

147

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IDENTIFICATION OF FIBROUS AND NONFIBROUS
AMPHIBOLES IN THE ELECTRON MICROSCOPE

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PLAINTIFF'S EXHIBIT

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The extensive and systematic epidemiologic and other studies of Selikoff and associates at Mount Sinai School of Medicine have aroused public recognition of adverse health effects due to exposure to asbestos dust, especially among insulation workers and others in the asbestos "trades."^{1,2} In these cases, monitoring concentrations of airborne fibrous dust in the work environment by phase-contrast optical microscopy is satisfactory. However, the suggestion that acicular fragments of nonfibrous amphiboles below the limit of optical microscopy may pose similar hazards has stimulated intensive development of electron microscopic techniques for identifying and counting particles in air and water samples.³

Until the complexity of differentiating between the numerous mineral species that might be present was recognized, an apparent aspect ratio of 3:1 and the resemblance of a selected area electron diffraction pattern to that from a standard sample seemed sufficient criteria for identification.³ However, a large number of silicate minerals have prismatic cleavage and unit cells similar to the commercial asbestos mineral varieties.^{4,5} Furthermore, the aspect ratio of fine particles produced by spontaneous or imposed comminution is a manifestation of anisotropy in crystallographic and physical characteristics. Thus, differences in macroscopic attributes, such as fibrosity and optical properties, readily observed in hand samples of fibrous and nonfibrous mineral varieties,^{5,6} reflect differences in crystallization history and the resultant microstructure.

It is probable that the surface properties of fibers and cleavage fragments, such as the occurrence of ledges or steps, uncompensated bonds, and the nature and concentration of any molecular material that may be absorbed, will be directly related to the particular crystal faces that predominate.⁷ Thus, the toxicity of particles of different mineral species can be expected to vary considerably for crystallographic and chemical reasons, in addition to differences in fracture strength, particle length, and aspect ratio.

Until the basic mechanism for toxicity is established, electron optical characterization of particulates used for laboratory investigations of biologic activity must include positive identification of the mineral species from crystallographic and chemical analyses, determination of particle face orientations, and measurements of the distribution of particle dimensions and aspect ratios. The same information should also be obtained on air samples, especially when effects of exposure to particulates with no known health hazards are being evaluated.

PARTICLE IDENTIFICATION

Unknown particles can be identified in the transmission electron microscope (TEM) with a high degree of certainty (~95%) if two selected area diffraction (SAD) patterns are obtained from the same particle by tilting into two distinct zone axis

orientations.⁸ The measured spacings and angles on both patterns should be compared with all possible matches with any crystalline phases that could be present and the measured d-spacings indexed as in FIGURE 1e, f. Nearly the same certainty of identification can be obtained by indexing one good zone axis SAD pattern and recording an x-ray emission spectrum to establish its chemical composition. These data, illustrated in FIGURE 1, should be compared with a complete suite of reference standards; computer procedures have been developed for this purpose.^{9,9} If these procedures are not used there is a high probability of misidentification, especially if SAD patterns do not correspond to a zone axis orientation and they are classified by visual inspection, i.e. the so called "characteristic pattern" technique. However, misidentification is not a problem if only one type of particle is present such as in emission samples collected from a known point source. Conversely, when many compounds and various mineral species may be present in ambient air samples taken near quarries, mines, industrial cities, roads, or cultivated farmland, "positive" identification procedures must be used if the results are to have any significance.

PARTICLE COUNTING

A large variability in apparent particle concentration can result from difficulties with sample collection, particle losses and/or contamination during preparation of the EM specimens, casual identification, subjective definition of 3:1 aspect ratio, and small number statistical limitations. The reproducibility problem is aggravated by the large scale factor inherent in electron microscopy so that an erroneous identification of a single particle may be reported as a concentration of 10^6 or more per cubic meter of ambient air.

The optimum specimen preparation procedure that has been developed utilizes (1) low-temperature ashing to eliminate organic debris, (2) low energy ultrasonic agitation to break up agglomerates, (3) dilution and refiltering to obtain a satisfactory particle separation, (4) carbon coating of this filter to provide an electron transparent support film, (5) punching of 3 mm disks, (6) dissolution of filter material, (7) capturing carbon film with entrained particulates on TEM locator grids, (8) coating TEM grids with gold to provide a diffraction standard, and (9) examination using energy dispersive spectroscopy (EDS) and SAD. Direct observation confirms that actual particle loss is minimal when this procedure is followed.^{9,10} Photographic records should be used for particle size and shape measurements and for possible future reference if required.

