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Differentiating Amphibole Asbestos from Non-Asbestos in a Complex Mineral Environment

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Key Words

Amphibole · Asbestos · Characteristics · Flowchart · Cleavage fragment

Abstract

Extending the TEM methods designed for the evaluation of atmospheres in which any primary mineral fibers present are derived from a commercial asbestos fiber is a challenging task. This is because the methods employed leave it to the expertise of the user to identify and evaluate interferences. Improper analysis of non-construction materials for asbestos content often results in the misidentification of non-asbestos amphibole particles as asbestos fibers. These errors have received widespread publicity in the media (such as the asbestos-in-crayons story) and have caused unwarranted reformulation of harmless products. The primary cause of these errors has been a poor understanding of mineralogy and analytical techniques among the many asbestos laboratories that arose following the passage of the 'Asbestos Hazardous Emergency Response Act' (AHERA) regulations. This study outlines a procedure based on published data that can be used to correctly classify a microscopic, elongated particle as either asbestos or non-asbestos.

Introduction

'Asbestos' is a commercial term applied to a group of naturally occurring minerals that have grown in a specific crystal habit and exhibit characteristics of flexibility, high tensile strength, large surface area, electrical resistance, and resistance to heat and chemical degradation. 'Asbestos' minerals are also capable of being manipulated, woven, or otherwise handled with minimal degradation of the fiber length. These minerals were originally defined by the characteristics of hand specimens and by their optical properties when examined using a polarized light microscope (PLM). The principal differences between commercial and non-commercial asbestos deposits are the size of the deposit and quality of fiber. The commercial fibers have been regulated as their health effects have become understood.

There are six minerals specifically regulated as asbestos by the Federal government, chrysotile (fibrous serpentine)

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and five varieties of amphibole fibers (anthophyllite asbestos, tremolite asbestos, actinolite asbestos, crocidolite, and amosite) [1]. Serpentine is a sheet silicate, whereas the amphiboles are double chain silicate minerals. The six asbestos minerals are only a few of the nearly 400 minerals that can grow in a fibrous, or 'asbestiform', habit [2].

Commercial deposits of these minerals occur worldwide, but large-scale exploitation has been limited to only a few countries. Chrysotile was, by far, the most economically important asbestos mineral mined or used in the United States. Historically, 95% of the consumption of asbestos has been chrysotile, with minor amounts of amosite and crocidolite [3]. Anthophyllite and tremolite were also used in very limited quantities for specialty products, but actinolite had almost no commercial value. Other mineral fibers, which did not exhibit the physical, chemical, and thermal characteristics described above, had little or no commercial value and were not considered to be asbestos.

The crystal habit of a mineral is the shape or form a crystal or aggregate of crystals takes during crystallization and is directly dependent on the environmental and geological conditions at the time of formation. The term 'asbestiform' is used to describe the unusual crystallization habit of minerals when the crystals form as thin, hair-like fibers, such as that which occurs with the six regulated asbestos minerals. The fibrous crystal habit is a less common form for amphiboles. The typical crystal habit of amphiboles is stubby prismatic. A prismatic crystal has one elongated dimension and two other dimensions that are approximately equal [4]. Cleavage refers to the preferential splitting of crystals along planes of structural weakness (cleavage planes) [5]. All monoclinic amphiboles have perfect (110) cleavage and orthorhombic amphiboles have perfect (210) cleavage [6]. Crushing or grinding of prismatic amphibole crystals may produce elongated particles that morphologically appear similar to asbestos, but do not possess the same unique physical properties.

Methodology

Application to Mineral Standards

The flowchart shown in Figure 1 was applied to a study of several mineral samples whose morphology could be described based on the appearance in a hand sample. These samples, described in Table 1, comprise a range of morphology from asbestos fibers to non-asbestos mineral particles. Three samples known to contain a mix of asbestos and non-asbestos particles were also included. Each sample was prepared for transmission electron microscope (TEM) analysis by suspending a small portion in a beaker containing deionized water. The suspension was allowed to settle for 1 min when an aliquot sample was removed and filtered through a polycarbonate filter. The filter was prepared and analyzed in accordance with published procedures [7].

Table 1 summarizes the classification of the particles using the flowchart shown in Figure 1. As shown by the data, samples that appear to be fibrous in a hand sample (Jamestown, Crocidolite, Amosite, North Carolina) show very high percentages of fibers classified as 'asbestos', while the non-asbestos sample (New York) indicates the population of fibers to be 'Non-asbestos'. The NIST tremolite sample (SRM 1867 a)[1] is a mixture of both fibrous and non-fibrous particles.

