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IDENTIFICATION OF SELECTED SILICATE MINERALS AND THEIR ASBESTIFORM VARIETIES

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Abstract

The problem of asbestiform particulates with its environmental and health implications has been compounded by the lack of precision with which the term "asbestos" has been used. In many instances, non-asbestiform mineral particles have been identified as microscopic fibers of asbestos-related minerals. This lack of precision in identifying these particulates not only works to the disadvantage of the minerals industry, but is also a handicap to rational science-based decision making by regulatory agencies.

This presentation summarizes methods and terminology suggested by the Bureau of Mines for the identification and characterization of asbestiform minerals and also sharpens the distinction between common serpentine and amphibole minerals and their relatively rare asbestiform varieties.[1] The continuing effort of the Bureau's Particulate Mineralogy Unit is to characterize mineral particles by morphological, compositional, and structural data using various instrumental analytical techniques and by developing new methods for identification and characterization.

Key Words: Asbestos; cleavage fragments; fibers; silicate minerals.

Introduction

The objective of this paper is to present a general introduction on the identification and characterization of asbestos-related minerals. Detailed discussions of specific analytical techniques are given in other papers presented at this workshop. At present there are three types of identification-characterization to supply the needs of regulatory agencies, medical researchers, and mineral scientists. It is hoped that through interactions such as this workshop a common mineralogical-based procedure can be developed that meets the needs of all concerned groups.

Until recently, emphasis in the United States was placed on occupational exposure of employees manufacturing or using asbestos products for insulation and other applications. 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 correlating

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[1] This paper is an abbreviated version of the sections on mineral identification and characterization in Bureau of Mines Information Circular 8751 - Selected Silicate Minerals and Their Asbestiform Varieties: Definitions and Identification-Characterization, 1977, 56 pp, authored by W. J. Campbell, R. L. Blake, L. L. Brown, E. E. Cather, and J. J. Sjoberg. Copies of IC 8751 are available upon request to W. J. Campbell.

health effects to the number of fibers observed. Exact definitions for asbestos-related mineralogical terms were 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 [1][2]. 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 [2]. 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 with the conventional light optical microscopic procedures. Therefore, the identificaiton 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 [4]. Detailed descriptions of the various analytical and characterization techniques are available in numerous publications and textbooks.

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.

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[2] Figures in brackets indicate the literature references at the end of this paper.

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 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. 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 [5,6]. The distinction between serpentine and amphibole minerals at the macroscopic level can be made by 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 [7] 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 chemical characterization of amphiboles, 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 [8]. 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.

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

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 [9]; however, this technique is limited to particles with diameters greater than about 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.

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 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. 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 [10]. Researchers indicate that the ability to distinguish between the fibrous and nonfibrous variety of amphiboles by SAED is still to be resolved.

At the very high magnification available with a TEM, chrysotile's hollow-tube (scroll-like) structure, approximately 5 nm in diameter, is visible (fig. 1). 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 several fibrils) will not display this characteristic because of stacking of the fibrils.

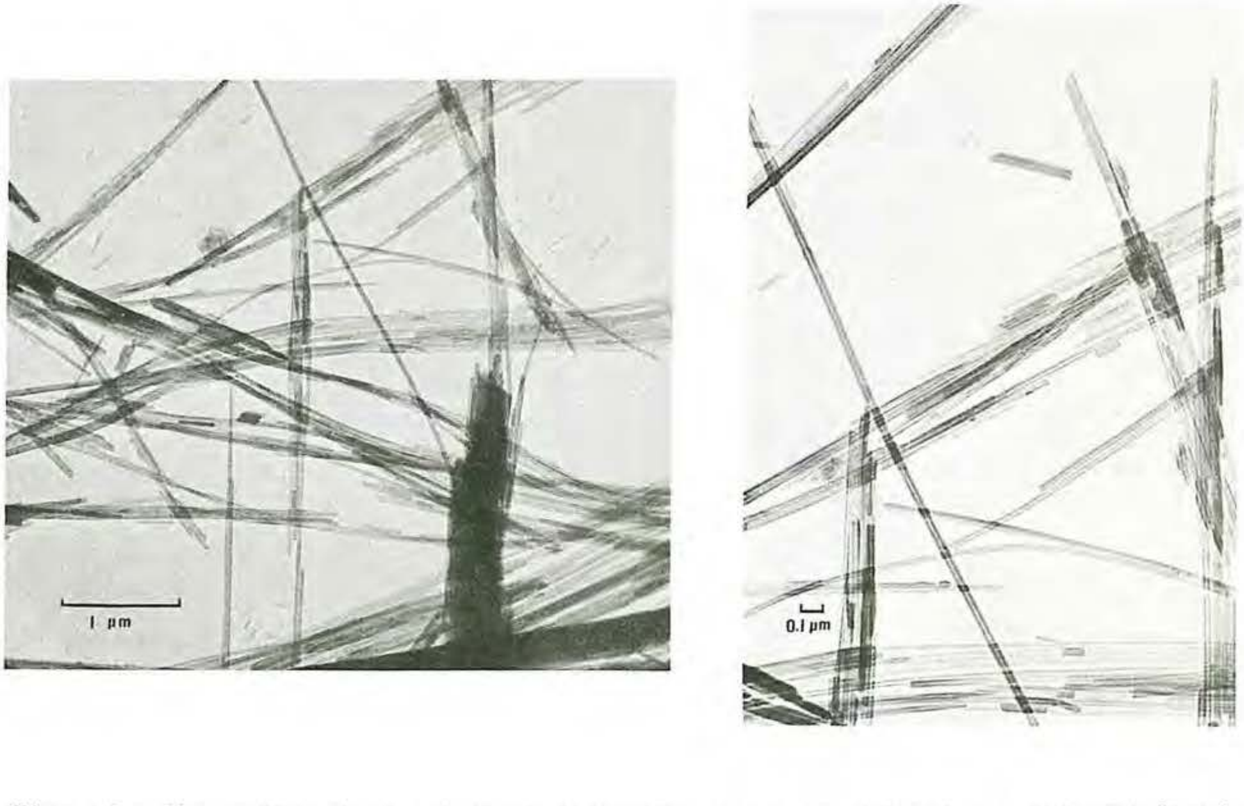

Figure 1. Chrysotile, showing individual fibrils, at two magnifications: X 18,000 (left) and X 35,000 (right). The hollow-tube structure is visible at the higher magnification. (TEM microphotographs.)

