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TECHNICAL NOTES

Tile Analysis using PLM

by Jim Richards

Simply put, vinyl asbestos floor tile and floor covering can not be analyzed by PLM for the absence of asbestos. The method is completely incapable of detecting the presence of asbestos fibers of the size that were commonly used in floor tile. The only suitable analysis is by electron microscopy.

Vinyl Asbestos floor covering used asbestos as a filler[1]. Fillers are used in plastic products primarily to lower cost. The only advantages in using fillers are opacity, ultraviolet light resistance, electrical properties, gloss control dent resistance, increased hardness and some minor processing improvements. .Physical properties of plastic compounds are usually impaired by the use of fillers. Furthermore, they are increasingly harder to process and increase wear on processing equipment. The most undesirable effect is lower tensile strength (pulling apart) , and compression (squeezing together) strength and poorer chemical resistance.

Calcined clays and water ground and precipitated calcium carbonates with a particle size of less than 3 micrometers (microns) as the most commonly used fillers in flooring materials. Asbestos is used in tile and sheet flooring with clays. When vinyl asbestos floor tiles were made calcined clay sold for 1.5 cents per pound and asbestos sold for 4 cents per pound. The vinyl polymer itself cost 25 cents per pound. Therefore high filler loading was desirable to reduce cost. The limiting factor was the compromise of strength process speed, and gloss. The following is a typical vinyl tile formulation[2]:

Copolymer (Vinyl Acetate/Vinyl Chloride PVC)	19.5%
Asbestos	27.3%
Calcium Carbonate	39.1%
Di2ethyl-hexyl-phthlate plasticizer	7.8%
Epoxy plasticizer	1.2%
Titanium Dioxide	3.9%
Penta-erythritol	0.8%
Heat Stabilizer (usually Ba, Mg, Zn derivatives)	0.4%

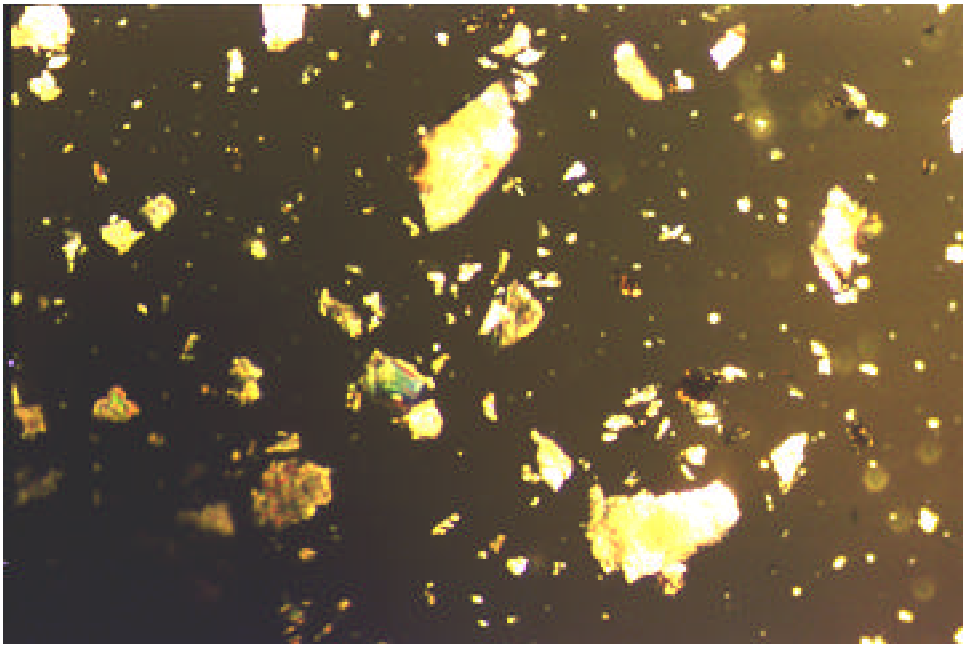
The observation of asbestos fibers in a Polarized Light Microscopy is limited to fibers that are at least 5 microns in length and have a width of at least 1 micron[4]. Fibers must also be large enough to identify using either dispersion staining or Becke line techniques[3].

To illustrate the problem, a well characterized sample was used. For this report we used sample #2 a vinyl asbestos floor tile from NIST NVLAP Bulk Proficiency Testing for Round M1 1998, March of 1998 .