There is a small error rate with this procedure as shown by the data in Table 1. Though the New York tremolite is a sample of non-asbestos material, a small portion of the particles would be classified as 'asbestos' using this procedure. A closer examination at magnifications higher than those normally used in asbestos analyses and using a field emission scanning electron microscope [8] indicated these few particles are sheet-like structures, and are not asbestos fibers. In a similar manner, the Jamestown tremolite shows a relatively large population of non-asbestos particles even though this material was shown in injection studies to be very highly toxic [9]. A closer examination of these non-asbestos particles suggests that many of them are actually bundles of very fine fibrils that have a cementitious binder filling the interstitial pores, thus giving the particles the appearance of a massive crystal. These data suggest the overall error rate for this procedure is ≈5–10%. This indicates the classification procedure described in this study can be used to determine whether a particle is an asbestos fiber or a non-asbestos particle.

Discussion

Issues Related to the Definition of Asbestos

Issues related to the definition of asbestos have been argued in court beginning in 1974 with the Reserve Mining case[2] [10]. The debate was not related to mineral identification, but to the issue of whether the fibers were asbestos or non-asbestos. Nearly concurrent with the Reserve Mining case was the development of the optical microscopy method for counting airborne asbestos fibers. The method, phase contrast microscopy (PCM),

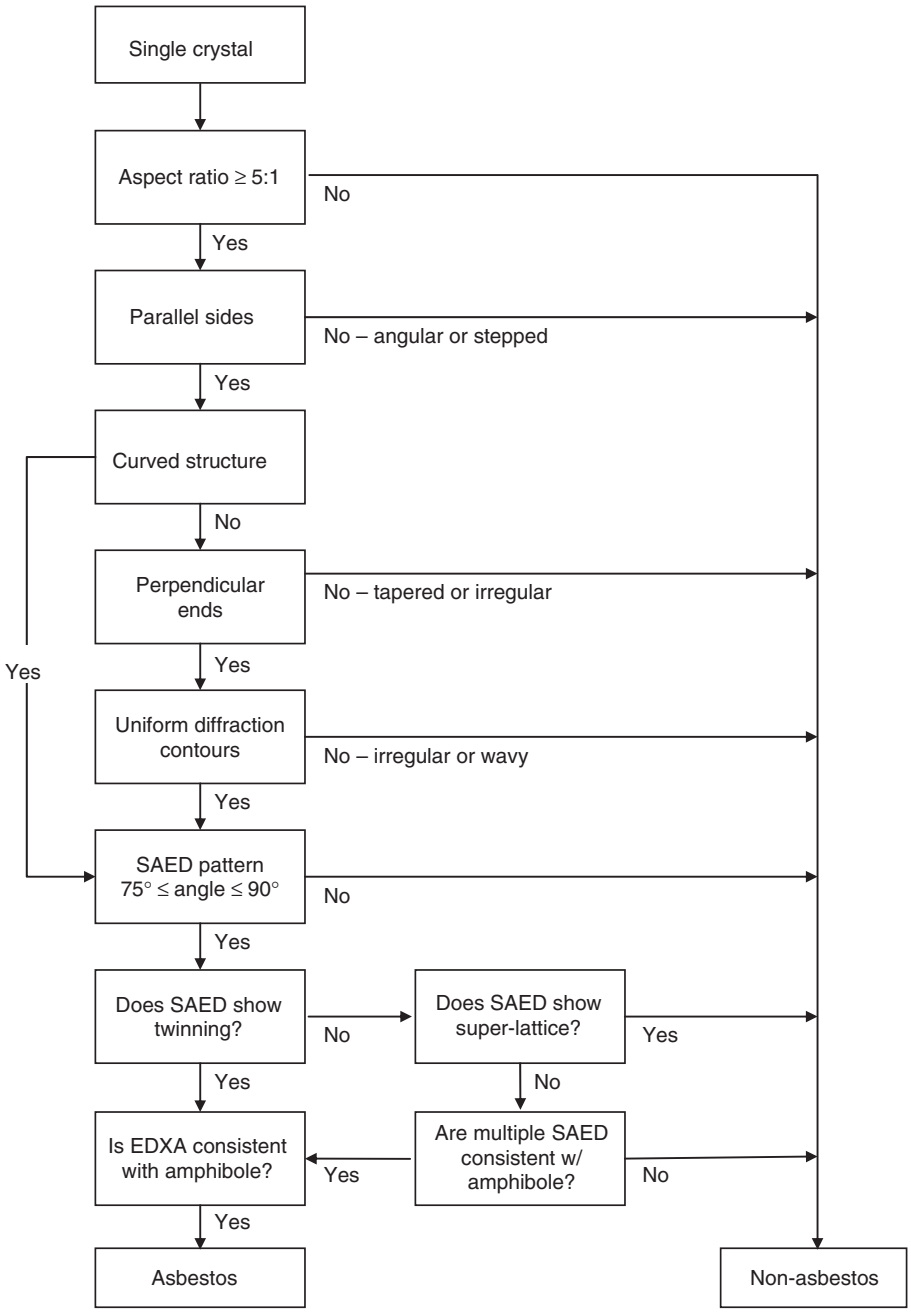

Fig. 1. Flow chart showing the various characteristics that can be used to determine if a particle is asbestos or non-asbestos.