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 [3].

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 beam diameters of 0.1 to 0.01 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. 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 chrysotile. A good example is the diameter size distribution of 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,[a] percent.

Diameter of chrysotile fibers, μm	- - - - - Sample - - - - -					
	1	2	3	4	5	6
0.02 - <0.04	10	70	57	17	15	17
0.04 - <0.06	47	24	28	29	33	49
0.06 - <0.08	24	5	8	28	20	15
0.08 - <0.10	14	1	2	12	26	6
0.10 - <0.12	2	0	1	7	3	6
0.12 - <0.14	0	0	2	3	1	1
0.14 - <0.16	1	0	1	2	1	1
0.16 - <0.18	0	0	0	1	0	1
0.18 - <0.20	0	0	0	0	1	1
0.20 - <0.22	1	0	0	0	0	1
0.22 - 0.24	0	0	1	0	0	1
>0.24	1	0	0	1	0	1

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

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 sample substrate.

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 in IC 8751. 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. 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 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.

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. 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. 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.

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 2 to 5. The samples shown in figures 2 to 4 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 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.

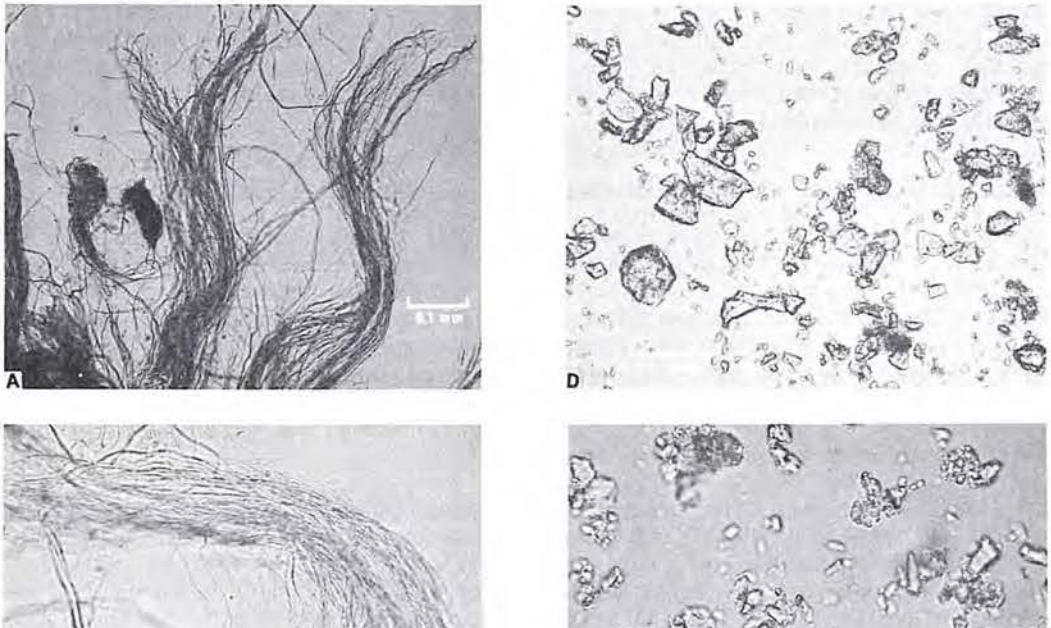

Figure 2. Light optical photomicrographs of chrysotile and antigorite-lizardite at three magnifications. Chrysotile (left) at A, X 100; B, X 500; and C, X 950. Antigorite-lizardite (right) at D, X 100; E, X 500; and F, X 950.