NIST NVLAP stated the following in the report of the results of testing "....Chrysotile fiber bundles are not evident in examination of edges of the broken tile. They are also difficult to detect in the residue after ashing and acid dissolution. The binder masks the fibers in the untreated sample and the titanium oxide pigment effectively coats the fibers in the residue after gravimetric reduction. While the fibers isolated from the residue are especially short, they exhibit typical chrysotile morphology and optical properties....".

It should be noted that of the 311 laboratories which received these samples 64 failed to report chrysotile. This means 21% of the laboratories could not identify chrysotile in a NVLAP proficiency round sample floor tile. One has to assume these laboratories made a more than nominal effort with this NVLAP proficiency sample because it is necessary to identify the presence or absence of asbestos in each test sample in order to pass the round. It is necessary for the laboratories to pass the round to maintain their accreditation. It is interesting to note that NIST NVLAP did not evaluate any of the participating laboratories optical properties for this sample nor did they provide the optical properties that they observed and measured for the asbestos fibers in this tile sample.

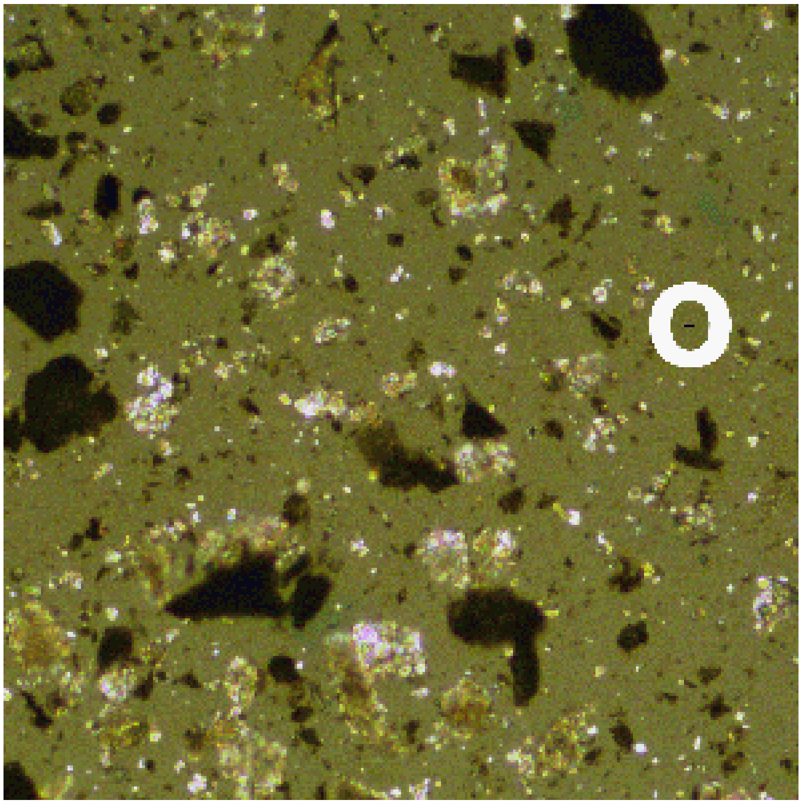
The following micrograph is of the NIST tile after pretreatment by heat ashing in a furnace at 450 degrees celsius for 15 hours (Gravimetry per EPA Method 600/R-93/116, July 1993). The sample was lightly crushed using a glass rod after it cooled. The resulting powder was placed on a microscope slide in 1.550 high dispersion refractive index oil and placed under the Polarized Light Microscope. It is obvious, even to the casual observer, that there are no fibrous structures.



NIST NVLAP M1 1998 Floor Tile filler after gravimetry viewed in the PLM (CSDS)