Table 1. Example of application of the classification procedure to amphibole samples of known morphology. The data indicate a reasonable degree of accuracy in classification of the samples

		Classification (%)	
Mineral	Description of hand sample	Asbestos	Non-asbestos
Jamestown	Fibrous tremolite used in animal studies, moderate fiber length	70	30
Crocidolite	An asbestos amphibole taken from an ore sample, very long fibers	100	0
Amosite	Commercial product, aerosolized to obtain a respirable fraction	95	5
North Carolina	Fibrous tremolite with some, moderate fiber lengths	84	16
NIST SRM 1867a	Mixed tremolite fibers and non-fibrous tremolite particles	11	89
New York Tremolite	Tremolite ore sample, acicular appearance in hand sample	2	98

was an update of the midget impinger method that had been in use for nearly 50 years. The PCM method counted all visible fibers that were at least 5 μm long and incorporated the minimum 3:1 aspect ratio[3]. PCM was an easy, inexpensive method for the evaluation of airborne fibers in workplaces where commercial asbestos was in use. There was no interest in this environment to discriminate between asbestos and non-asbestos fibers; it was necessary to control the concentration of airborne fibers in order to minimize the disease incidence [11].

In the late 1970s, the U.S. Environmental Protection Agency (EPA) began the development of asbestos analytical techniques for evaluating the asbestos content of building products and to establish the asbestos fiber concentrations in the air and water [12]. The primary purpose of these methods was to evaluate materials, air, or water that either contained commercial asbestos or were impacted by commercial asbestos. The issue was not the evaluation of environmental samples to determine whether non-asbestos minerals were present, but whether the commercial asbestos in use in the workplace was affecting the environment. The use of a minimal aspect ratio (3:1) was maintained from the PCM method and became the basis for EPA regulations and methodology. Most mineralogy experts, however, thought it was inappropriate to define mineral fibers with a 3:1 aspect ratio [13,14].

There is some indication from the early 1980s that the EPA recognized that the amphibole minerals may be present in some products as contaminants, not as a deliberately added ingredient, and that these amphibole minerals may not be asbestos. They suggested that asbestos fibers had very high aspect ratios, but did not alter the methods to reflect this fact. In 1987, after a negotiated rulemaking, the EPA slightly increased the minimum aspect ratio to 5:1 in the 'Asbestos Hazardous Emergency Response Act' (AHERA) regulation [1]. Many experts on the AHERA committee had argued for a 10:1 or 20:1 minimum aspect ratio, but this was viewed as too great a change from the historical regulatory process.

The 1987 AHERA regulations caused tremendous growth in the asbestos analytical community. Hundreds of laboratories sprung up, seemingly overnight, to analyze the upsurge of samples from schools created by AHERA. Based on the history of asbestos usage, the vast majority of asbestos identified by these laboratories was chrysotile, though the accuracy of these determinations was not always what one would expect [15]. As part of the AHERA regulations, air samples are collected at the end of an abatement project to document that the clean-up of the area was acceptable. Since the abatement project involved removing a known asbestos-containing material in a controlled environment, the new laboratories and their microscopists erred on the side of caution by assuming the minimum aspect ratio (5:1) defined an elongated particle as asbestos. Since most abatement projects involved chrysotile, which has a scrolled sheet structure and is crystallographically different from amphibole fibers, this distinction was not important and the laboratories became very proficient at identifying chrysotile asbestos [16,17].

The issue of using an aspect ratio becomes more important when evaluating amphibole minerals. The Occupational Safety and Health Administration (OSHA), at the recommendation of the National Institute of Occupational Safety and Health (NIOSH), attempted to remove the distinction between the asbestiform minerals and their non-asbestos analogues, thus negating the need to differentiate between them. However, in 1992, after a court challenge, OSHA removed this distinction and has separately regulated the asbestos minerals as asbestos and their non-asbestos analogues as a nuisance dust. OSHA made this decision on the basis of epidemiologic studies that were either inconclusive or revealed no adverse health effect from non-asbestos minerals [18]. OSHA recognized the potential interference of non-asbestos amphiboles in the context of determining asbestos concentrations, but left it to the user to provide a viable method of discrimination between asbestos and non-asbestos minerals. Conversely, NIOSH has continued to argue for a regulation of non-asbestos minerals as asbestos [19]. Given the OSHA regulatory position, and the need in risk analysis to ensure that the physical properties of the measured population correspond to the properties of fibers with known risk profiles, there exists a need to reliably differentiate the asbestos amphibole fibers from the non-asbestos amphibole particles.

