

Laboratory Report

PLAINTIFF'S
EXHIBIT
WRG-738

Raw Materials - Others

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Vermiculite

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W.R. Grace Co.

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Laboratory

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MICROSCOPIC EXAMINATION OF FOUR SAMPLES OF
W.R. GRACE CO. EXPANDED VERMICULITE

Environmental Control-Product Safety (EC-PS) submitted four samples of W.R. Grace Co. expanded vermiculite for the purpose of identifying fibers which had been observed by the U.S. Gypsum laboratory. The EC-PS group was particularly interested whether or not the fibers might contain any asbestiform serpentine or amphibole minerals. The four samples were designated as 1) Sulphur Springs-fine, 2) Sulphur Springs-coarse, 3) Troy-No. 2, and 4) Troy-No. 3.

These four materials were first examined by binocular stereomicroscope, then representative examples of the detected fibers were selected for examination under the petrographic microscope, and in the scanning electron microscope (SEM).

Binocular Microscope Examination

Samples of each of the vermiculites were removed from their sample bags by means of a spatula. The materials were passed through a 70 mesh sieve, and the -70 M fraction was examined under the microscope. The fiber search was largely confined to this fraction because the U.S. Gypsum SEM study had shown that the fibers were quite small in cross-section.

A trace amount of white fiber bundles was detected in each of the four samples. The appearance and distribution of the fiber bundles is shown in Figure 1. The length of the bundles ranges from approximately 0.3 to 2.0 mm, but the most common bundle length is 1.0-1.5 mm. An outstanding physical characteristic of the bundles is that they may be easily broken into individual acicular fibers of greater than 5:1 (length: width) aspect ratio by applying pressure with a steel needle probe. The amount of such fiber bundles in each sample of vermiculite is certainly less than 1% by volume, and is probably less than 0.1-0.2%.

Other particles whose aspect ratio is greater than 5:1 are present in each sample, but these cannot be broken into asbestiform fibers. These are probably vermiculite, biotite, non-fibrous amphibole, and other mineral phases.

Petrographic Microscope Examination

Fiber bundles were removed from the binocular microscope samples with steel needle and/or forceps, and placed upon glass slides for examination under the petrographic microscope. All the fiber bundles appear to be the same mineral phase. One of the prismatic refractive indices is approximately 1.63, and the birefringence is in the range of 0.01-0.02. Most important is the non-parallel extinction of the individual fibers. The Δc angle ranges from 15-25°. This data indicates that the fibers are not the most common asbestiform mineral, chrysotile.

The fiber bundles have the optical properties of the asbestiform amphiboles (i.e. tremolite-actinolite). Other elongate, but not fibrous grains were also examined under the petrographic microscope, and these appear to be similar to the hornblende (i.e. amphibole) group.

Scanning Electron Microscope and Energy Dispersive Spectrometry (SEM/EDS)

Since the petrographic microscope examination showed that the fiber bundles from all four samples were the same, detail SEM/EDS work was only done upon material from the Sulphur Springs-fine sample. Figure 2 is a typical fiber bundle, at moderate magnification. At higher magnification, the ends of the bundles have the "splintered wood" form shown in Figures 3 and 4. Then, when one separates the bundle with a steel needle probe and forceps, the individual fibers have the appearance shown in Figure 5.

For comparison purposes, a known sample of asbestiform tremolite was examined. The microstructure of this material is shown in Figures 6 and 7. The optical properties of the known tremolite fibers are similar to those of fibers from the white fiber bundles.

The final point of investigation leading to the identification of the fiber bundles was EDS analysis of individual fibers. The first step in this process was to make EDS spectra of known asbestiform tremolite and known massive (i.e. non-fibrous) tremolite. These spectra are Figures 8 and 9. Figures 10 and 11 are EDS spectra of individual fibers in the bundles displayed in Figures 2-5. The similarity in the chemical compositions of the fiber bundles in the vermiculite and the known tremolites adds to the weight of evidence that the fiber bundles are asbestiform tremolite.