The original volume of air sampled can be related to grid area through the series of dilution and scale factors involved. Typically, one full grid opening corresponding to about 10 liters of clean ambient air or to about 10 ml of very dusty air or stack samples resulting in "average" concentrations of ~2-10 particles per grid opening. Smaller values lead to prolonged searching to obtain adequate statistics, and higher particle concentrations complicate identification procedures.

The concentration (particles/cm³) in an air sample is determined by multiplying the number of particles observed by the average volume of air sampled by each grid opening and dividing the result by the number of openings examined:

$$C_i = \frac{N}{M \cdot V_{pgo}}$$

where

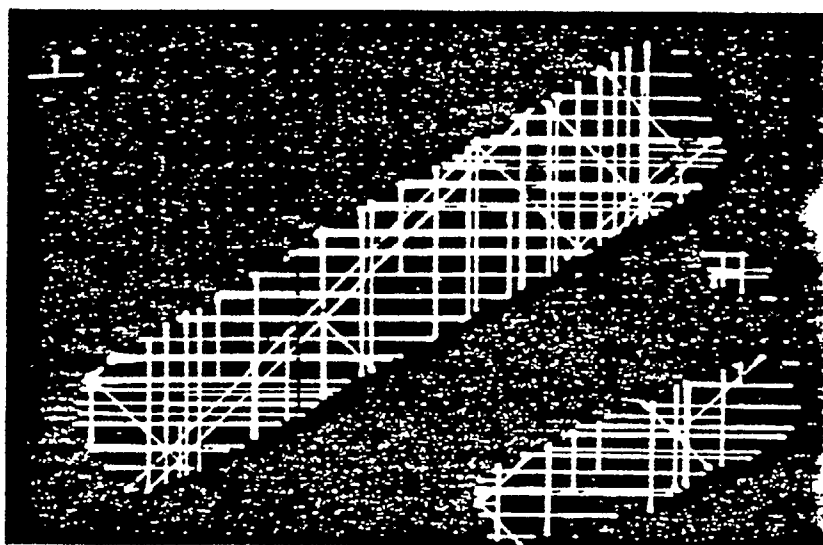
N = number of particles observed

V_{pgo} = (total volume of air collected) $\times$ (fraction of collection filter ashed) $\times$



(a) SE

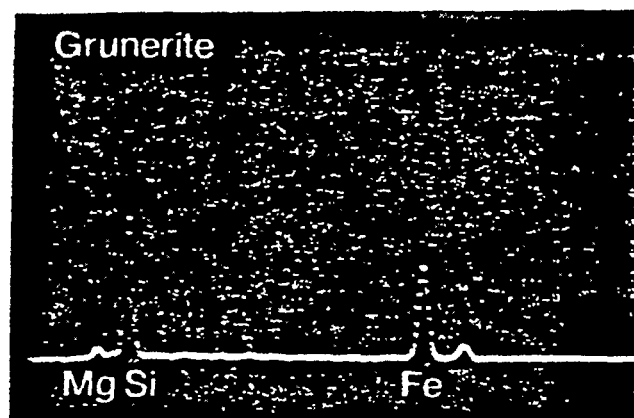
20,000X



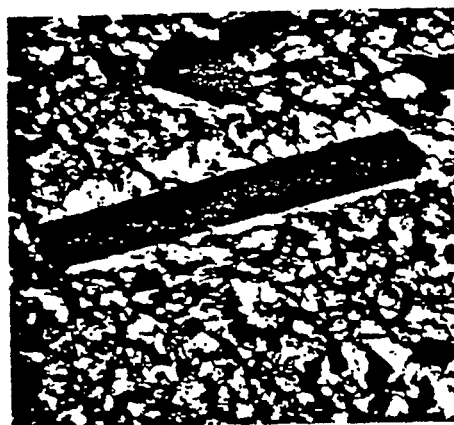
(b) AIA Trace

40,000X

FIGURE 1.(a,b) Representative data used in particle identification procedure using the SEM and TEM.



