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Detection of Chrysotile Asbestos in Airborne Dust from Thermosetting Resin Grinding

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ABSTRACT: Airborne dust samples generated during grinding thermosetting resin plaques containing 0.8 to 18% chrysotile and reference samples were examined optically and on the electron microprobe up to magnifications of $\times 900$.

Chrysotile in dust samples was analyzed by new methods using iodine in solution or as a vapor for selectively staining chrysotile and increasing its visibility and by the NIOSH phase contrast method. No free fibers of chrysotile were detected in dust collected from 0.8% chrysotile-polyester plaques. Only a low-level concentration was found in dust from 4-18% chrysotile-polyester plaques. The difference in texture of chrysotile and cellulose ester membrane (CEM) filters was emphasized by vacuum deposition of aluminum and carbon.

The importance of care in handling chrysotile-bearing CEM filters and the improvement in retention with carbon coating are described.

Electron microprobe methods are presented that distinguish between chrysotile completely encapsulated in resin and chrysotile with a free surface. The airborne chrysotile in the grinding dust was usually nonfibrous, encapsulated in resin, and closely associated with other materials. In the samples analyzed, the number of chrysotile fibers with a free surface varied from zero to two per thousand dust particles.

KEY WORDS: thermosetting resins, serpentine, iodine, chrysotile, light (visible radiation), phase contrast, resin-encapsulation, vacuum deposition, electron microprobe

At the present time, the amount of fibrous asbestos in airborne dust collected on membrane filters² is generally determined by the phase contrast method developed by Edwards and Lynch [1], hereafter designated the NIOSH (National Institute of Occupational Safety and Health) phase contrast method. The most evident defect in this procedure is the failure to discriminate between various kinds of fibers. Crossmon [2] and Julian et al [3] identified each of the six asbestos-type minerals with dispersion staining, an optical method of producing color that depends on variations in refractive index of the mounting medium and the constituent being identified with different wavelengths of light. Thus, its application is limited. This paper describes methods that rapidly detect free chrysotile asbestos in dust samples.

Reference samples prepared from blends of known materials and from dust samples produced by grinding chrysotile-bearing thermosetting resins, polyester, and epoxy were collected by settling in water and on membrane filters. These samples were

examined optically and on the electron microprobe. We used new procedures for selectively staining chrysotile and for emphasizing the difference in texture of the chrysotile and the filters by vacuum deposition of carbon. The retentive power of membrane filters for chrysotile was investigated. An electron microprobe method for distinguishing between resin-encapsulated and free chrysotile and for determining the percentage of free chrysotile particles in dust samples was developed.

Sample Preparation and Description

Resin plaques containing 0.8 to 18% chrysotile were ground with a power-driven hand grinder equipped with a 7-in. (177.8-mm) diameter, 18-grit abrasive disk simulating the fabrication operations found in boat yards and the automobile industry. Compositions of the plaques are listed in Table 1. The grinding was carried out in a thoroughly cleaned, closed, 8½ by 8½ by 8-ft (2.6 by 2.6 by 2.4-m) room. The plaque, approximately ½-in. (12.7-mm) thick by 1 by 2 ft (0.3 by 0.6 m), was clamped to a bench 30 in. (76.2 mm) from the floor approximately in the center of the room. The edge was ground for a period of 4-5 min with the grinder rotating in a direction to throw the heavy particles toward the floor.

Four petri dishes filled with distilled water with a total surface area of 425 cm² were used to collect samples of dust as it settled from the air during each grinding operation. Another set was collected over a 2-h period after each grind. Three of the sample dishes in each set were placed in the corners of the room, two at the 5-ft (1.5-m) level and one at the 2-ft (0.6-m) level and the fourth was located on top of the bench, 3 ft (0.9 m) from the grinding area. Each set was combined into one sample and the residues concentrated by evaporation at room temperature.

In addition, individual samples were collected before, during,

TABLE 1—Compositions of chrysotile-bearing plaques.

Sample No.	Resin		Chrysotile		Fiber Glass	Silica	Limestone
	Type	Weight, %	Type*	Weight, %	Weight, %	Weight, %	Weight, %
1	polyester	79.2	RG-244	0.8	20.0	...	...
2	polyester	99.2	RG-244	0.8	...	...	...
3	polyester	98.0	RG-244	2.0	...	...	...
4	polyester	96.0	RG-244	4.0	...	...	...
5	epoxy	76.0	RG-144	4.0	20.0	...	...
6	polyester	35.0	RG-110	5.0	10.0	10.0	40.0
7	polyester	35.0	RG-110	10.0	10.0	10.0	35.0
8	polyester	30.0	SG-200	18.0	...	2.0	50.0

¹ Senior scientist, Metals Div.; consultant, Metals Div.; air monitoring engineer, Caudria Asbestos; and technology manager, Caudria Asbestos, respectively, Union Carbide Corp., Niagara Falls, N.Y.