Conclusions and Discussion

The results of the stereomicroscopic, petrographic microscopic, and the SEM/EDS examination of white fiber bundles (0.3-2.00 mm long), which are present in trace quantities in the four samples of expanded vermiculite, suggests that the fiber bundles are asbestiform tremolite-actinolite, $\text{Ca}_2(\text{Mg, Fe}^{++})_5\text{Si}_8\text{O}_{22}(\text{OH, F})_2$.

The chemical composition of the fiber bundles (by EDS) is in good agreement with data for tremolite fibers published by Dement (1). The microscopic fiber bundles are similar in appearance to a macroscopic photograph of fibrous tremolite from Tuscany, Italy presented by Haartz and Lange (2).

The occurrence of tremolite-actinolite with vermiculite has been reported by Buie and Stewart (3) in the Enoree, So. Carolina vermiculite deposits. These workers suggest that the chemical alteration (hydrothermal and weathering) of amphibole and biolite produces vermiculite.

The point is that the presence of asbestiform amphibole probably indicates an area of the vermiculite deposit where the vermiculite-forming reactions are not complete. One could then expect vermiculite materials mined from different areas of such deposits to contain more or less of the asbestiform amphibole. This may be the reason why W.R. Grace vermiculite customers have noticed variations in the amounts of asbestiform material in shipments received at different times.

In regard to the potential health hazard of the tremolite-actinolite fiber bundles, it must be remembered that the bundles must be physical crushed, abraded, or pulled apart in order to produce respirable fibers, i.e. the asbestiform fibers are not loosely bound as in the reference tremolite material (compare Figures 6 and 7 to Figure 2). Perhaps the mechanical handling of the vermiculite at Sulphur Springs or Troy could destroy some of these fiber bundles, but it would certainly be necessary to take air samples of the work area to establish whether or not the limits of "safe" concentration had been exceeded.

R.P. Stevens:jes

RPS



Figure 1:

The distribution of asbestiform tremolite-actinolite bundles (Sulphur Springs-fines sample) in a field of vermiculite flakes is shown. The particles indicated by (X)s are identifiable asbestiform bundles. 18X.

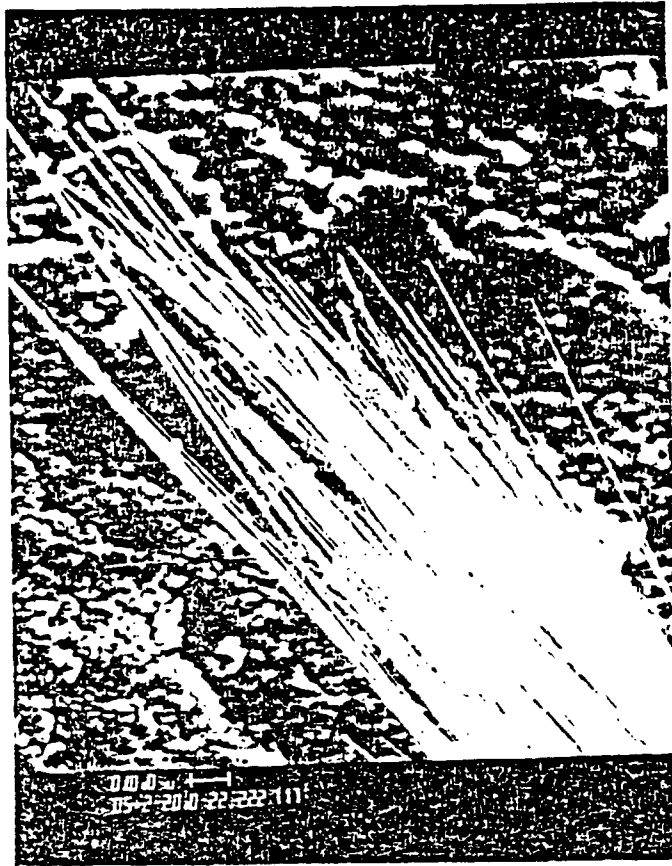


Figure 2:

The SEM photograph shows the end of a tremolite-actinolite fiber bundle. The broom-like bundle can be mechanical broken into individual asbestiform fibers. 500X.