(c) X-ray Spectra



(d) TE 7200X

FIGURE 1. (c,d)

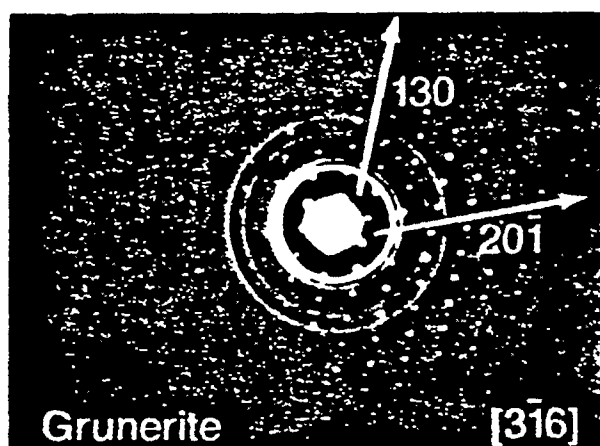
$(\text{fraction of ash redeposited}) \times (\text{mm}^2 \text{ per grid opening}) \div (\text{mm}^2/\text{laboratory filter})$

M = number of grid openings examined

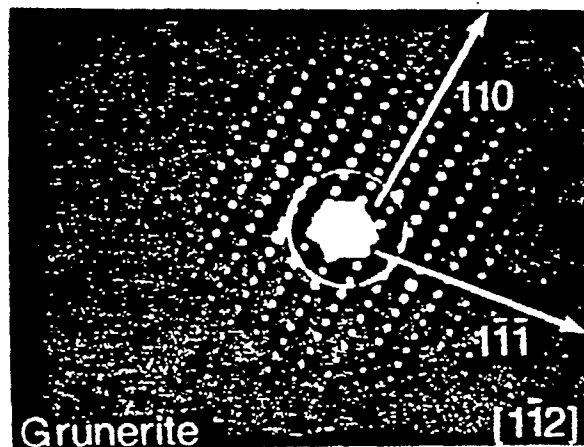
i = type of particle

The distribution of particles on properly prepared TEM grids should follow a Poisson distribution where the number of particles per unit area is small but constant. For heavily loaded, nonuniform, or agglomerated samples the Poisson model is not appropriate. A simple test to apply to the sample data is to calculate the mean and variance for the number of particles per grid opening. If these are approximately equal, the sample can be assumed to have come from a Poisson distribution. A

quantitative statistical test of this hypothesis utilizing the number of grid openings counted and measured, sample variance and mean should be carried out to derive the Poisson distribution parameters. This procedure makes it possible to report maximum and minimum concentrations of particles at a selected confidence level (e.g. 0.95). Tabulated data on particle numbers in air samples that have not been subjected to rigorous statistical analysis should be examined with some skepticism. It should be noted that the applicability of Poisson statistics to this counting problem imposes a significant variability in reported results. This distribution effect can only be overcome by counting large numbers of grid openings.



(e) SAD Pattern



(f) SAD Pattern

FIGURE 1. (c,f)

RAPID ELECTRON MICROSCOPE ANALYSIS METHOD

The identification and counting procedures reviewed above are, unfortunately, very time consuming and expensive. A simpler but generally satisfactory method is to group particles observed in the microscope into classes based on their dimensions and their x-ray emission spectra, i.e. their approximate composition. For example, the relative intensity of the elemental peaks (Mg, Si, Fe) in the x-ray spectrum in FIGURE 1 is compatible with particles in class H in TABLE 1 as well as several pyroxene and phyllosilicate minerals. Thus the x-ray spectrum effectively limits the possible identity of the particle to 2 or 3 minerals.

