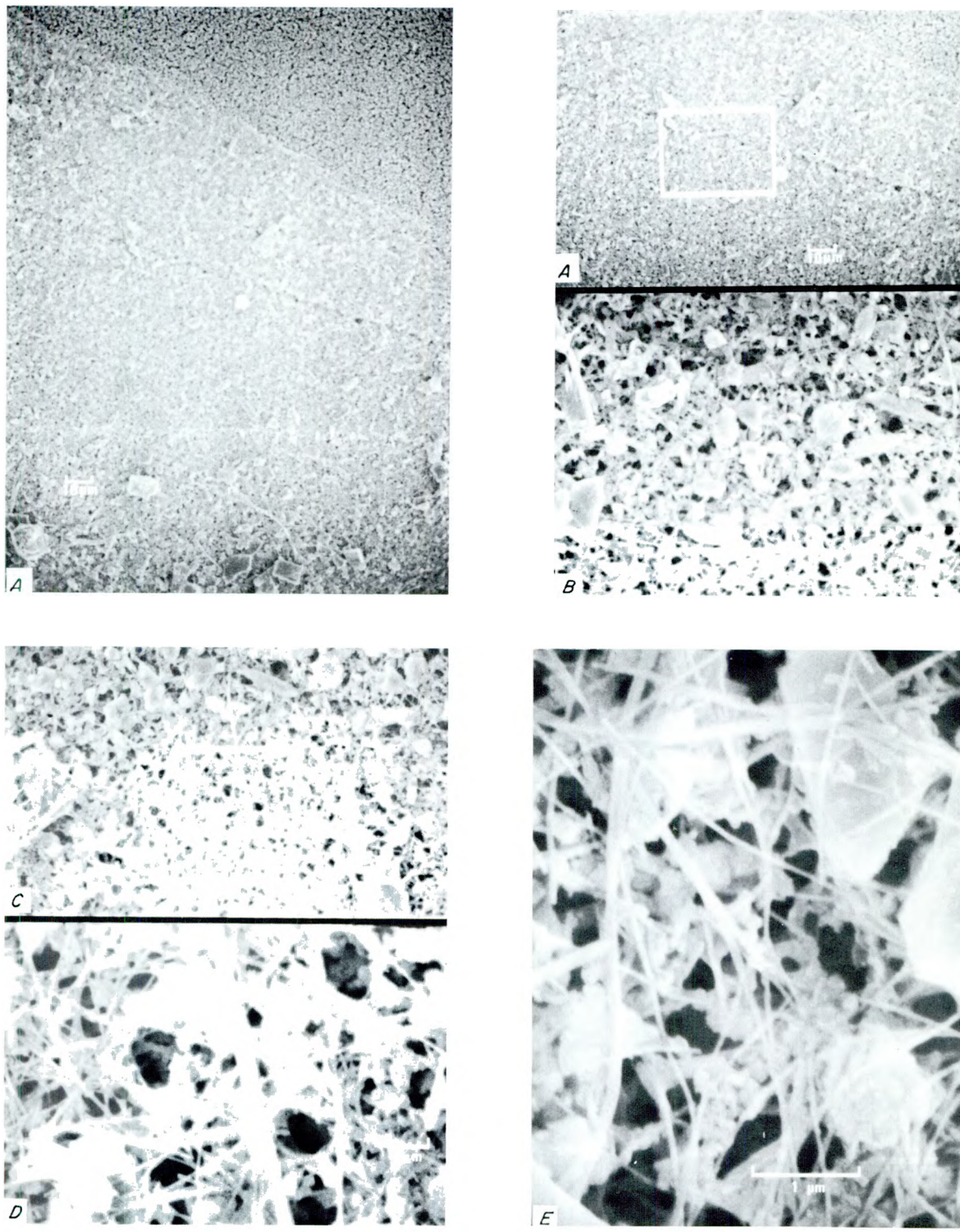


FIGURE 47. - Mixture of nonasbestiform serpentine and chrysotile at five magnifications: *A*, X 450; *B*, 2,250; *C*, X 9,000; *D*, 1,800; and *E*, X 18,000. Rectangles indicate the area shown in the next panel.