Misidentification of Amphibole Minerals

The amphibole mineral group contains a large number of species with such a wide variety of chemistries that no complete recognized classification system exists. In mixed mineral environments, a variety of different minerals form concurrently resulting in a complicated blend of fibers, fragments, and elongated rock fragments. These minerals may have similar particle shapes and chemical signatures, but greatly varying physical properties. The degree of scientific rigor necessary to correctly identify and quantify a specific mineral from a naturally occurring mixed-mineral environment requires a more stringent methodology. While analytical methods for asbestos analysis

clearly specify that only the asbestiform variety of the amphibole minerals are to be counted as asbestos, they do not provide unambiguous guidance on when the particle in question is not asbestiform. Combined with laboratories' limited experience with amphibole asbestos, this has created the impression that true non-asbestos, non-fibrous, prismatic amphibole particles are asbestos and, as a result, they are often counted as such.

In recent years, several events have occurred that illustrate these problems. As an example, several years ago, asbestos was reported in children's crayons [20,21]. A report was made by a laboratory or 'expert' that amphibole asbestos had been observed. Following this discovery of asbestos in crayons and subsequent national publicity, competent scientists from several laboratories examined the crayons and scientifically proved that there was no amphibole asbestos present, only non-asbestos cleavage fragments [22,23].

The EPA has recently concluded that very long, thin fibers have the highest potential for carcinogenicity. The basis for this revised consideration is a study by Berman and Crump [24], which showed that tumor generation is related to the concentration of fibers longer than 40 μm. This information was then incorporated into a new risk model in which the concentration of fibers longer than 10 μm and thinner than 0.4 μm was considered to be the most relevant. This risk model did not differentiate between asbestos and non-asbestos fibers, but did acknowledge that an aspect ratio of 20:1 or greater should eliminate most of the non-asbestos particles.

Determination of Asbestos Fibers

The definition of asbestos, one of the most widely published descriptions of any toxic material, suffers from a lack of precision at the microscopic level. The problem occurs for several reasons, though primarily due to the incorporation of an aspect ratio in the operational definition of asbestos in regulatory methods. All regulations clearly state that asbestos is being regulated and recognize that minerals occur in both asbestos and non-asbestos habits. The methods for asbestos analysis first require that asbestos be identified during an analysis and then determine if the asbestos fibers conform to the counting rules of the analytical protocol. The analytical procedures provide descriptions of asbestos characteristics ranging parallel sides [1,12] to information on diffraction characteristics of the minerals [1,7]. However, most analytical protocols also specify a minimum aspect ratio or length of the asbestos fiber to be considered for inclusion. As a result, the requirement that the fibers whose concentration is to be determined are the asbestiform variety of the amphibole minerals is generally lost in the practice of asbestos counting. In many laboratories the standard operating procedure is to identify as asbestos any particle that meets the aspect ratio specified in the method and is consistent with the chemistry of the regulated mineral, making the aspect ratio the de facto definition of asbestos. Many laboratories and industrial hygienists also employ a non-scientific theory, "If in doubt, count it," under the misguided assumption that false positives are less significant than false negatives. This was true in the era of high concentrations of asbestos, but is not when very low concentrations of environmental asbestos are being measured.

Other distinguishing characteristics of asbestos, such as parallel extinction in the PLM or the presence of internal diffraction contours in the TEM, are often not considered by various laboratories in determining whether a particle is asbestos before deciding if it meets the counting rules of a particular method.

Each asbestos analytical method has a definition of a 'fiber' and of 'asbestos', but no clear mineralogical definition of what is an asbestos fiber and what is an elongated non-asbestos particle [1,25]. A recent risk model makes an attempt to incorporate a width characteristic into the definition of asbestos by specifying only fibers thinner than 0.4 μm be considered for risk estimation [24]. Amphibole asbestos fibers should be defined on a combination of size, fiber width, chemistry, and diffraction characteristics, as well as aspect ratios.

Asbestos fibers normally exhibit anomalous optical properties that are distinctive. Asbestos fibers will exhibit parallel extinction (to the fiber axis) using PLM when viewed under crossed Nichols prisms. This is particularly true for anthophyllite, which is an orthorhombic mineral, but is also true for the monoclinic amphibole asbestos (crocidolite, amosite, and tremolite/actinolite). The explanation for the parallel extinction of the asbestiform of the monoclinic amphiboles is that they are "composed of many unit cells whose chain directions are parallel but differently rotated about (001)" [26]. It has also been suggested that these optical properties may be the result of stacking and twinning faults in the overall crystal structure [26]. For some amphiboles, the asbestos variety may exhibit extinction at angles that are non-zero but still are less than the non-asbestos variety for the same crystallographic orientation. The amphibole asbestos fibers may also show only two principal indices of

refraction when three are observed for the non-asbestos varieties of the mineral.