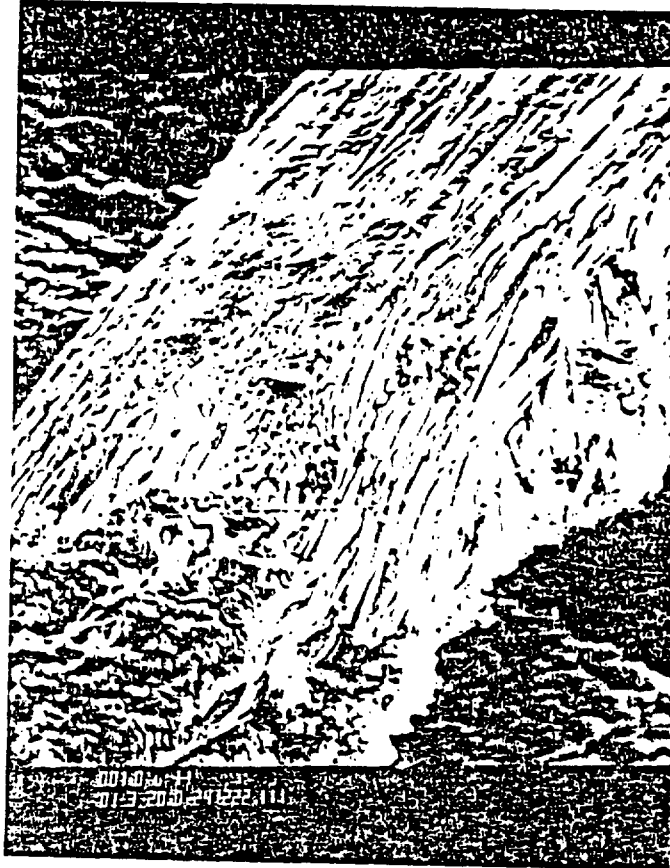


Figure 3:

The tip of a fiber bundle is shown. Some of the individual fibers are less than 1 micron across. 1000X.

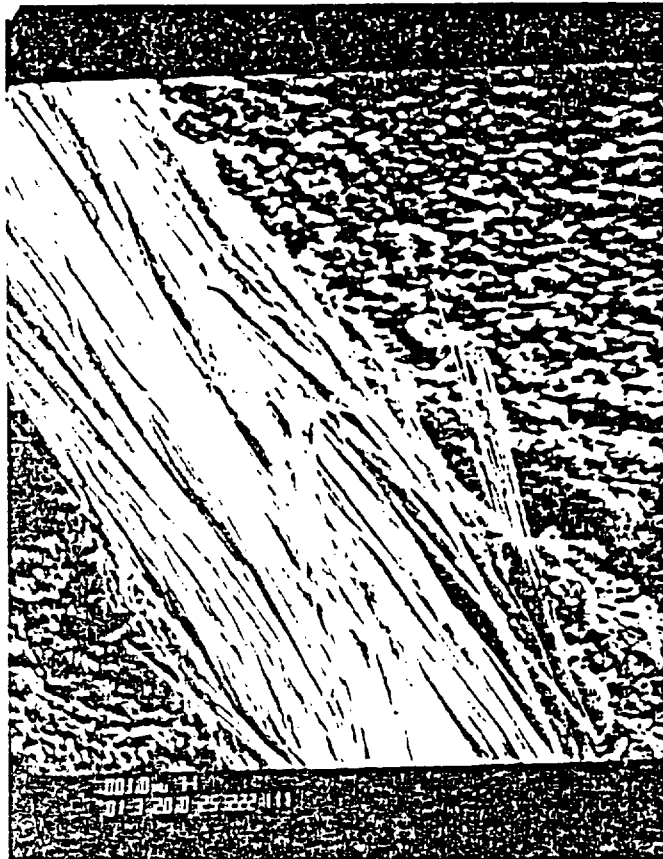


Figure 4:

Some of the bundle tips have a definite "splintered wood" structure. Again, individual fibers of less than 1 micron diameter can be seen. 1000X.