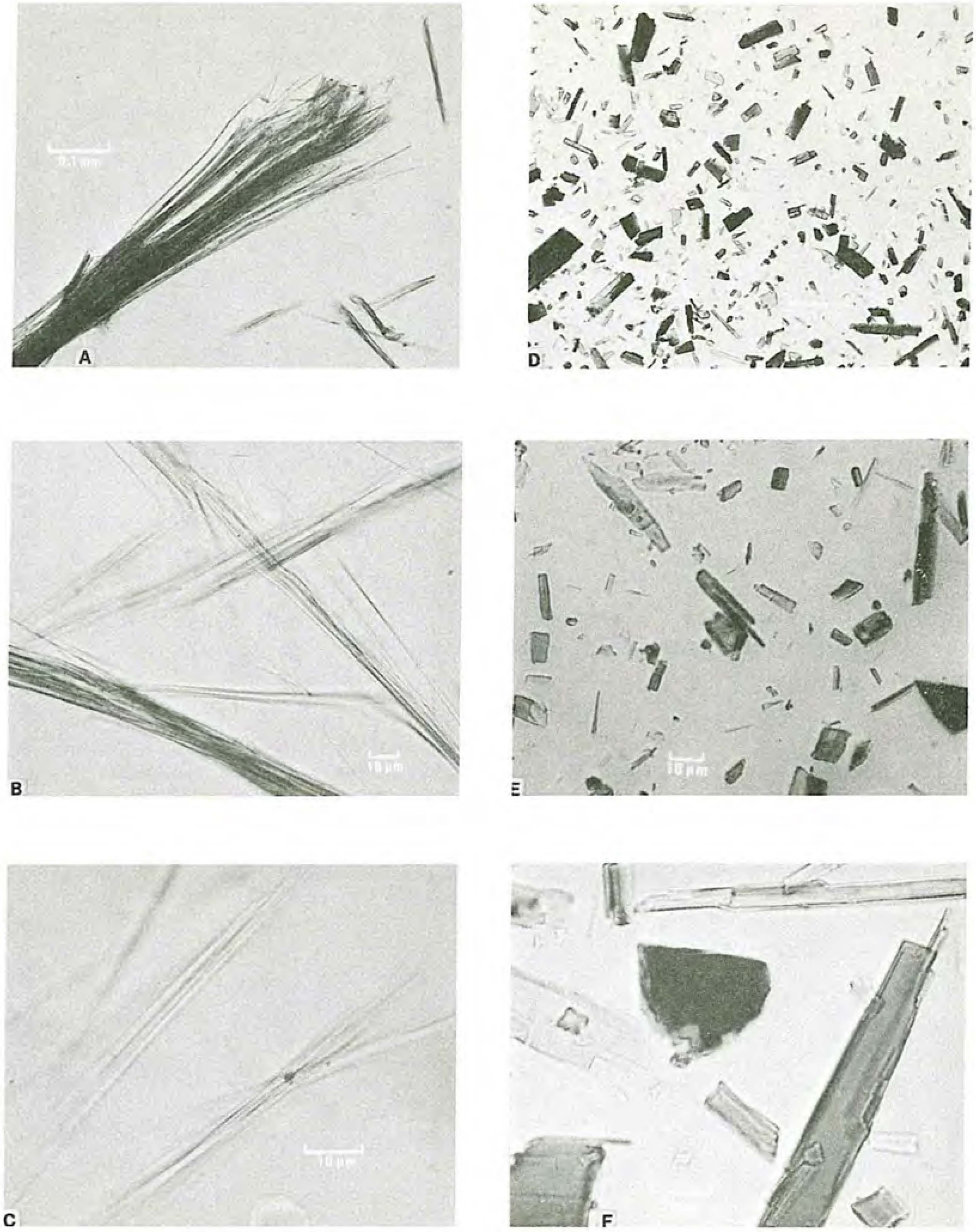

Figure 3. Light optical photomicrographs of crocidolite and riebeckite at three magnifications; Crocidolite (left) at A, X 100; B, X 500; and C, X 950. Riebeckite (right) at D, X 100; E, X 500; and F, X 950.