The unit cell values of the monoclinic amphiboles are generally: $a \approx 9.45$–10.0 Å, $b \approx 17.8$–18.45 Å, $c \approx 5.25$–5.35 Å, and $\beta \approx 105$–$109°$. For example, the unit cell of anthophyllite is: $a = 18.5$–18.6 Å, $b = 17.7$–18.1 Å, and $c = 5.27$–5.32 Å [27]. A selected area electron diffraction (SAED) pattern exhibiting a row spacing of around 5.3 Å has routinely been used by TEM protocols and microscopists to definitively conclude that the structure is amphibole and is asbestiform regardless of conflicting chemical data from energy dispersive X-ray (EDX) analysis and conflicting particle morphology. The 5.3 Å spacing is insufficient for determining mineral speciation. This spacing is not unique to amphiboles. The *c* parameter of the pyroxene unit cell is ≈5.2 Å [28]. The *a* parameter in talc is ≈5.28 Å [29]. The *a* unit cell of vermiculite and many of the micas is also ≈5.3 Å [30,31].

As part of an investigation into the amphibole mineral found in marble located at a quarry in Southdown, NJ, a TEM procedure (derived from well-known, published characteristics of single fiber amphibole minerals) was developed to differentiate between asbestos and non-asbestos fibers [32]. This procedure, accepted by EPA Region 2 for the Southdown project, is shown in Figure 1 and described below.

Fiber Width

Amphibole asbestos fibers grow very much longer than they grow wide [33]. Testing performed by Gibbs and Hwang [34] showed that 72% of amosite fibers from a mining operation and 58% from a bagging station were thinner than 0.3 μm. For similar operations at a crocidolite mine, 98 and 97% were thinner than 0.3 μm for mining and bagging, respectively. A related article reported median diameters for amosite of about 0.4 and 0.25 μm for crocidolite [35]. Other researchers have published similar information [36,37]. Contrasting with the width of asbestos amphiboles are the widths of known non-asbestos amphibole particles. Wylie et al. [14] reported the widths of known non-asbestos particles to be ≈1 μm or larger. Similarly, Wylie and Bailey [38] showed that the average width of non-asbestos airborne particles near an asbestos mine were slightly >1.2 μm.

Aspect Ratio

Various minimum levels of aspect ratio have been suggested, ranging from 3:1 [12] to 5:1 [1] to 20:1 or greater [13]. Wylie et al. [14] showed that the average aspect ratio of asbestos fibers is ≈8–10 times greater than that of non-asbestos particles. Kelse and Thompson [39] have shown how the different minimum aspect ratios would affect fiber count in mine samples by including (or excluding) various particles. They also showed that populations of asbestos fibers and non-asbestos particles have different aspect ratio distributions. ASTM [40] adopted a definition of asbestos based on a population of fibers longer than 5 μm, which was later adopted into EPA methodology for PLM, which suggests that asbestos fibers have aspect ratios greater than 20:1 or 100:1. At a minimum, the aspect ratio of asbestos fibers should exceed that of the analytical protocol currently in use.

Morphology

When examined under a light microscope, hand specimens of asbestos minerals have fibers that are easily separated and exhibit a 'polyfilamentous' characteristic. The 'polyfilamentous' characteristic is the single most important morphological characteristic of asbestos fibers and is a term that refers to bundles of long fibers that have grown when the unit cells form a chain-like structure. Many of the structures observed during analysis are single crystals and other morphological features must be evaluated to differentiate asbestiform from elongate cleavage fragments that are the result of parting.

Sheet structures, whether from the alteration of amphibole and pyroxene to a sheet silicate or not, often display ribbon-like structures. Ribbon structures commonly exhibit parallel sides, regular terminations, and flexibility. Some may even appear to have a bundle-like appearance due to sheet orientation relative to the electron beam. However, these structures commonly are very thin relative to their widths. The pores of the carbon replica on a TEM sample grid can clearly be seen through the width of the structure.

Parallel Sides

While seemingly the most obvious definition of a fiber, it is important that the fiber show parallel sides (Figure 2) [1,12,13,18,21,25,39,40]. Tapered or irregularly shaped sides indicate the particle is not asbestiform [7,9,41–49]. The sides should exhibit a smooth and nearly constant diameter along the length with no ledges. Cleavage fragments can show parallel sides as well, but their surface often has a ragged or irregular appearance.