A complete SAD identification can then be carried out on randomly selected particles from each class to establish a positive identification of the class, and determine the homogeneity of the class. For the particle in FIGURE 1, indexing two zone axis SAD patterns positively identified the particle as grunerite. This scheme cuts the time required by a factor of 5-10, and permits calculation of the uncertainty of identification for the particles in each class using standard statistical methods. Thus, while some detail is lost by not positively identifying each particle, there is a

TABLE 1
DIMENSIONS AND COMPOSITION OF PARTICLE "TYPE" CLASSES

Type	Av. Size, μm	Aspect Ratio	Composition,* Percent							
			Na	Mg	Al	Si	K	Ca	Ti	Fe
A (Serpentine)	8	30	—	32	—	62	—	—	—	6
B (Pyroxene)	5	5	—	16	4	60	—	—	—	20
C (Clay)	7	19	—	1	40	50	1	—	—	8
D (Chlorite)	5	7	—	12	28	38	3	1	—	19
E (Mica)	6	8	—	10	24	40	9	1	1	14
F (Silica)	7	8	6	—	9	80	1	3	1	1
G (Augite)	5	6	—	8	—	49	—	22	—	21
H (Grunerite)	5	7	—	11	2	61	—	1	—	26

*Relative intensity.

greater level of confidence in the reported concentrations for each type because of the large number of particles analyzed.

A summary of what might currently be termed "best available technology" (BAT) for the identification of particles in multicomponent samples is given in TABLE 2. Clearly, the identification of the mineral species belonging to each class is of paramount importance. Our procedures have been described elsewhere. They make use of several pieces of information, including matching of experimental data with calculated zone axis SAD parameters. As a result, the criteria for weighing the various data have been developed. These criteria are given below, and an example of the identification process is discussed using the data in TABLE 3.

Identification criteria:

1. If the x-ray spectrum is available, those matches not compatible with the spectrum are rejected.
2. If more than one diffraction pattern was recorded, the matches for the pattern with the largest d-spacing are taken as the most significant.
3. The match with the lowest R value in any pattern is regarded as the best fit.

TABLE 2
BEST AVAILABLE TECHNOLOGY PARTICULATE COUNTING

Record size, shape, composition and location of all particles.
Assign each particle to type category.
Record, measure, and interpret SAD patterns from randomly selected particles.
Estimate uncertainty in the identification of each particle type. Sources of uncertainty:
• Compositional interferences
• Crystallographic interferences
• Pattern quality.
Continue analysis until predetermined statistical criteria are satisfied.
• Variability in number per unit area
• Confidence level of concentration.

provided it does not disagree with the conditions specified in 1 and 2 (see Table 3).

4. The largest d-spacing observed is regarded as a measure of the unit cell size. Thus, if the largest d-spacing in two patterns was 3 Å, it is unlikely that the unit cell size is very large. This is reflected in the zone axis indices U, V, W.

If none of the preceding criteria suffice to distinguish between two minerals, the match with the lowest order zone axis is taken as the best fit. The application of these criteria eliminates most of the ambiguity in identifying mineral fragments. However, in those cases where none of the crystal structures provide a match to the measured

TABLE 3
MINERAL ZONE AXIS DIFFRACTION PATTERNS—MATCHING PATTERNS
FROM A REPRESENTATIVE TYPE CLASS E PARTICLE

Pattern 62398 Mineral	d ₁ -4.58 Zone Axis	d ₂ -3.64 (hkl)1	PHI-73.20 (hkl)2	Std Error*
Anthophyllite	438	201	340	0.003
Biotite	111	110	112	0.026
Clinochlorite	912	021	114	0.010
Clintonite	121	111	113	0.037
Minnesotaite	432	102	023	0.047
Penninite	912	021	114	0.010
Phlogopite	111	110	112	0.028
Prochlorite	912	021	114	0.011
Stilpnomelane	331	110	013	0.033
Talc	221	110	114	0.038
Xanthophyllite	111	110	112	0.027

Pattern 62399 Mineral	d ₁ -4.54 Zone Axis	d ₂ -4.19 (hkl)1	PHI-67.23 (hkl)2	Std. Error*
Biotite	112	110	021	0.020
Phlogopite	112	110	021	0.034
Stilpnomelane	112	110	111	0.029

X-ray Spectrum (Relative Intensity)								
	Na	Mg	Al	Si	K	Ca	Ti	Fe
Type Class E	—	10	24	40	9	1	1	14

$$*R = \sqrt{(\Delta d_1)^2 + (\Delta d_2)^2 + (\Delta \phi)^2}$$

parameters, where the x-ray spectrum and computer-generated "matches" are incompatible, or where two or more minerals have similar compositions and unit cells, a unique identification may not be possible.