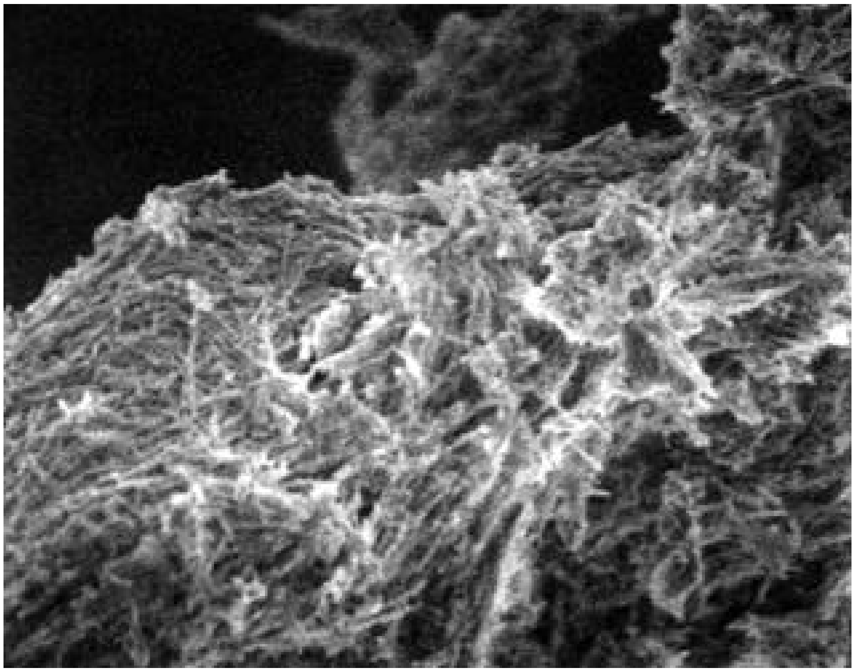
Furthermore in the following photograph of a floor tile matrix which is more than 50% chrysotile asbestos an artificial fiber exactly 10 microns by 1 micron was drawn in at 100% black. It is circled so that you can find the fiber. As explained above, the optical properties of this fiber would be very difficult to measure. Fibers narrower than one half micron will be invisible[4]. The large lumps in the micrograph are huge masses of sinuous fibers that are therefore arranged in random order which results in the inability to obtain meaningful optical properties. There are fibers in the North/South orientation as well as the East/West and ever direction in between. Fibers are also oriented randomly in the z axis as well further complicating the dispersed light. This three dimension mix of random fibers causes all optical data to be lost. Furthermore, there is titanium dioxide mixed in with the fibers that makes some of the particles opaque.

As previously mentioned t the fiber that was added to the picture is 100% black against a background that is 17% black. There is therefore a high optical contrast between the fiber and the background. This would not be true for a real chrysotile fiber as the refractive index of the fiber (1.545 - 1.559) is very close to that of the oil which is 1.550. As a result the fiber would have far less contrast than in this example photograph.



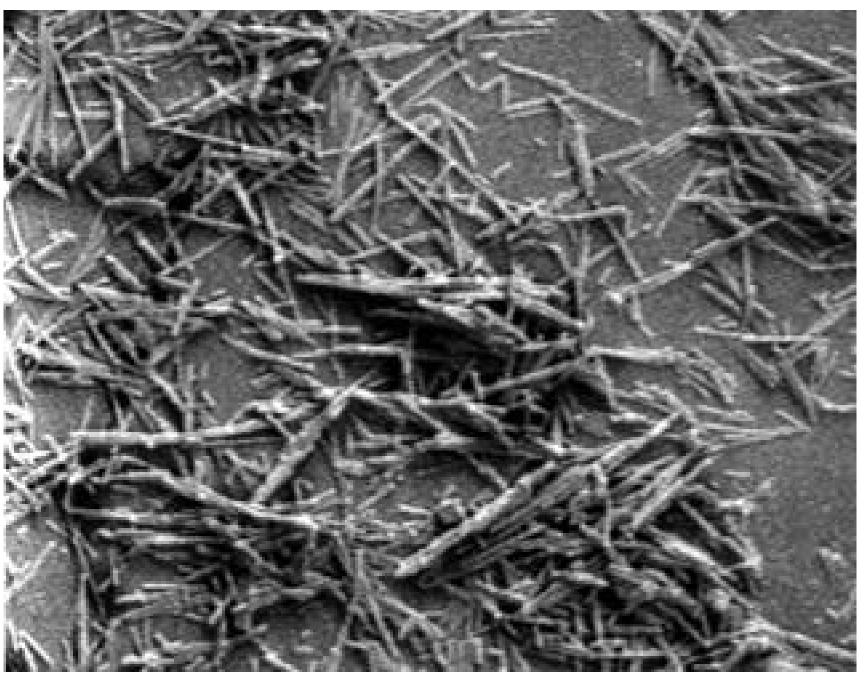
Optical micrograph of floor tile matrix with artificial 10 micron fiber drawn in.