Curvature

Apparent flexibility and curvature typically are perceived as indicative of asbestiform structures [50–54]. The absence of flexibility does not indicate that the structure

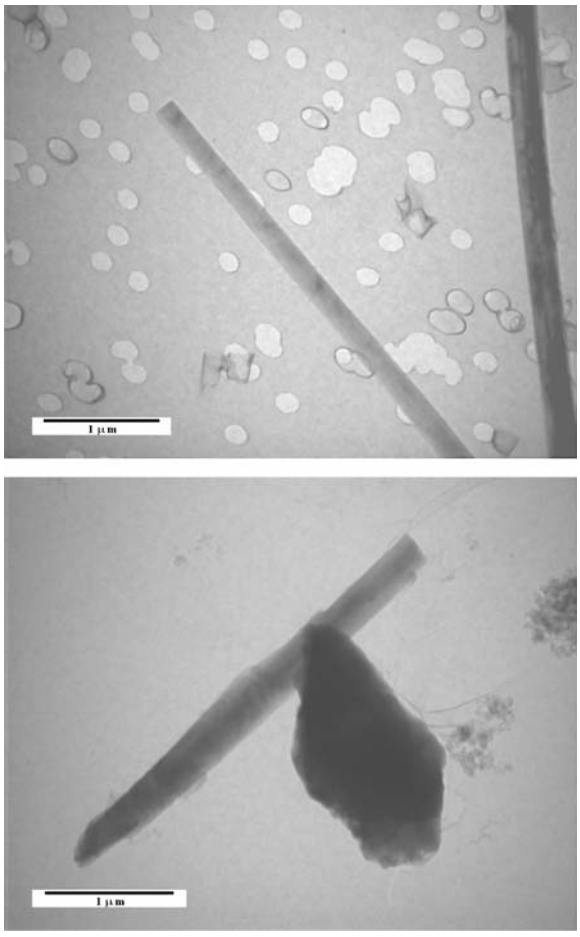

Fig. 2. Examples of particles with parallel (top) and nonparallel sides (bottom). Each particle meets the a minimum 3:1 aspect ratio, but only the top particle exhibits a hair-like appearance.

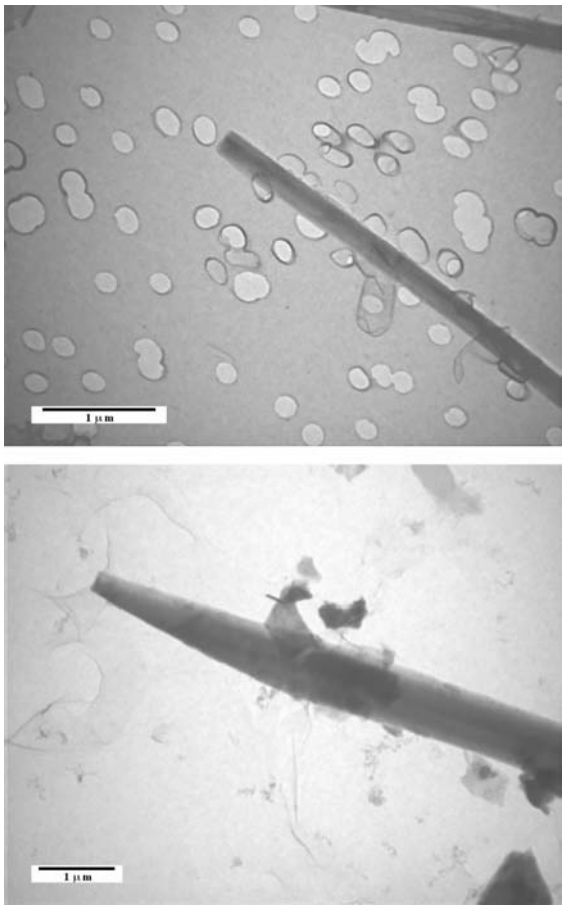

Fig. 3. Each particle exhibits a moderate aspect ratio, but only the top particle shows square terminations of the particle. The end of the bottom particle tapers toward a point, indicating the edge is a cleavage surface or parting plane.

is not fibrous. Delaminating sheet structures often show curvature as well and are not truly fibrous. However, most of these structures are eliminated on electron opacity.

Regular Termination

The ends of asbestos fibers are not ledged, pointed, or tapering acicular, but show regular, square termination (Figure 3) [22,49,55,56]. Occasionally, the orientation of the structure relative to the electron beam may make the termination appear to be angled relative to the sides, but in those cases both ends will be parallel to each other.