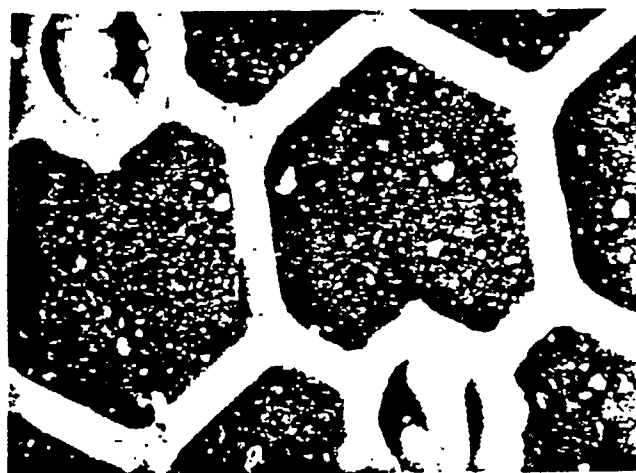
As shown in TABLE 3, a number of minerals match pattern 62398, whereas there are only three matches with pattern 62399. Of these, biotite has the smallest standard error (R) in both cases. Although the presence of titanium in the spectrum suggests the mineral is biotite, an unambiguous identification cannot be made because a large number of minerals exhibit pseudo-hexagonal diffraction patterns when lying on their basal plane. Thus, a positive identification could not be made. However, the possibility that it was an amphibole could be ruled out on the basis of pattern 62399 even though the first SAD pattern (62398) showed a very good fit with anthophyllite.

AUTOMATIC IMAGE ANALYSIS IN THE SCANNING ELECTRON MICROSCOPE

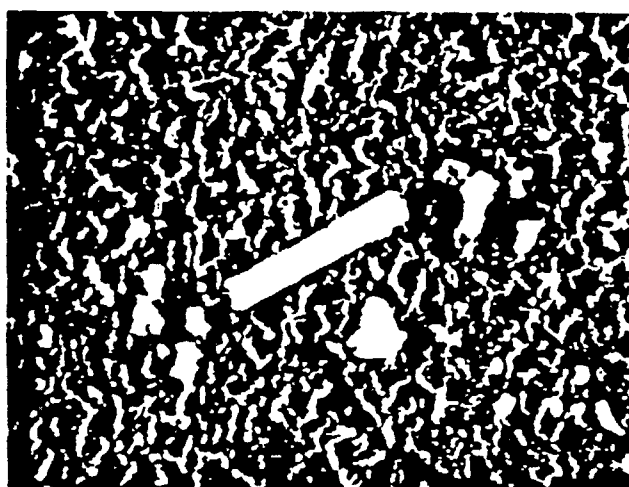
As emphasized earlier, complete characterization of particulate samples for health effects studies should include accurate determination of the distribution of particle size and aspect ratio. The same data will also be required when monitoring public and occupational exposure to any dusts that are found to be hazardous from laboratory studies. As discussed previously, photographic recording and manual measurement of individual particles is very time consuming but fortunately the advent of digital scan controls for the SEM has opened up the possibility of automatic measurement and recording and on-line data processing with a dedicated minicomputer. Progress in this approach and some as yet unresolved difficulties are discussed below.

The basis of automatic image analysis (AIA) is that the electron beam is step-scanned over the specimen surface in increments with a digital scan generator rather than an analog circuitry producing a continuous raster. The number of picture points per field of view is controlled by software and can be set for a matrix of 10×10 to 4096×4096 , depending on the resolution required for accurate measurement. To avoid an excessive expenditure of time on unnecessarily fine steps, the generator is programmed to switch automatically from a coarse search-mode matrix to a fine measurement-mode matrix when a particle is located by a change in detector signal intensity above (or below) a preset threshold level. When a particle is encountered the position of its centroid is determined as illustrated in FIGURE 2. From the centroid the beam sweeps through series of diagonals at either 15° or 45° intervals to determine and store the terminating coordinates of the maximum, minimum, and average traverse across the particle. Finally, the AIA-equipped SEM can be programmed to return the beam to the centroid; pause to accumulate an x-ray spectra, and assign the particle to a type class before continuing to search for another particle. The coordinates of the particle can be retained so the particle can be relocated to do an SAD analysis.