How do we know that these particles are in fact sinuous lumps of fibers? We turn to the scanning electron microscope for the answer. The scanning electron microscope has the resolving power to see particles as small as 0.025 microns (micrometers) which is 20 times smaller than the optical microscope. In the following micrograph we can examine these large lumps. Observation shows they are in fact sinuous lumps of chrysotile fibers and titanium dioxide particles. Notice how the fibers are orientated in almost every direction. These particles are also quite thick and therefore the light would pass through hundreds of particles giving the dispersion for a large number of randomly positioned particles rather than for one discrete crystal. This results in meaningless data.

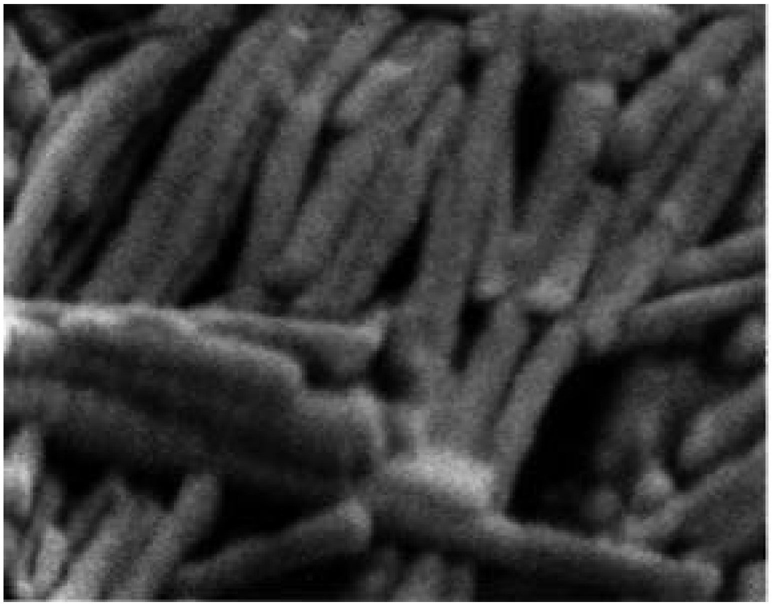


SEM Micrograph of large bundle in NIST NVLAP M1 1998 floor tile

Occasionally there are huge bundles of asbestos fibers that are arranged in essentially one direction. These bundles do offer optical properties that demonstrate that they are chrysotile asbestos. That is why some tile samples do show positive results. In the micrograph below we see single fibers which are randomly arranged.



In the following micrograph the magnification is 100,000X and shows the tubular nature of the asbestos fibers. Note that the fibers continue to show random orientation. There are fibers in the north-south direction as well as east-west direction and northwest-southeast direction, etc.



It should be noted that 9X9 floor tiles were generally made by a different process than 12X12 floor tile and sheet vinyl products[1] The 12X12 tiles are a newer style and has therefore benefited from improved processing technology. As mentioned before, the mechanical properties of tile is affected by the filler concentration and type. In most cases the strength is reduced. It was discovered that very small particle size actually improves mechanical properties at low loadings and certainly reduces the degradation effects significantly at high loadings. Therefore the newer 12X12 floor tile and sheet vinyl tend to have shorter asbestos fibers. Thus, 12X12 are more difficult to analyze (because of the short fibers) so they are more often found to be falsely negative than 9x9 floor tiles.

Point counting of a tile or sheet vinyl flooring material is useless simply because the Polarized Light Microscope can not reliably resolve fibers this small. Therefore, if on the occasion that there was a bundle of these short fibers all aligned in the same direction then being large enough to be observed in the PLM and not made opaque by other fillers; one would then have a very unrealistic low estimation of the concentration of asbestos in the tile.

There seems to be an almost religious belief in point counting even when the laws of the physical universe do not support this belief. It reminds this author of Bertrand Russell's remarks ..."The most savage controversies are those about matters as to which there is no good evidence either way. Persecution is used in theology, not in arithmetic[5]."

References:

1 PolyVinyl Chloride, Harold A. Sarvetnick, van Nostrand Reinhold, New York, 1969, LCCCN 69-18948, page 108

2 The Stabilization of PolyVinyl Chloride, Fernand Chevassus, Translated from the French by C. John R. Eichhorn and Esteban E. Sarmiento, St. Martin's Press, Inc., New York. 1963 (LCCCN 63-19023) page 303.

3 EPA 600/M4-82-020, December 1982. (40 CRF Part 763, Appendix A to Subpart F)

4 Optics and Optical Instruments, B.K. Johnson, Dover Publications, New York, ISBN 0-486-60642-2, page 89

5 "Unpopular Essays, An Outline of Intellectual Rubbish", Bertrand Russell.