Internal Diffraction Contours

When observing the amphibole minerals during a TEM analysis, contours due to the internal diffraction by the electron beam can be observed. These contours are crystallographic shear planes that are referred to as Wadsley defects (Figure 4) [22,49,55–60]. Generally, these shear planes will occur on the (010) crystal face [26,55,61]. Crawford [61] has suggested that the Wadsley defects also occur in the (110) plane and result from non-stoichiometric stacking of the crystal. In the asbestos amphiboles, these defects occur with some regularity, giving rise to regular diffraction contour patterns within the mineral.

SAED Pattern Exhibits a Phi Angle of 74° to 90° Between d_1 and d_2

Of greater use than a row spacing of 5.3 Å for differentiating potential amphibole structures from non-amphiboles is the angle, phi or Φ, between the vector along the rows (d_1) and the vector between rows (d_2). d_1 and d_2 are the d-spacings which are the interplanar distances between repeating rows of reflection spots and phi is the angle between d_1 and d_2. d_1, d_2 and phi define the hkl plane in the diffracting crystal. This angle is less sensitive to inaccuracy in calibration than

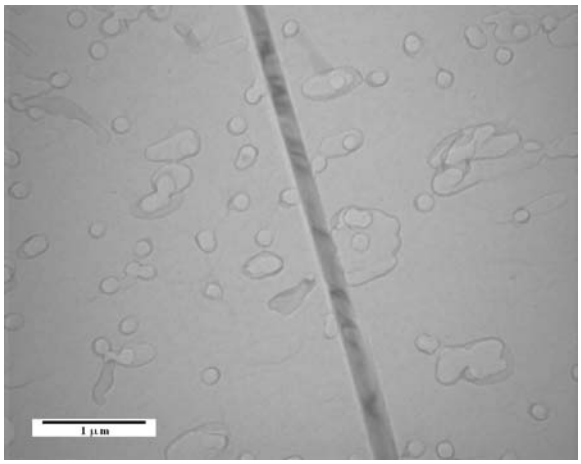

Fig. 4. This fiber is an example of one that has internal diffraction contours (Wadsley defects).

lattice measurements. In addition, unique zones that differentiate single and double chain silicates from each other will occur between 74 and 90°. Zones with a phi angle <74° in the amphibole and pyroxene produce (h,k,l)s that could easily have multiple interpretations, particularly when allowing for high measurement errors. Multiple SAEDs on the same particle are needed for definitive identification when the structure is particularly thick or when orientation relative to beam, a grid bar, or matrix interferes.

Twinning vs. Superposition of Amphibole-Sheet Silicate Lattice Indicative of Phase Alteration

Twinning is commonly used to indicate the fibrous nature of an asbestiform structure. However, the lack of twinning does not indicate that a structure is not fibrous. Some amphibole fibers are more likely to twin than others. Twins are also observed in prismatic crystal habits as well [33,55,58]. Easily confused for twinning is the SAED pattern that results from the alteration of double and single chain silicates to sheet silicates. Although similar in appearance, twinning patterns are the result of two or more mirror or reciprocal twinned crystals. The resulting pattern is closely spaced reflection spots along one or more layer lines. The intensity of the spots may or may not vary in some orderly arrangement [59]. In amphiboles, the measured d-spacing of the twinned layer line will be a d-spacing measurement higher than the *a*-spacing reported for amphiboles. An SAED of an alteration phase has a similar appearance. However, the measured d-spacing will not exceed the *a*-spacing of the unit cell. The reflection spot will show an orderly pattern of bright reflection spots and faint reflection spots. The bright spots show the position of the shared lattice sites. It is also common that an extra set of spots may be present in one layer line from the center spot. In some alteration phases, the faint spots in the SAED may have an ellipse or smeared pattern. The exact cause of this is not yet fully understood. It may be due to disorder in the lattice or offset of the sheets (Allison K, Van Orden D, Lee RJ, Unpublished data).

EDX Consistent with Amphibole

During the TEM analysis of amphibole particles, EDX is used to determine the chemical fingerprint of the particle. EDX is perhaps the most subjective of the diagnostic tools available to electron microscopy. Exact results can be dramatically affected by the quality of the detector, collection time, orientation of the particle relative to the detector, orientation relative to the grid bars, orientation relative to other particulate, and particle thickness. It is imperative that not only the detector be maintained in top condition, but that the unknowns are compared to standard material collected on the same detector in the same time period on particles of comparable thickness and orientation.

The nomenclature of amphiboles has been defined by Leake et al. [62]. Applying the Leake rules to EDX results provides one clue to the identification of the particle. However, the Leake nomenclature applies only to amphiboles so it does not differentiate amphiboles from non-amphiboles. Unknowns must be evaluated with other comparative nomenclature for non-amphibole mineral phases such as that done by Morimoto [63] for pyroxenes.