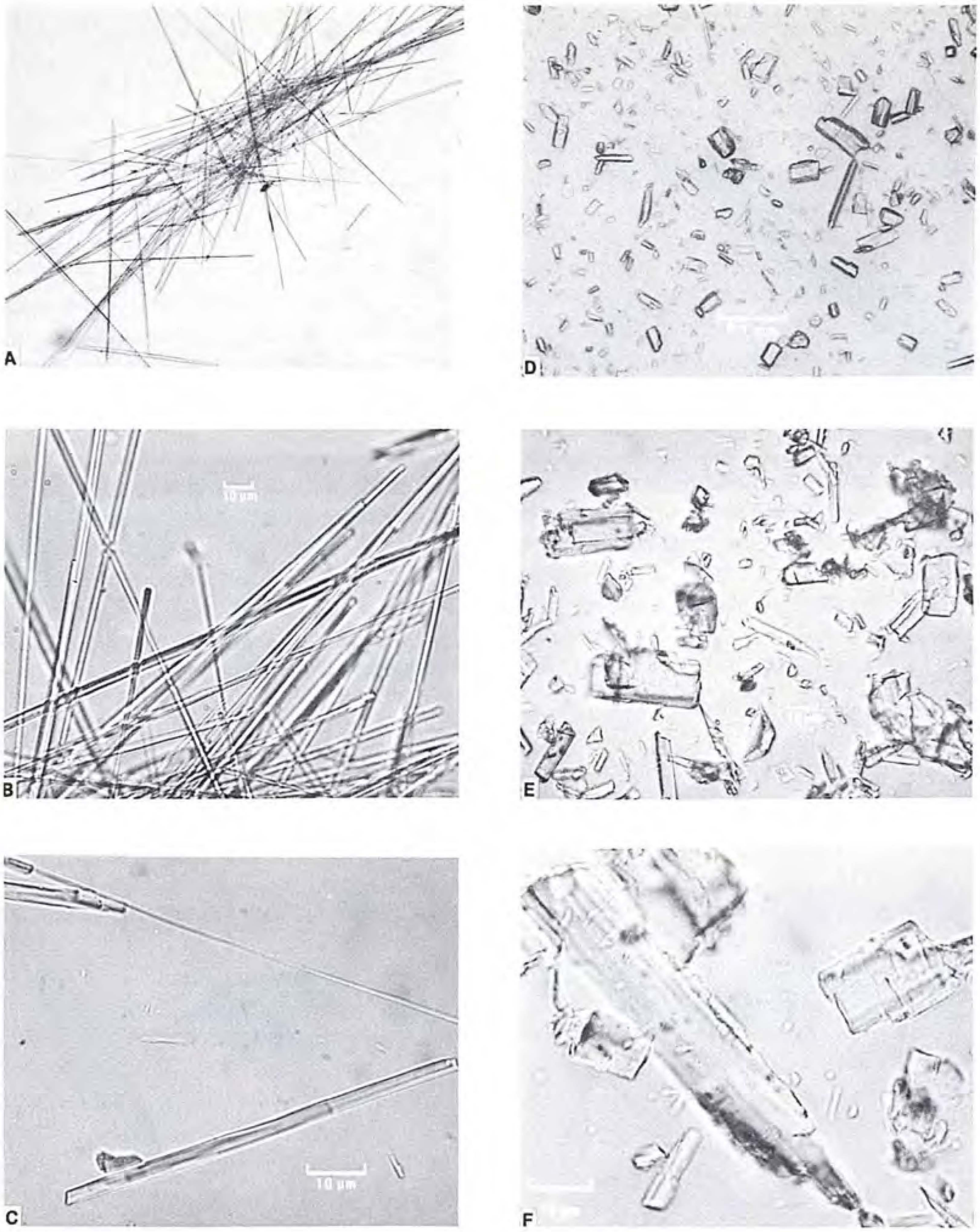

Figure 4. Light optical photomicrographs of tremolite asbestos and tremolite at three magnifications. Tremolite asbestos (left) at A, X 100; B, X 500; and C, X 950. Tremolite (right) at D, X 100: E, X 500; and F, X 950.

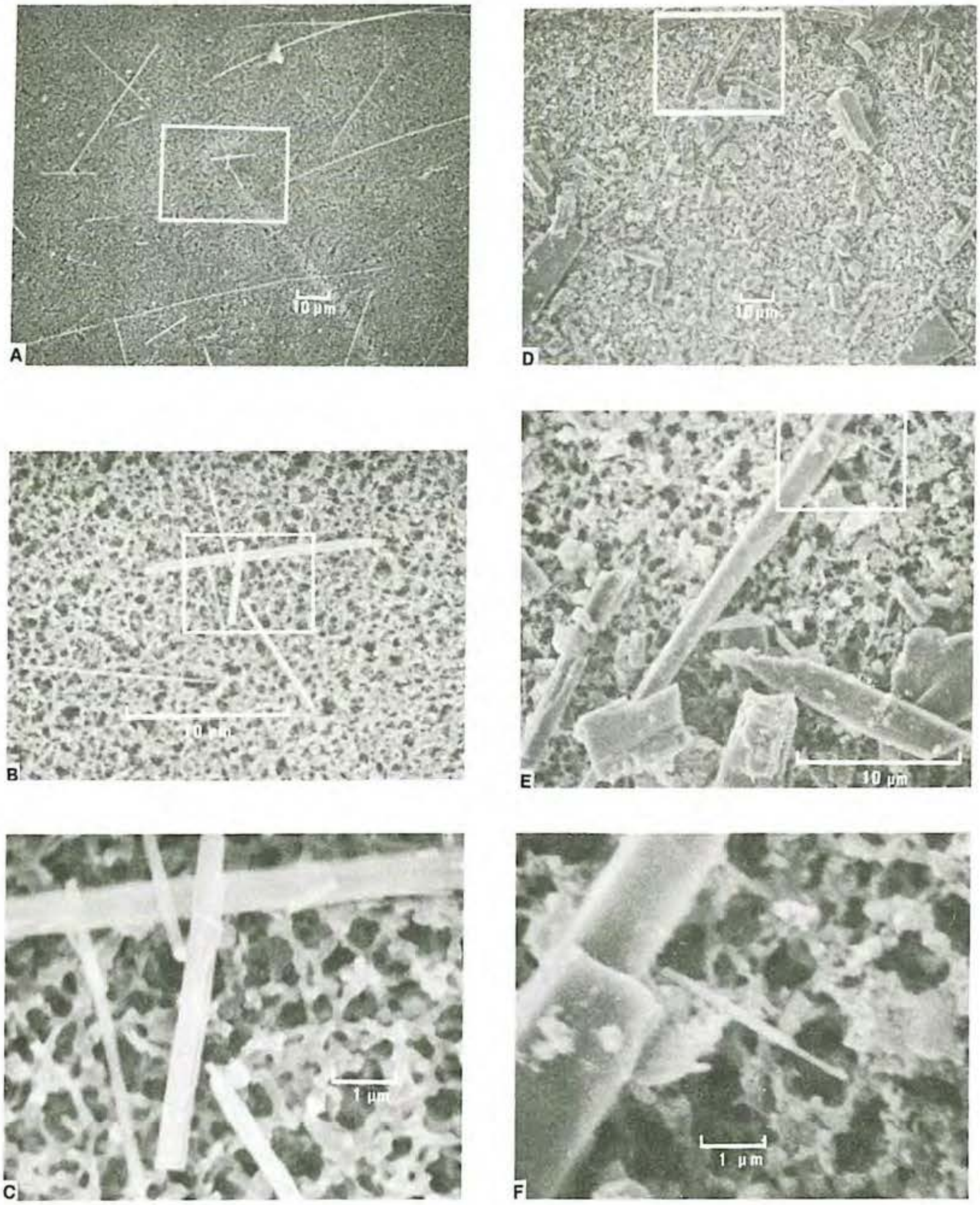

Figure 5. SEM photomicrographs of crocidolite and riebeckite at three magnifications: Crocidolite (left) at A, X 500; B, X 2,500; and C, X 10,000. Riebeckite (right) at D, X 500; E, X 2,500; and F, X 10,000. Rectangles indicate the area shown at the next higher magnifications.

Riebeckite and crocidolite particles are compared at higher magnifications in figure 5. 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. The aspect ratio has little mineralogical significance for individual particulates but is applicable statistically 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 6 to 9.