Although AIA-SEM generates the basic information required to characterize particles rapidly according to particle size, aspect ratio and chemical composition, the present state of instrumentation severely limits its use for this purpose. As discussed previously, the chemical compositions of many minerals are quite similar, and small differences may not show up above the statistics in the short counting intervals used. In addition, interference from nearby particles or size effects can result in an erroneous classification on the basis of chemistry alone. However, a more serious problem is achieving an acceptable compromise between the efficiency of the particle search mode and resolution of the measurement mode in relation to the SEM magnification selected. A magnification of about 500X is usually convenient as this results in a useful number of particles (~ 100) appearing in the field of view. However, at this magnification the spatial resolution is $\sim 0.07 \mu\text{m}$ which is comparable to the



(a) SE 400X



(b) SE 6000X

FIGURE 2. Example of AIA measurement of particle size, aspect ratio and centroid coordinates. The AIA trace (b) is at 2 $\times$ the SE micrograph.

diameter of the smaller particles. A 5X increase in magnification would solve the measurement problem but reduce the number of particles per field by 25X and negate much of the present advantage of AIA. However, appropriate hardware and software could be developed to use dual magnifications for scan and analysis analogous to the dual point grids now used for search and measurement. Other problem areas include

resolving crossed or overlapping fibers, and resolving 400 Å diameter fibrils in the SEM.

The essential point here is that instrumented methods of drastically reducing the large amount of time now required for particle counting are under development. These new advances in technique should be pursued since they will not only greatly reduce the high cost of air sample analysis but will also provide much more accurate and reliable data. Examples of preliminary data from fibrous and nonfibrous amphiboles (cummingtonite and amosite) are shown in the computer printouts reproduced in FIGURE 5.

FIBROUS AND COLUMNAR MINERALS

There are a number of factors that may be involved in the crystallization of either fibrous or nonfibrous varieties of a particular mineral. These include chemical composition of the parent magma or rock, cooling rate, and the magnitudes of any temperature gradient or hydrostatic or shear stresses that may be present. Apparently the right combination of these factors can result in extensive or highly localized growth of extremely long but very narrow crystals with rather weak bonding to adjacent crystals because of the occurrence of voids, large angle grain boundaries or faults, as in the case of crocidolite fibrils.^{11,12}

The possible equivalence of nonfibrous grunerite and grunerite asbestos (amosite) particles on a microscopic scale has been the subject of concern for some years.³ The pronounced tendency for comminuted amosite particles to lie on a (100) face has been noted by several authors.⁹ In contrast, nonfibrous grunerite has a tendency to lie near (110) faces, although all grunerite samples will have some particles with large (100) faces.

The observed orientations of the nonfibrous particles are readily explained on the basis of cleavage properties.^{4,6} However, the reason for the orientations of the fibrous grunerite (amosite) particles is not as apparent. We have mounted, polished, and chemically etched bulk samples of nonfibrous grunerite and amosite, both parallel and perpendicular to their c-axis. As seen in FIGURES 3 and 4, these sections are radically different. The nonfibrous grunerite in FIGURE 3 shows evidence of (110) cleavage traces, (100) parting and exsolution lamellae, as expected.

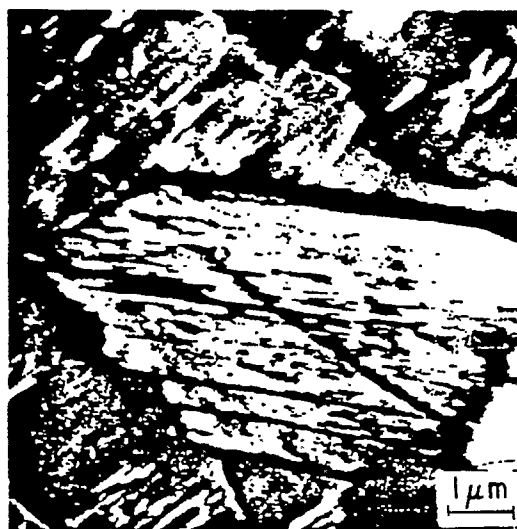
The amosite section taken perpendicular to the c-axis is comprised of weakly bonded bundles of fibers (Figure 4a), about 25 to 100 μm in diameter. When separated, as in Figure 4b, the individual fibrils have a rectangular cross-section with relative dimensions about 1 × 12 to 1 × 20. The long dimension of the cross-section varies from about .1 to .5 μm. Similar characteristics are observed in the longitudinal (parallel to the c-axis) sections illustrated in Figure 4c, d.