² Millipore Filter Corp., Bedford, Mass.; cellulose ester membrane filters.

* Types RG-110 and RG-144 are opened fibers; SG-200, pelletized; RG-244, silica coated (Union Carbide Corporation designations of refined California chrysotile commercial products).

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and after each grinding operation by aspirating at the rate of 2 litres per min through Type AA membrane filters (0.8 μ m pore size).

Sections of the plaques were polished on both sides to less than 0.25 mm in thickness and examined with transmitted ordinary and polarized light. The chrysotile was present as fibrous particles frequently associated with strain in the resin matrix.

A low magnification ($\times 100$) examination of concentrated dust samples showed the samples contained not only the material from the plaques but also materials such as fibers of cotton, wood, nylon, and glass (Fig. 1). The presence of extraneous airborne materials emphasizes the importance of distinguishing chrysotile from other fibrous materials.

Equipment Description

The optical equipment used in the transmitted ordinary and polarized light studies consisted of a Leitz petrographic microscope, Kohler-type illumination with a tungsten light source, $\times 45$ [0.65 numerical aperture (N.A.)] and $\times 90$ (1.32 N.A.) objectives and a positive, $\times 10$ eyepiece. The electron microprobe analyzer was an Acton model no. MS-64.

Staining Methods

Morton and Baker [4] reported that a 1% solution of iodine in glycerine selectively stains chrysotile, serpentine, brucite, and, to a lesser extent, hydromagnesite. The same authors also reported that other organic solvents such as ethyl alcohol, carbon tetrachloride, butyl Carbitol,[®] and tricresyl phosphate may be used. This stain differentiates chrysotile fiber from all other fibrous materials including organic and inorganic fibers and depends on the activity of the iodine and the hydrated magnesia surface of the chrysotile. (Chrysotile is composed of a layer of magnesium hydroxide on a silica substrate.)

Mixtures of polyester, epoxy, mineral wool, calcite, sisal, or any combination, with 0.5% chrysotile and dust samples were treated with sublimed iodine and iodine dissolved in various solvents and were examined with transmitted light at $\times 450$ and $\times 900$. Of these materials only chrysotile was stained a

characteristic orange color and the staining appreciably improved its visibility. However, some solutions were superior. Figures 2-4 show the colors developed by the chrysotile after being stained by various methods. Optical observations, staining solutions, and the new method for staining with sublimed iodine are given below.

Solution 1

Solution 1 (1 g iodine + 100 cm³ glycerine) is an orange-brown color and stains chrysotile a pale orange; polyester, pale yellow; and epoxy, dark red-brown. (No photomicrographs are shown.) Before staining, polyester and epoxy are transparent and without color. Color changes in the other materials were not noted. The stained chrysotile was easily distinguished in the water-collected, air-dried samples.

Solution 2

Iodine is appreciably more soluble in a solution of ethyl alcohol and glycerine than in pure glycerine. Solution 2 (2.5 g iodine + 50 cm³ ethyl alcohol + 50 cm³ glycerine) produced darker colors than Solution 1, resulting in chrysotile being more easily detected (Fig. 2).

Solution 3

The orange-stained chrysotile is easily noted in Solution 3 (1 g iodine + 100 cm³ benzene) because of its contrast with the pale violet color of the liquid (Fig. 3). Iodine is violet in nonbasic solvents such as benzene and orange or brown in basic solvents such as ethyl alcohol and ethyl alcohol plus glycerine. After the solution was placed on the specimen, a drop of Crown immersion oil was added to decrease the evaporation rate of the benzene. Small fibers of chrysotile were detected that would probably have been overlooked if stained with Solution 1 or 2. Other materials were also examined (sisal, cotton, wood, synthetics, and glass), and only the epoxy and polyester were stained.

Chrysotile is rapidly stained in any of the three solutions; however, about $\frac{1}{2}$ h is required for the epoxy and at least 6 h for the polyester to develop the indicated colors: dark red-brown



FIG. 1—Dust collected in water after grinding 0.8% chrysotile-polyester plaque: (left) ordinary light and (right) polarized light.

and yellow, respectively. One week later the specimens were reexamined. The characteristic color of the stained chrysotile, polyester, and epoxy intensified with time.

Solution 3 was also carefully applied to sections of membrane filters containing reference and grinding dust samples. The filter was placed on Crown immersion oil to make it transparent and examined with transmitted light. Although Crown immersion oil was used throughout this investigation, other types of immersion oil should be equally effective. The NIOSH standard phthalate-oxalate-membrane solution could not be used since it bleaches the stain.