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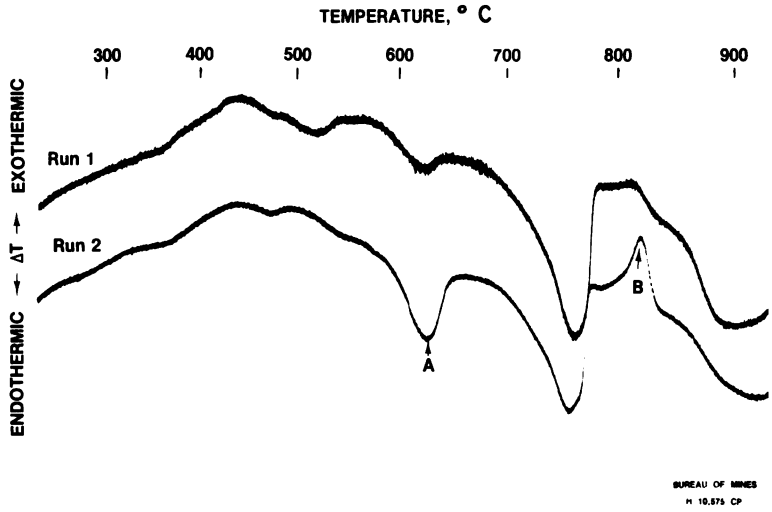


FIGURE 48. - Differential thermal analysis of sample from school ceiling, showing endothermic (A) and exothermic (B) peaks of serpentine. Run 1 is the sample as received; run 2 is a mixture of 95 pct of the as-received sample and 5 pct chrysotile.

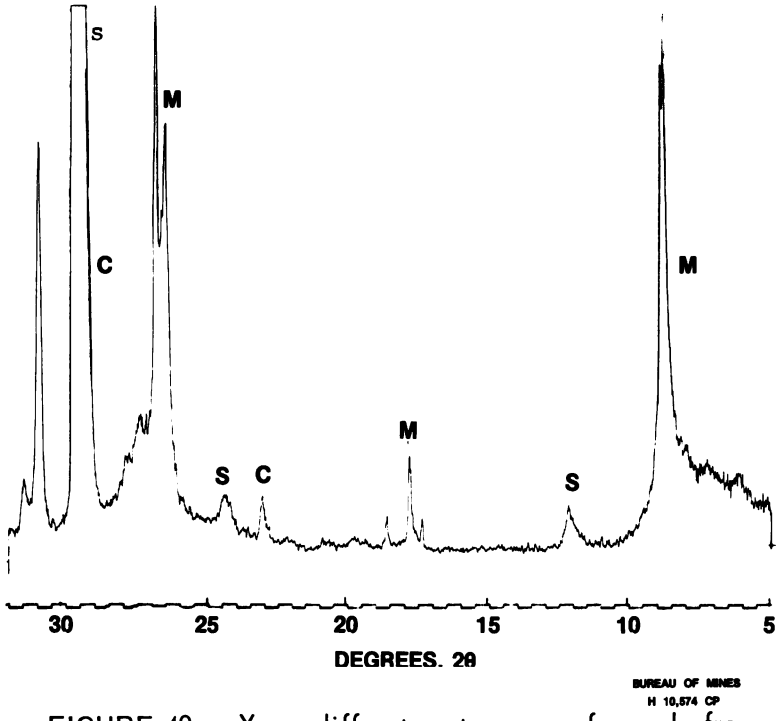


FIGURE 49. - X-ray diffractometer scan of sample from school ceiling, showing the presence of calcite (C), mica (M), and serpentine (S).

1.0 weight-percent. An X-ray diffractometer scan of the 2θ range for major serpentine peaks is shown in figure 49. The magnitude of the characteristic peaks for chrysotile are a function of several factors, including degree of fiber orientation and the type of milling or crushing used to process the sample. Also, the sensitivity of both methods is affected by the presence of other minerals that have characteristic thermal or diffraction peaks in the same region as those of the minerals of interest.

Some samples will be composed of a mixture of synthetic and natural fibers, such as the mixture of fiberglass and chrysotile shown in figure 50. Generally, it is not difficult to identify the synthetic fibers based on their larger diameter and the more uniform appearance.

Amphiboles and Talc

Asbestos-related health regulations are having a significant impact on the domestic talc industry from occupational exposure at the mines and mills and at various manufacturing plants that use talcs in their operations. Certification that the talc does or does not contain asbestiform minerals is important because the occupational health requirements are much more restrictive if the talc is designated as containing asbestiform serpentine or amphibole minerals.