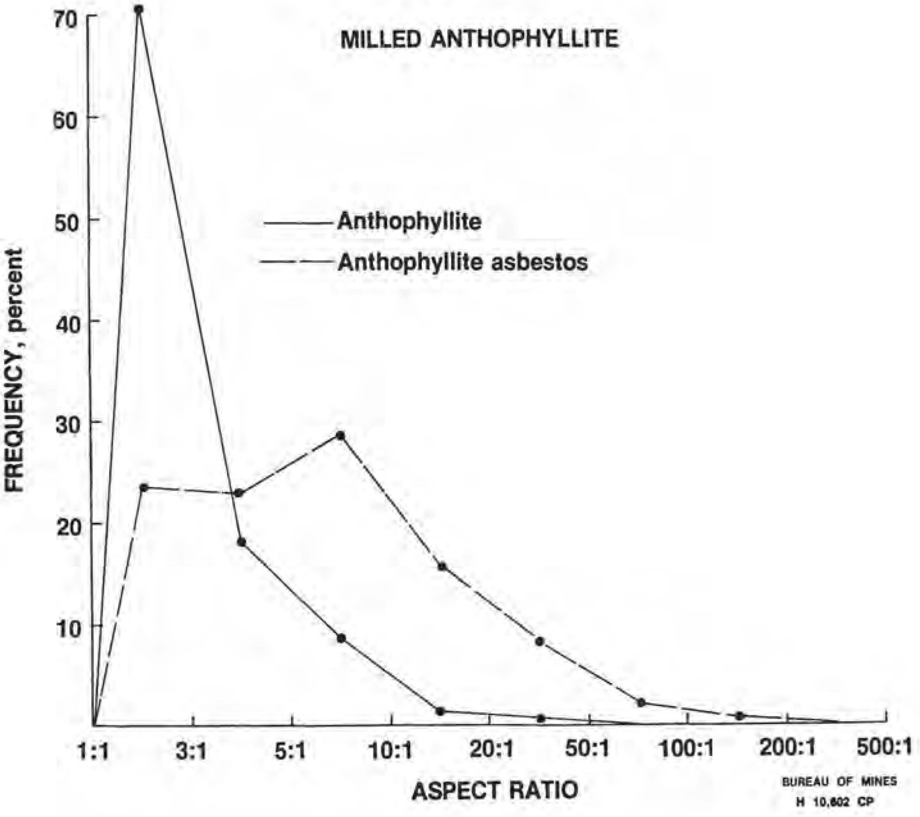

Figure 6. Frequency polygons for the aspect ratios of anthophyllite and anthophyllite asbestos.

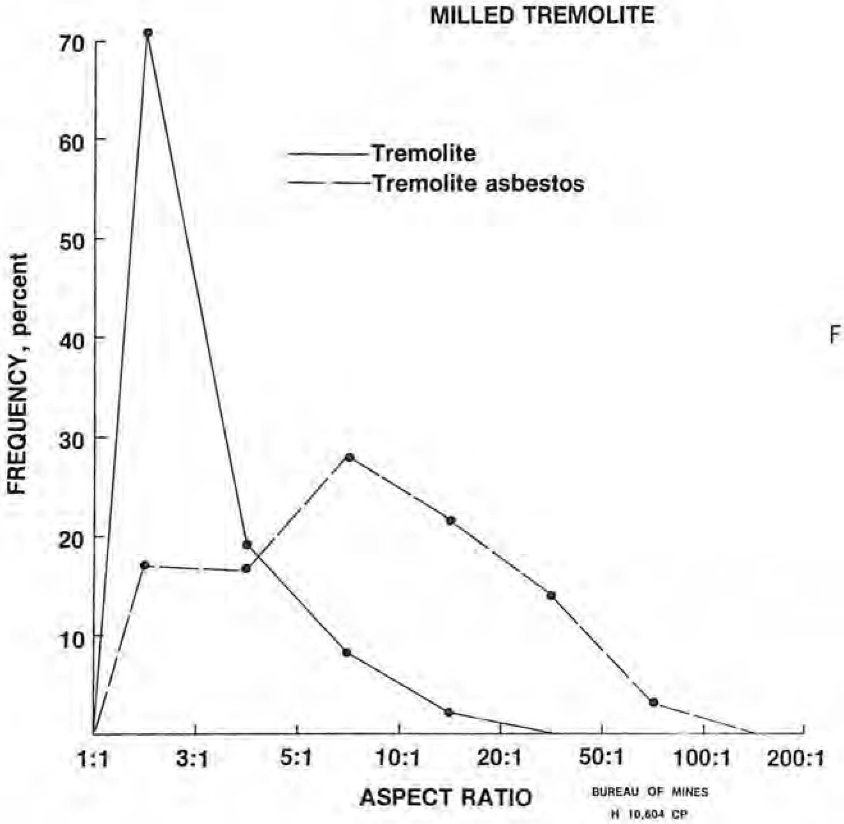

Figure 7. Frequency polygons for the aspect ratios of tremolite and tremolite asbestos.