Sublimed Iodine

Chrysotile reference standards and air-dried dust samples were placed in covered containers with iodine. On standing at room temperature, the iodine sublimed and stained the free chrysotile orange, and after several hours the chrysotile inside the polyester resin was also stained. For example, Fig. 4 shows orange-colored chrysotile fibers inside a pale yellow polyester particle. The color of the chrysotile faded on standing at room temperature (<1 h), particularly when it was not encapsulated in resin. This was avoided by mounting the stained sample in immersion oil and using a cover glass.

Chrysotile deposited on membrane filters was also stained by exposure to the iodine atmosphere. Although the chrysotile stained in 20 min, an exposure time of about an hour produced optimum contrast between the chrysotile and the filter. After staining, the filter was placed on one to two drops of immersion oil and covered with a cover glass and the chrysotile identified. Stained chrysotile is also more easily detected with phase contrast microscopy than unstained.

Microscopic Analyses

The water-collected dust samples previously described were concentrated by evaporation. Five slides of each sample stained with Solution 3 were examined at $\times 450$ with transmitted ordinary and polarized light. A field typical of the dust from a 10% chrysotile-bearing sample as it appears with parallel polars

is shown in Fig. 5. This field contains sisal, fibrous glass, resin, and calcite but not chrysotile.

The chrysotile in the dust samples was generally in the form of agglomerated masses of fibers rather than fibers or bundles of fibers; however, it was easily distinguished from the other phases by the orange stain. The agglomerated nonfibrous-shaped chrysotile particles are probably generated by the chopping or shredding of bundles of fibers during the grinding operation since only fibrous chrysotile was detected in thin sections of the plaques. Although other investigators [5] indicated that chrysotile curls up in water, an optical examination of the chrysotile used in this work did not show any tendency to change shape, even after immersion for extended periods. As additional identification characteristics, chrysotile has weak birefringence and a shimmery appearance when rotated in polarized light with crossed polars.

The other materials present in the dust were identified as follows: sisal, by its lack of staining and high birefringence; epoxy and polyester, by the colors produced on staining (red brown and pale yellow); and fibrous glass, by its distinctive shape. Epoxy, polyester, and fibrous glass are isotropic in polarized light. Calcite has high birefringence and a refractive index of about 1.66 in the ordinary direction.

Stained fibrous chrysotile particles with a ratio of length to diameter (L/D) > 3 and more than $3 \mu\text{m}$ in length were counted on five slides of each concentrated dust sample. The results are shown in Table 2. The number of free fibers increased with chrysotile content of the polyester resin plaques: no fiber at 0.8%, four at 2%, and a maximum of ten in the 4–18% level. No free fibers were found in the dust from the 4% epoxy resin plaque. This sampling and counting procedure was not used to establish a quantitative measure of airborne chrysotile fibers. However, the results indicate that only a few fibers of chrysotile were released by grinding chrysotile-bearing thermosetting resins and that epoxy resin, which "wets" the asbestos surface more readily than polyester, possibly releases fewer fibers than polyester resin.

Specimens collected on membrane filters for various times

TABLE 2—Comparison of optical and electron microprobe analysis of chrysotile asbestos in dust samples.

Examination Method	Microscopic						Electron Probe					
	stain	stain	NIOSH	NIOSH	NIOSH	NIOSH	Method 1	Method 1	Method 1	Method 2	Method 2	Method 2
Grinding Period	during	after	before	during	during*	after	during	during	after	after	during	during
Collection Method	water	water	membrane	membrane	membrane	membrane	water	water	water	water	membrane	membrane
Time, min	7	90	60	7	7	90	5	5	120	120	7	7
Particle Shape	$L/D > 3$	$L/D > 3$	$L/D > 3$	$L/D > 3$	$L/D > 3$	$L/D > 3$	non-fibrous	fibrous	non-fibrous	fibrous	non-fibrous	fibrous
Particle Length, μm	> 3	> 3	> 5	> 5	> 5	> 5	> 3	> 3	> 3	> 3	> 3	> 3
Sample No.	No. fibers/5 slides		No. fibers/ cm^2 of air				No. particles/1000 dust particles					
1	0	0	0	0	0	0	...	...	...	...	...	...
2	0	0	0.01	0	0	0	...	...	...	...	...	...
3	4	2	0.01	0.2	0	0.01	3	0	...	...	4	1
4	10	3	0.01	1.2	0.7	0.02	0	0	5	0	6	1
5	0	0	0.06	0.1	0.1	0.05	...	...	...	...	7	1
6	0	0	0	1.2	0.6	0.02	...	...	28	0	0	0
7	4	5	0	0	0	0	...	...	10	2	0	2
8	8	5	0.03	0.9	0.3	0.06	...	...	...	...	...	...

* Breathing zone.

