

## TEM Quantification of Amphibole in Asbestos Containing Materials: A Summary of Data 20 Years in the Making

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The use of the minerals commonly referred to as "asbestos" has a lengthy history dating back to the turn of the first century, if not earlier [1]. More modern uses beginning with a surge at the end of the First World War involved implementation in a variety of building materials, industrial products, and consumer products. Over 90% of the asbestos that was used for these products utilized the specific asbestos type chrysotile, a member of the serpentine mineral family. Whereas the *amphibole* class of asbestos minerals include tremolite, actinolite, anthophyllite, crocidolite, and amosite.

While all forms of asbestos are known to be carcinogens, toxicological profiles suggest that mixed exposures to multiple types of fibers may be critical to disease causation [2]. Because of this and other assertions that suggest the amphibole minerals are more toxic than the chrysotile variety of asbestos an emphasis on the investigation of materials for amphibole specifically has been an important research aspect. It has also been well established that many chrysotile mines from which the long fiber was obtained were also host to amphibole minerals tremolite and actinolite [3]. A method was developed to look for these amphiboles in chrysotile ore [4] and subsequently modified to investigate finished products containing chrysotile for the presence of amphibole [5]. These techniques have also been incorporated into an international method for the detection of asbestos in bulk samples [6].

The method involves a matrix reduction procedure in which a product is ashed to remove any organic components; treated with acid to remove carbonates and other acid soluble components; then ultimately treated with alkaline solvent to eliminate the chrysotile itself. The residual material would include only particles that are resistant to heat, acid, and alkaline environments such as amphibole asbestos. The residual material is suspended in filtered deionized water and prepared for examination by transmission electron microscopy (TEM) using standard asbestos TEM preparation methods [5, 6]. The residue was examined by TEM at magnifications of approximately 20,000 times, supplemented by the use of selected area electron diffraction and energy dispersive x-ray spectroscopy.

The use of these techniques to detect asbestos in the residue has allowed for the determination of concentrations of low levels of amphibole fiber that might be present in a given sample. In the past 20 years, over 100 different products have been analyzed including asbestos containing samples of building materials (joint compounds, floor tiles, cement shingles, etc), automotive products (brakes and clutches), industrial products (like gaskets, packing, fabrics, electrical components), and consumer products (asbestos insulated appliances for example). What this research has shown is that many chrysotile containing material also contain trace (<1%) amounts of tremolite, actinolite, or both.

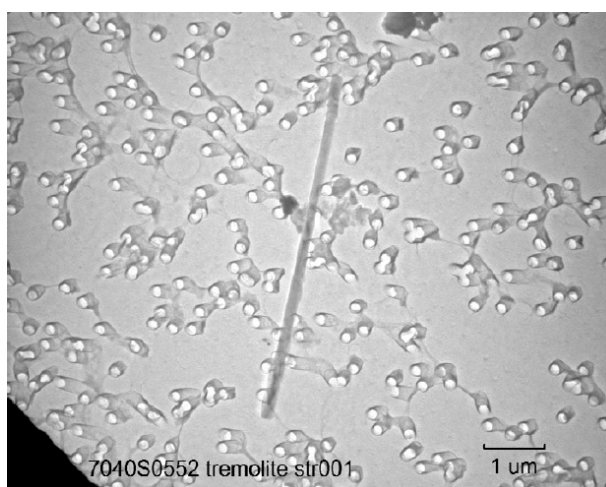
Amphibole fibers were detected in over 90% of the chrysotile-rich samples investigated with an average concentration of approximately 0.03% by weight and ranging from approximately 0.00005% to 0.5%. Anything less than 1% is often referred to as "trace"; however, trace levels can still correspond to millions of fibers per gram of material due to the microscopic widths of these mineral fibers. A subset of these samples, taken exclusively from friction materials, have been carefully cataloged to determine

the range and average statistics for individual amphibole fibers observed during this 20 years of analyses. In these friction samples, asbestiform bundles as well as individual fibers were detected. Fiber lengths, widths, and aspect ratios (length/width) vary from sample to sample, but do follow certain trends.

The observed fiber lengths for individual fibers ranged from 0.5 micrometers (the minimum reportable length) to over 23 micrometers, with an average length of 3.2 micrometers. Reported fiber widths ranged from 0.04 to 1.9 micrometers, with an average width of approximately 0.3 micrometers. Fiber aspect ratios range from 5 (the minimum reported aspect ratio) to over 90 (i.e. fibers 90 times longer than they are wide). With an average width of 0.3 micrometers, many of these fibers are below the limits of resolution for optical microscopy techniques more commonly used for asbestos identification, like PCM and PLM. This means that many of the particles in this population of amphibole fibers would not be reported by most PLM investigations of bulk materials or of PCM/TEM investigations of air samples that focus only on fibers greater than 0.25 micrometers in width [7].

#### References:

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- [4] J. Addison and L.S.T. Davies, *Ann. Occup. Hyg.* **34** (1990), p. 159.
- [5] Millette J.R. *et al*, *Microscope*, 57(1):19-22, 2009.
- [6] ISO 22262-2:2014. Air quality – Bulk materials – Part 2: Quantitative determination of asbestos by gravimetric and microscopical methods. Geneva, Switzerland.
- [7] Dr. James Millette is thanked for his leading role in conducting many of the studies referenced herein.



**Figure 1.** TEM micrograph of a tremolite fiber observed after matrix reduction of a chrysotile-containing friction material.