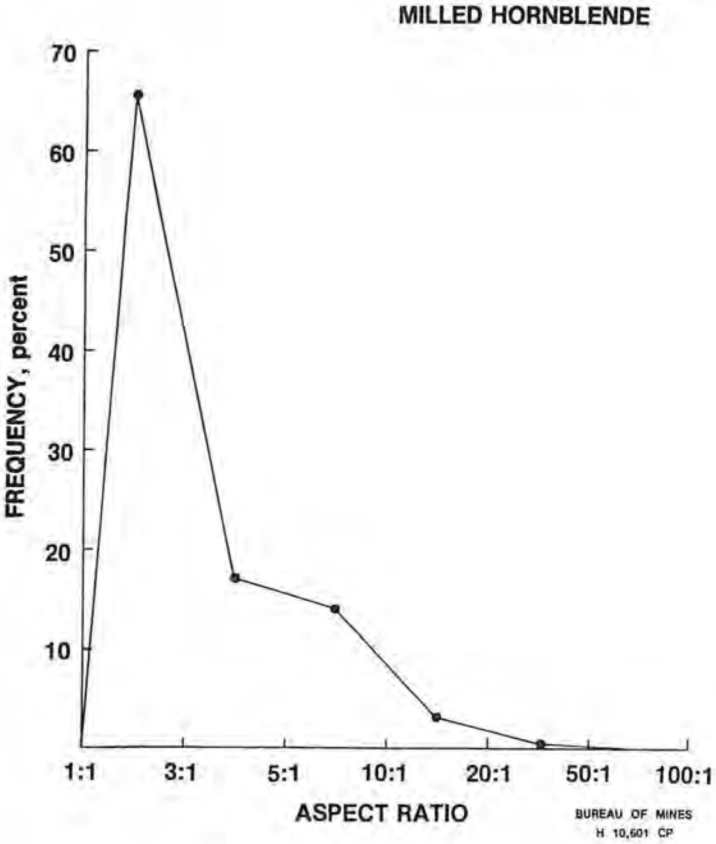

Figure 8. Frequency polygons for the aspect ratio of hornblende.

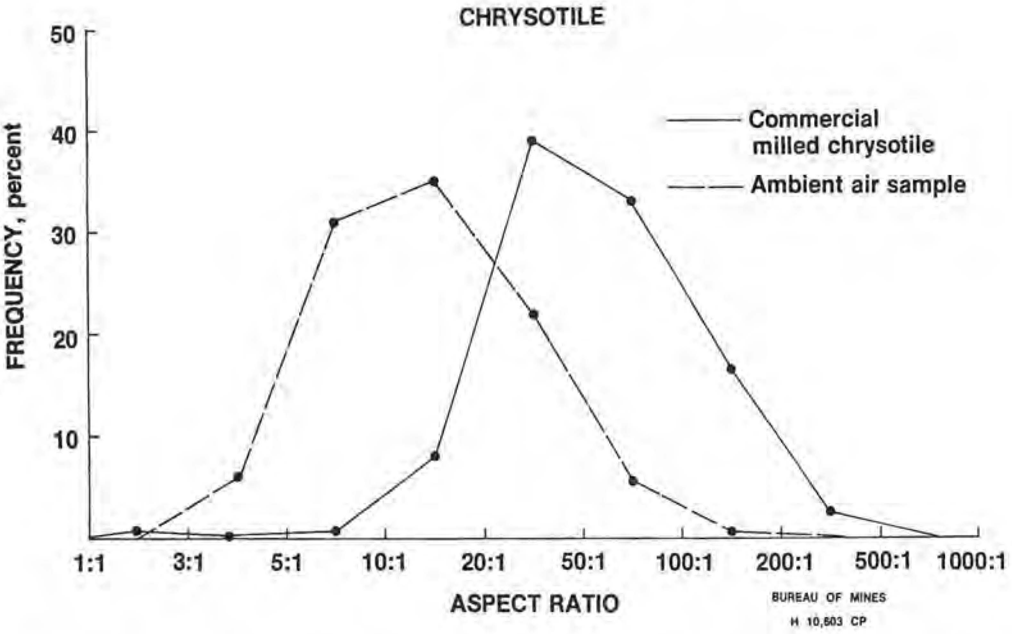

Figure 9. Frequency polygons for the aspect ratios of commercial-grade chrysotile and chrysotile in ambient air.

Figures 6, 7, and 8 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 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 maxima 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 9 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 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 [11]. 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.

Ambient-Air Samples Collected Near Serpentine 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 (figure 10). 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. 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 1,000A (Table 3).

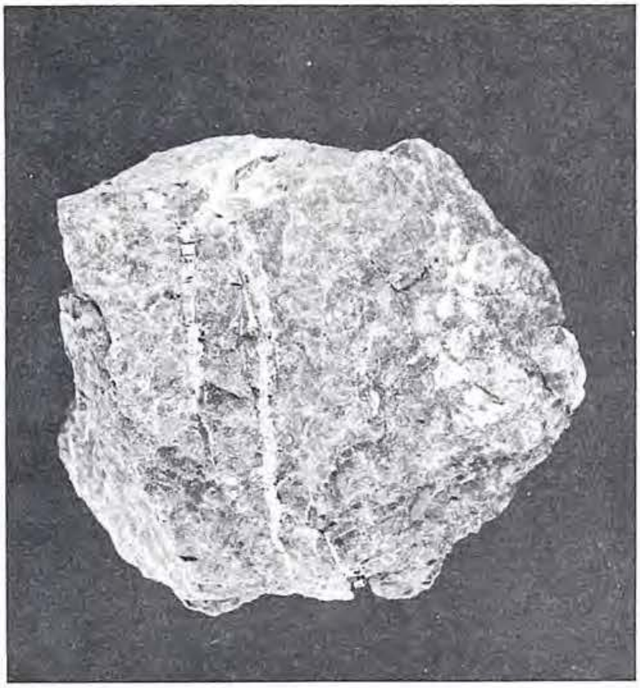

Figure 10. Macrophotograph showing chrysotile veins in serpentine rock (X 1).

Figure 11 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.