Figure 5:

The separated asbestiform fibers range from 0.3 to 2 microns in diameter, and bear a strong resemblance to known asbestiform tremolite (see Figure 7). 2000X.



Figure 6:

This moderate power SEM photograph of known asbestiform tremolite shows much looser "packing" than the fiber bundles in the vermiculite (see Figure 2). 400X.



Figure 7:

At higher power the known tremolite fibers appear to be morphologically similar to the fibers separated from one of the bundles (see Figure 5). 1000X.

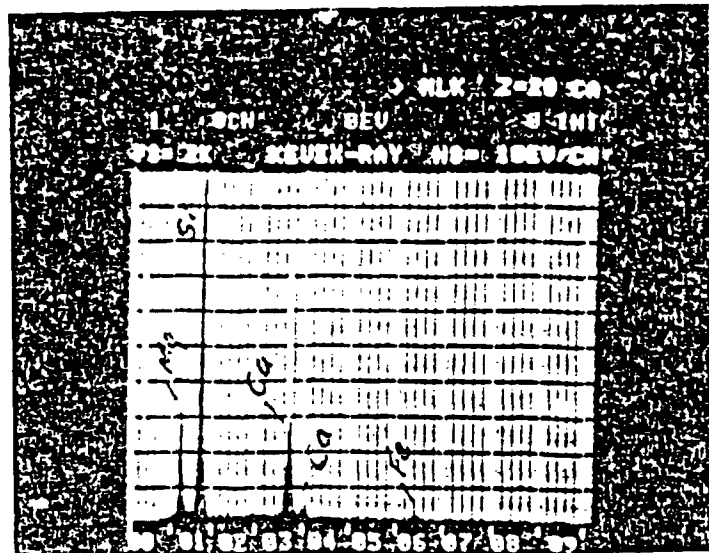


Figure 8:

The EDS spectrum of a known asbestiform tremolite single fiber is shown. The major amounts of SiO_2 , MgO , and CaO and the trace amount of FeO are typical for tremolite.

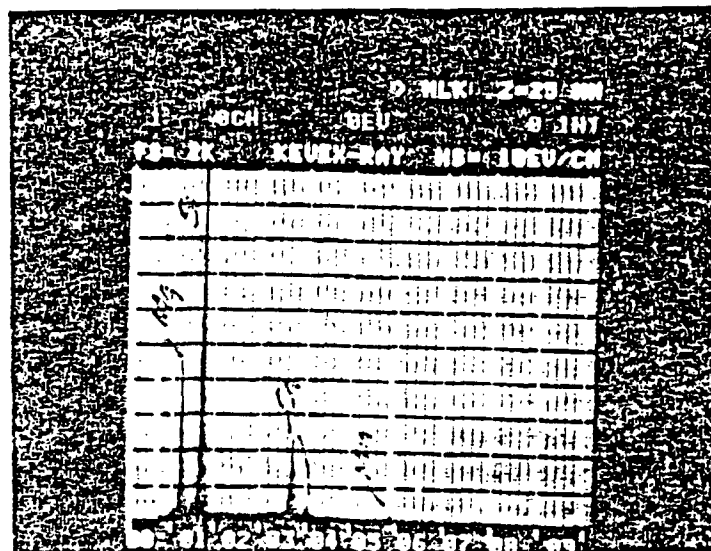


Figure 9:

The EDS spectrum of known massive (i.e. non-fibrous) tremolite grain shows the same major elements as the asbestiform variety, but in this case a trace of MnO is present.