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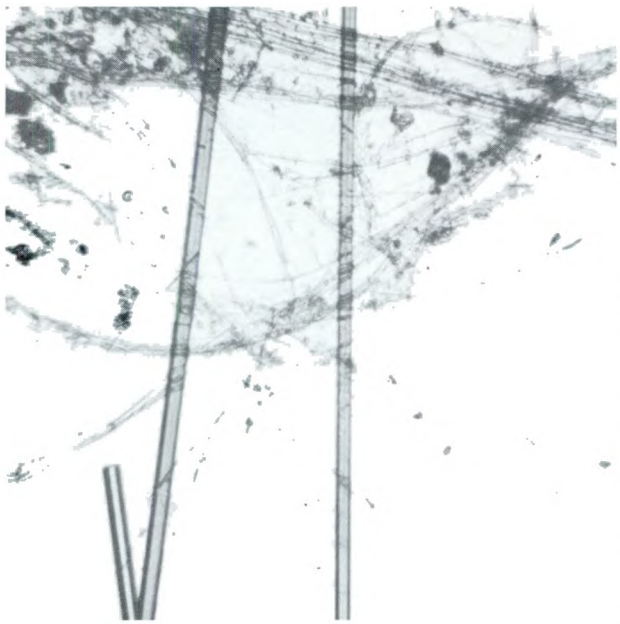


FIGURE 50. - Sample from university building, showing a mixture of chrysotile and fiberglass (X 140).

Talc is both the name of a specific mineral, $Mg_3Si_4O_{10}(OH)_2$, and a commercial term for a mixture of minerals ranging from essentially 100 percent talc to blends where the mineral talc is a minor constituent (12, 23). Semiquantitative estimation of the serpentine and/or amphibole mineral concentration, if present, can be obtained by X-ray diffraction and differential thermal analysis. Several talc deposits contain a variable amount of tremolite. Therefore, the essential question faced by the analyst is whether or not the tremolite is fibrous. Figure 51 shows the typical platy morphology of talc; no tremolite (amphibole) was detected in this sample by X-ray diffraction. Figure 52 illustrates the type of particles obtained from a mixture of tremolite and platy talc. The cleavage fragments of tremolite are typical of the nonasbestiform variety. Better judgment is required of the analyst with the type of sample illustrated in figure 53. This sample consists of platy talc, cleavage fragments of tremolite, and minor to trace amounts of fibrous tremolite. For this latter sample, the 3-to-1 aspect-ratio criteria would greatly overestimate the number of fibrous tremolite particles collected on air filters or other monitors.



FIGURE 51. - Typical platy morphology of talc (X 600).

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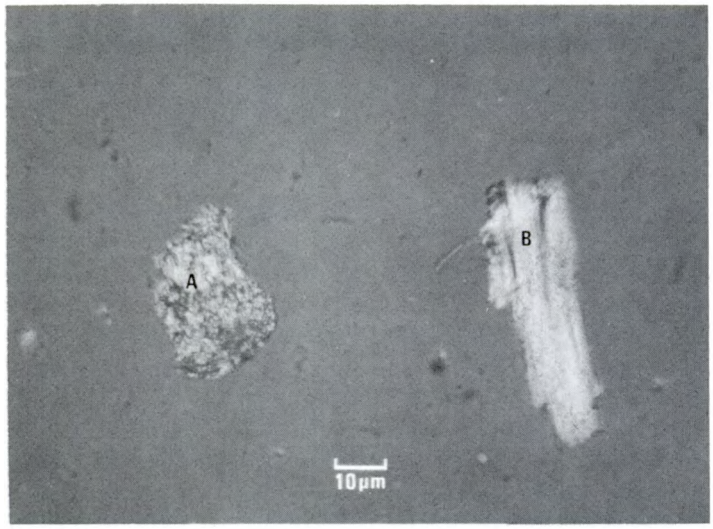


FIGURE 52. - Platy talc (A) and tremolite cleavage fragment (B) (X 640).