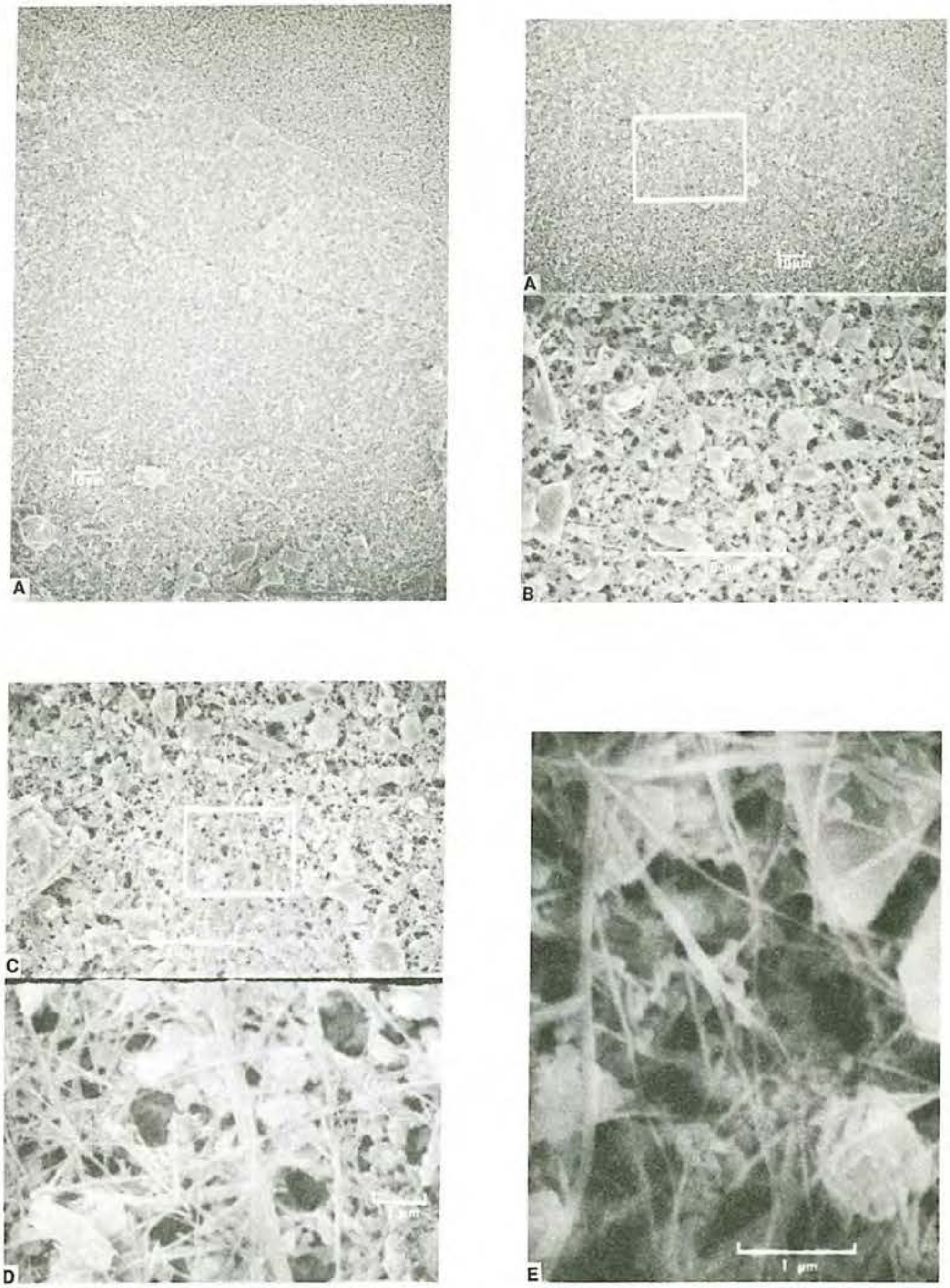

Figure 11. Mixture of nonasbestiform serpentine and chrysotile at five magnifications: A, X 450; B, X 2,250; C, X 1,800; D, X 9,000; and E, X 18,000. Rectangles indicate the area shown in the next panel.

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 test. Three complementary analytical methods for screening, identification, and semi-quantitative 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.

Some samples will be composed of a mixture of synthetic and natural fibers, such as the mixture of fiberglass and chrysotile shown in figure 12. Generally, it is not difficult to identify the synthetic fibers based on their larger diameter and the more uniform appearance.

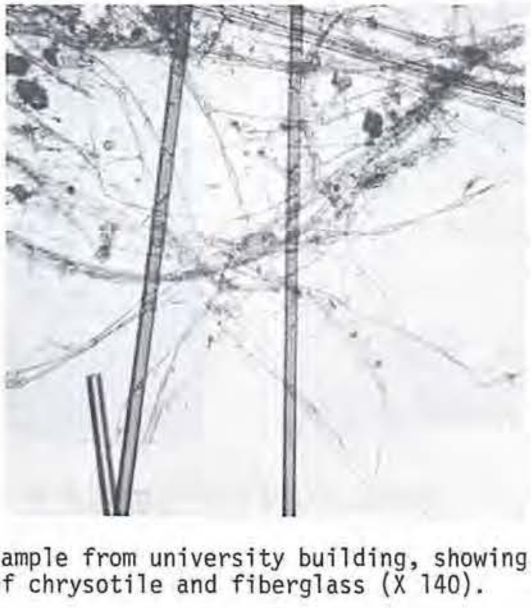

Figure 12. Sample from university building, showing a mixture of chrysotile and fiberglass (X 140).

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.

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 [12,13]. Semi-quantitative 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. Judgment required of the analyst is illustrated by the sample shown in figure 13. This sample consists of platy talc, cleavage fragments of an amphibole, and minor to trace amounts of fibrous amphibole. For this latter sample, the 3-to-1 aspect-ratio criteria would greatly overestimate the number of fibrous amphibole particles collected on air filters or other monitors.