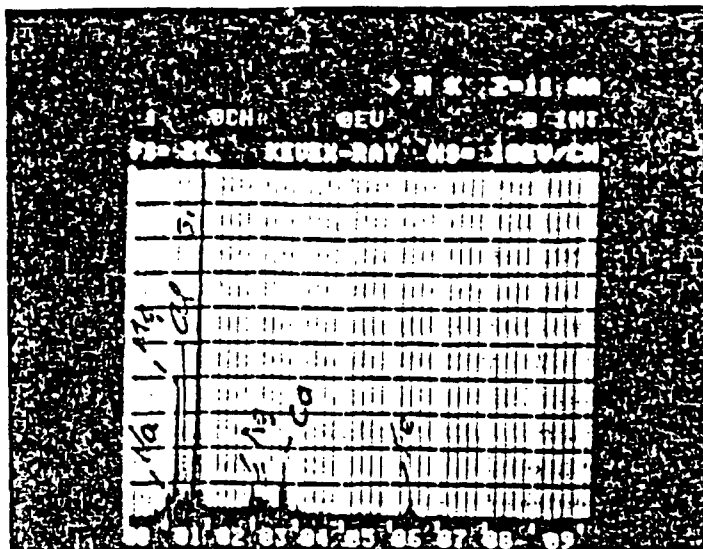


Figure 10:

The EDS spectrum of a fibers in a fiber bundle (Figures 2-5) are similar to those of known tremolite, but the smaller amount of CaO, and the presence of Na_2O , Al_2O_3 , and more FeO suggests that the fibers are asbestiform tremolite-actinolite solid solution modified by the presence of some alkali-bearing amphibole. The Ag peaks are from material used to cement the fibers to the SEM stub.

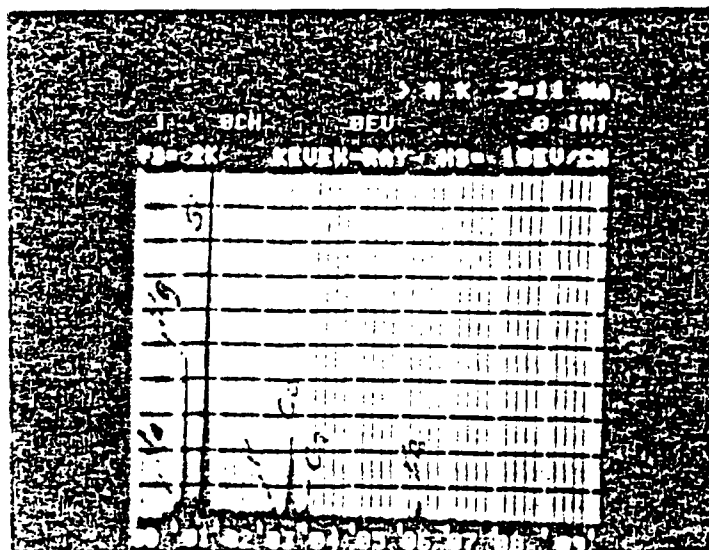


Figure 11:

This EDS spectrum of a fiber similar to those shown in Figure 5 shows that the unknown fibers are compositionally quite similar to known asbestiform tremolite (see Figure 8).

REFERENCES

- 1) John M. Dement, "Asbestiform Minerals in Industrial Talc", NBS Special Pub. 506, Workshop on Asbestos: Definitions and Measurement Methods, p. 318.
- 2) J.C. Haartz and B.A. Lange, "Selection and Characterization of Fibrous and Non-fibrous Amphiboles for Analytical Methods Development", Op. Cit., p. 298.
- 3) B.F. Buie and O.F. Stewart, "The Origin of the Vermiculite at Tigerville, So. Car., (abstract) G.S.A. Bulletin, Vol. 65, No. 12 (1356-57).