These data strongly suggest that the unit amosite fibril has a rectangular cross-section with a large (100) face. Thus, the single crystal SAD patterns obtained from amosite particles in the TEM result from one or more fibrils with common c-axis and common (100) faces. This interpretation leads to the conclusion that amosite fibrils and the densely twinned structure reported by others¹¹ are produced during crystallization. The resulting "whiskers" can withstand large stresses and elastic deformation without fracturing.

A natural consequence of these distinctions as seen in bulk samples is that very few cleavage fragments from ground or milled samples of non-fibrous grunerite have aspect ratios in excess of 10:1, whereas a large fraction of amosite fibers showed aspect ratios in excess of 10:1, as illustrated in the preliminary data presented in FIGURE 5.

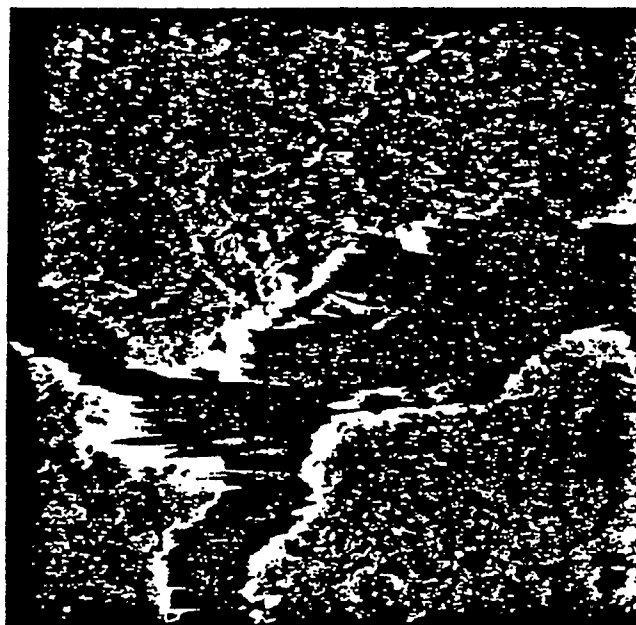


(a)



(b)

FIGURE 3. SEM micrographs of polished and chemically etched non-fibrous grunerite. Cleavage traces (110), parting, and hornblende exsolution lamellae (white) are evident in both (a) and (b).



(a)

4000X



(b)

4000X

FIGURE 4. SEM micrographs of polished and chemically etched grunerite asbestos. (a), (b): transverse section, perpendicular to the fiber axis.



(c)

400X



(d)

2000X

FIGURE 4. SEM micrographs of polished and chemically etched grunerite asbestos. (c), (d) Longitudinal section, parallel to the fiber axis

SAMPLE ID PENG E AMOSITE

WIDTH TO LENGTH RATIO DISTRIBUTION FOR ALL TYPES TYPE NO = -1

MOST PROBABLE= 7.50E-02 MEDIAN= 1.06E-01 AVG= 2.46E-01 SIGMA= 1.08E-01

CLASS	0	1	2	3	4	5	6	7	8	9	10
LIMIT	0	1	2	3	4	5	6	7	8	9	10
COUNT	0	0	0	0	0	0	0	0	0	0	0
%	0	0	0	0	0	0	0	0	0	0	0
5.00E-02	116	6.98E-01	20:1								
1.00E-01	277	16.68E-01	10:1			24%					
1.50E-01	269	16.20E-01	7:1			76%					
2.00E-01	231	13.91E-01	5:1								
2.50E-01	155	9.33E-01	4:1								
3.00E-01	116	6.98E-01	3:1								
3.50E-01	77	4.64E-01									
4.00E-01	111	6.68E-01									
4.50E-01	71	4.27E-01									
5.00E-01	58	3.49E-01									
5.50E-01	34	2.05E-01									
6.00E-01	53	3.19E-01									
6.50E-01	15	0.90E-01									
7.00E-01	29	1.75E-01									
7.50E-01	29	1.75E-01	1.3:1								