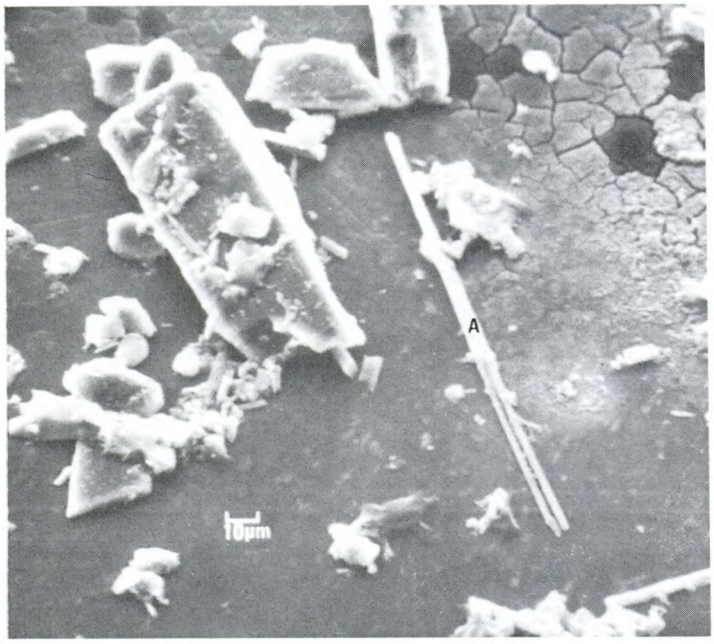


FIGURE 53. - Platy talc, tremolite cleavage fragments, and a fibrous tremolite particle (A) (X 400).

RESEARCH NEEDS

There are several areas in particulate identification-characterization where further research is warranted. Areas of research that are immediately applicable to asbestos are briefly summarized.

Commercially available electron optical instruments are generally limited to morphological characterization for mineral particles with diameters less than 0.2 μm. As pointed out in the identification-characterization section, both the signal-to-background ratio for energy dispersive X-ray spectra and the SAED pattern are significantly degraded for elongated particles less than 0.2 μm in diameter. Field emission electron optical microscopes with their higher vacuums and smaller beam diameter may have some advantages over conventional SEM instruments. Also, other microprobe techniques, in particular ion microprobe mass spectrographs and laser Raman microprobes, should be evaluated for particulate characterization.

Although electron microscopic methods can generally positively identify chrysotile in air and water samples, the quantitative aspects of the measurements need substantial improvement. Sample treatment and measurement errors need to be isolated from sampling variance. Because of the ambiguity of fiber counts, results should be reported both in mass equivalents and in fibers per unit volume.

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Fundamental studies should be conducted to determine if there are unique chemical and physical characteristics of a mineral fiber as compared to elongated cleavage fragments. For example, the surface properties of chrysotile are similar to those of magnesium hydroxide, whereas the nonasbestiform varieties of serpentine have the surface characteristics of a silicate (30). Variations in surface properties, if any, between asbestiform particles and cleavage fragments of amphiboles should be investigated. Surface characterization techniques to be considered should include Auger electron spectrography and low-energy X-ray spectrography. Research at the University of Minnesota indicates that asbestos fibers have an extensive surface charge over the whole surface, whereas cleavage fragments have a significantly lower surface charge (40). Extinction angle measurements are another possible approach to distinguishing asbestiform from the nonasbestiform varieties of amphiboles (37).

There is a critical need to reexamine the 3-to-1 aspect ratio as a criterion for a mineral fiber. The aspect ratio for fibers from asbestiform minerals were as much as 200 to 1 or higher, whereas the ratio for cleavage fragments is about 3 to 1, as illustrated by the data in this report. The 3-to-1 aspect ratio may be valid for the industrial hygiene control of asbestos-processing plants; however, its applicability to existing nonasbestos mining and ore processing plants requires critical evaluation. There is also need to evaluate the restriction of the mineral particulate measurments to light optical microscopy because many particulates of interest, especially chrysotile fibrils, are not visible by this technique. Low-cost scanning electron microscopes are in the same price range as research-grade petrographic microscopes, and the skill requirements for the operator are comparable for both instruments.

Particulate measurements by microscopic procedures are time consuming and expensive. Two possible approaches to reducing the time and cost are (1) automation of the particulate identification-characterization measurements using computerized image analyzers and (2) development of chemical reagents that give a specific response with either chrysotile or the various asbestiform amphibole minerals. Using the chemical-reagent approach, mass-concentration values could be obtained by measurement of some response such as color, ultraviolet fluorescence, X-ray spectral intensity, etc.

Health studies related to inhalation and ingestion of fibers have been essentially limited to well-defined commercial types of asbestos. The fundamental question to be resolved is the biological effects of cleavage fragments compared with those of true mineral fibers. If shape and size are the critical parameters, the analyst could establish and measure suitable analytical parameters to monitor the particulates of interest. Likewise, if the health scientists find a correlation between health and the amount of trace metals, adsorbed organics, surface area, etc., the analyst can respond accordingly. Therefore, there is a primary need for an adequate quantity of well-characterized amphiboles and serpentine of both the normal and asbestiform varieties for use in health-related studies.

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