Figure 13. Platy talc, tremolite cleavage fragments, and a fibrous tremolite particle (A) (X 400).

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Discussion

J. LEINEWEBER: You brought up the question of cleavage fragments vs fibers, and asbestiform vs non-asbestiform varieties. I would like to ask why you attach so much significance to this. I think Dr. Kotin couched it most directly yesterday: the body doesn't have a dictionary. When we see fibers, if they are in the size range and if we accept this philosophy, does it matter where they come from?

W. CAMPBELL: I think all health data has been based on commercial asbestos, correct?

LEINEWEBER: Not necessarily commercial asbestos, but fibers of one type or another.

CAMPBELL: OK, fibers.

LEINEWEBER: Man-made mineral fibers or natural mineral fibers.

CAMPBELL: There has been little medical studies made upon cleavage fragments. Now these may be just as harmful as fibers, but until you find this out you should call them by their proper names. To call a cleavage fragment a fiber does not help anybody.

LEINEWEBER: I don't see any reason for muddying the waters with the semantic differences.

CAMPBELL: I think there is some dispute whether or not there is a difference between a fiber, based on surface properties and a much larger length-to-width, and a cleavage fragment. Until you find this out you should call it either a fiber or a cleavage fragment. They may be equally harmful if they are both 20:1 and 0.5 µm in diameter, but this really has not been studied. The whole problem with the Lake Superior region was the debate whether or not the cummingtonite fragments were the same as the amosite asbestos.

LEINEWEBER: This, in that context, was an argument based on the shenanigans that normally take place in the court of law, and here we are in a scientific environment.

CAMPBELL: I am not a medical scientist. Obviously I don't know if a cleavage fragment is the same harmful particle as an asbestiform particle, but until you find this out; you just call it by the proper name. It does not help to call them both the same when they may be different.

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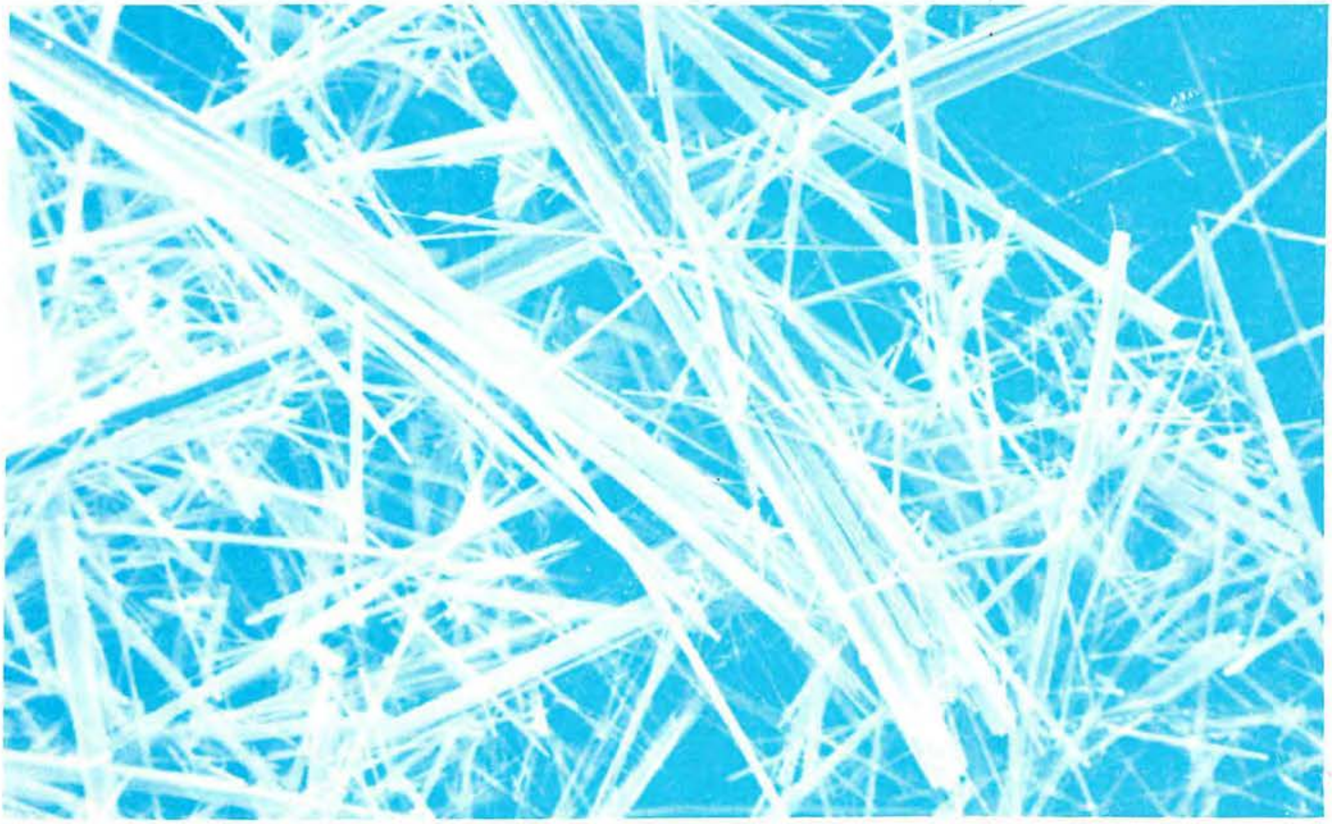
U.S. DEPARTMENT OF COMMERCE / National Bureau of Standards

Workshop on Asbestos: Definitions and Measurement Methods



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Edited by

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