Most probable ratio ~13.3:1

SAMPLE ID: CUMMINGTONITE S. DAKOTA

WIDTH TO LENGTH RATIO DISTRIBUTION FOR ALL TYPES TYPE NO = -1

MOST PROBABLE= 4.75E-01 MEDIAN= 3.61E-01 AVG= 3.77E-01 SIGMA= 1.88E-01

CLASS	0	1	2	3	4	5	6	7	8	9	10
LIMIT	0	1	2	3	4	5	6	7	8	9	10
COUNT	0	0	0	0	0	0	0	0	0	0	0
%	0	0	0	0	0	0	0	0	0	0	0
5.00E-02	4	0.21E-01	20:1								
1.00E-01	73	3.79E-01	10:1			4%					
1.50E-01	147	7.63E-01	7:1			96%					
2.00E-01	184	9.55E-01	5:1								
2.50E-01	166	8.62E-01	4:1								
3.00E-01	175	9.09E-01	3:1								
3.50E-01	172	8.92E-01									
4.00E-01	189	9.81E-01									
4.50E-01	158	8.20E-01									
5.00E-01	212	11.06E-01	2:1								
5.50E-01	96	4.98E-01									
6.00E-01	112	5.82E-01									
6.50E-01	69	3.58E-01									
7.00E-01	67	3.48E-01									
7.50E-01	46	2.39E-01	1.3:1								

Most probable ratio ~2.1:1

FIGURE 5. Computer printouts of preliminary attempts to classify particles according to aspect ratio by automatic image analysis in the SEM. Comparison with direct measurements confirmed that the numerous very narrow fibers in amosite are not included in the analysis for the reasons discussed in the text resulting in a low median aspect ratio for this sample. Top: amosite; bottom: cummingtonite.

DISCUSSION

The methodology of air and water samples analysis has progressed a great deal in the past several years. It is possible to identify particles by electron microscopy and diffraction with a high degree of certainty, and using a type classification procedure the concentration of various mineral species present can be measured to a statistically deduced confidence level. Unfortunately these methods were not available several years ago when concern was first raised about possible health hazards posed by particulates in ambient air and water supplies so that much very uncertain data were

put into the public record. It is also unfortunate that accurate and reliable methods are time consuming and expensive so that they are not widely used even now that it is still difficult to reach agreements on the true identity and concentration of particulates.

It is of paramount importance that samples used for animal health effects studies be fully characterized so that some relationship with public and occupational exposure can be established in the future. Most previous health effects studies are of very limited value because of uncertainties about the actual particulate content of the test materials.

Aside from establishing reference levels for particulates in ambient air, the detailed data developed for the laboratory research on toxicity and carcinogenic activity would provide further clues as to the basic mechanism involved. Some believe that the very factors responsible for the usefulness of asbestos, e.g. fiber strength, flexibility, chemical inertness, and large aspect ratio are also to blame for their adverse health effects. Assessment of the characteristics of particles found within afflicted lung tissue support this opinion.

As pointed out by Zoltai, this viewpoint raises questions about the potential hazards of any mineral which, on rare occasion, crystallizes in an asbestiform habit and has essentially the same properties as asbestos, and particularly a very high aspect ratio of individual fibers. From the analysts' point of view the prospects for classifying particulates into fibrous >10:1 and nonfibrous <10:1 rapidly and inexpensively are very promising. Support for such a clearcut and realistic separation may come from health effects studies in the near future. The present 3:1 "definition" of a fiber does not make sense from mineralogical reasoning and can never be established from biological tests since preparation of test samples above and below this aspect ratio is not possible.

SUMMARY

Individual particles observed in air samples by electron microscopy can be identified with a high degree of certainty by recording accurate electron diffraction and x-ray spectrographic data on each particle for comparison with a computer file on all compounds that may be present. The procedure is time-consuming, but an acceptable level of accuracy can be obtained if positive identification is made of a small number of particles representative of type classes based on appearance and composition. Electron diffraction analysis has confirmed that lathlike particles of grunerite result from cleavage, whereas fibers of amosite result from their asbestiform growth habit, which is quite evident on scanning microscopy of bulk samples. Classification of particles according to aspect ratio by automatic image analysis (AIA) in the SEM shows promise as a rapid method of analyzing air samples.

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