SAED and EDX Confirm Mineral Identification

As noted by most analytical procedures, there are numerous minerals that have similar chemistries to the regulated amphiboles, such as talc and pyroxene. Owing to the similarity of the chemistry of the regulated amphibole and other minerals, it is necessary, at a minimum, to examine the SAED pattern for the mineral particle. Ideally, the pattern should be recorded and matched to published diffraction data.

The size of the unit cells in mineral speciation is useful when resolving a zone unique to the mineral group. For example in the monoclinic pyroxenes, the unit cell values are $a \approx 9.6–9.8$ Å, $b \approx 8.9–9.0$ Å, $c \approx 5.22–5.25$ Å, and $\beta \approx 105–109°$ [28]. It is necessary to tilt the structure relative to the electron beam into a zone that will produce a unique zone axis. The practice of using zones produced at zero degree tilt is insufficient for mineral identification in a mixed mineral sample.

Owing to relative orientation of the crystal to the electron beam it may be difficult in all cases to tilt into a decisively unique zone and it may be necessary to use multiple zones clearly indicative of the mineral species [7,41,42]. In the differentiation between similar amphibole phases, it may also be necessary to imprint the unknown SAED pattern with an internal standard such as gold or platinum. Using SAED to differentiate asbestiform from prismatic particles of the same mineral phase requires an understanding of the crystallography of both and preferential position of each relative to the electron beam. Although there is a higher probability that certain zones are more likely to be present in asbestiform structures and other zones are more likely to present in elongate cleavage fragments, there are no absolutes.

Conclusions

A more rigorous scientific standard and test TEM methodology is needed when evaluating samples for asbestos that contains a complex assemblage of mineral phases. As a result of the increasing disturbance of amphibole containing rock formations by developers, and bans emerging on products containing asbestos at even trace levels, standardized procedures are needed for determining low levels of asbestos in raw materials, as well as soils and sediments. These procedures are needed to certify materials for import or use in which asbestos fibers may be present as a result of naturally occurring contamination of a material.

The method described in this study is based on published and well known characteristics of asbestos and non-asbestos particles. Unlike asbestos, non-asbestiform minerals grow in three dimensions to produce the non-fibrous (massive) form of the same mineral. When non-asbestos minerals are crushed, fragments are cleaved away from the main crystal mass, a process that produces 'cleavage fragments'. The massive minerals will tend to fracture along sets of systematic planes within the mineral crystal and some long thin fragments may result, although the majority of the fragments will be short, non-fibrous particles. These cleavage fragments may have a similar microscopic appearance to that of true asbestos fibers. Distinguishing characteristics, such as size, optical extinction characteristics, and morphology, can be used to segregate the asbestos from the non-asbestos in the hand sample and by optical microscopy. However, there has not been a well-defined method for discriminating between individual asbestos and non-asbestos particles in the TEM.

This study outlines a systematic procedure for characterizing amphibole particles in both commercial asbestos samples, and in mixed mineral samples. The method relies on previously published characteristics of asbestos and non-asbestos minerals, and has been independently peer-reviewed previously [32]. The results of the application of the procedure to a variety of samples produced results consistent with the known physical characteristics of the materials. The apparent error rate is small, $<10\%$ of the particles in the commercial asbestos samples (crocidolite and amosite) were identified as non-asbestos particles, and $<10\%$ of the particles in the known non-asbestos sample were identified as asbestos particles. The proportion of asbestos and non-asbestos particles in the mixed mineral samples, (Jamestown, and North Carolina, and NIST) were consistent with the macroscopically observed characteristics, and with the proportions of asbestiform fibers identified by PLM.

This method can be easily implemented in analytical asbestos laboratories. As it relies on standard TEM evaluations, the procedure offers a cost-effective approach to standardizing the classification of amphibole particles. The primary limitations of the method are in the characterization of thicker (0.5 μm diameter) acicular mineral particles with high aspect ratios, in determining whether or not apparent fibers protruding from larger masses are actually independent fibers or part of the larger mineral fragment, and in the characterization of mineral fragments that are very thin compared to their width or length. The limitation in these situations stems from the two-dimensional nature of the TEM image, resulting in false positive results. The particles were not formed as bundles of thin readily separable particles. In these cases, stereo microscopy using the field emission scanning electron microscope allows the evaluation of the particle morphology in three dimensions to supplement the two-dimensional TEM analysis [8].

Notes

1. The National Institute of Standards and Technology (NIST) certifies and provides Standard Reference Materials (SRM) for laboratory instrument calibration and laboratory accuracy measurement.
2. Trial of the Reserve Mining Co. over dumping of waste rock into Lake Superior at Silver Bay. It was alleged the waste contained mineral fibers.
3. This defines what are today called WHO fibers: >5 μm long, <3 μm wide, and with an aspect ratio $\geq 3{:}1$.

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