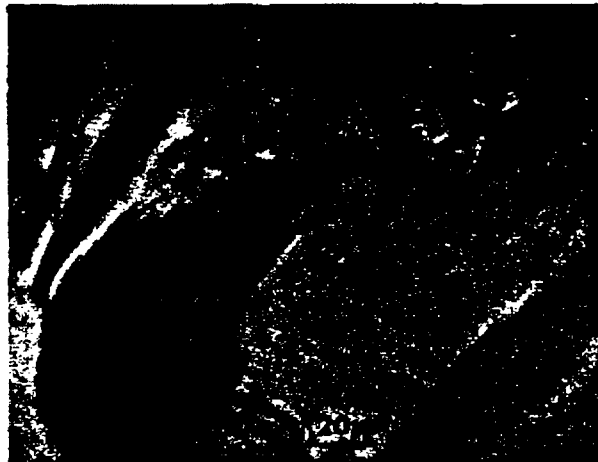
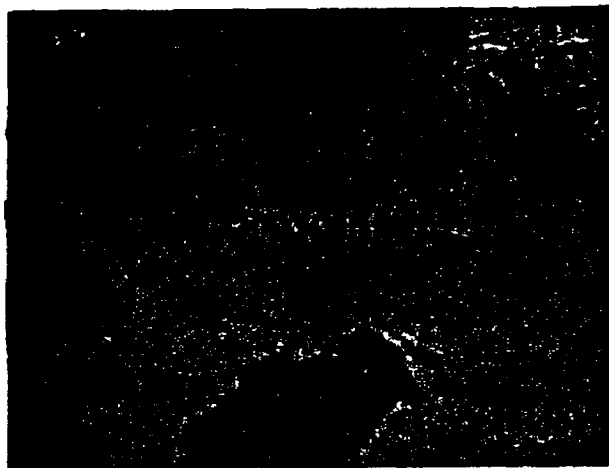


FIG. 2—Chrysotile and epoxy stained with iodine-glycerine-alcohol solution (Solution 2): (left) $\times 100$ and (right) $\times 500$.



FIG. 3—Chrysotile stained with iodine-benzene solution (Solution 3): (left) $\times 100$ and (right) $\times 500$.

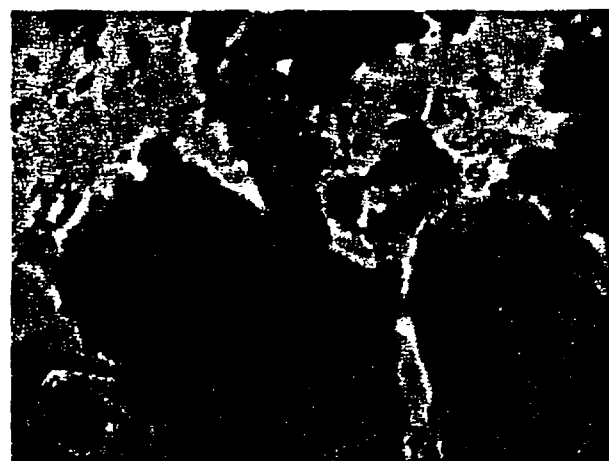


FIG. 4—Chrysotile, free and encapsulated, in polyester resin, stained with sublimed iodine ($\times 500$).

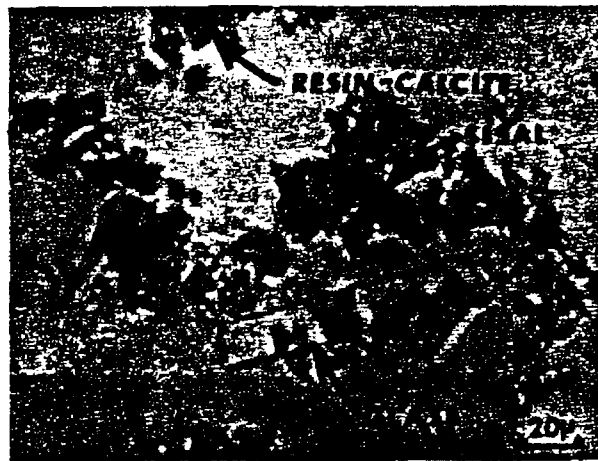


FIG. 5—Typical dust sample collected during grinding of polyester resin, chrysotile (10%), glass, sial, and calcite plaque in iodine-benzene solution (Solution 3, $\times 500$).

were analyzed by counting particles longer than 5 μm with $L/D > 3$ by the NIOSH phase contrast method. Fibrous glass and other materials that satisfied the size criteria but were clearly not asbestos were excluded. These counts include fibers other than chrysotile that cannot be identified by this technique. The results, listed in Table 2, indicate a trend similar to that of the staining data. The correlation of the two methods is particularly evident in the number of fibers detected in the dust from the 4% chrysotile-polyester plaque.

Evaporated Coatings

Carbon was vacuum deposited on chrysotile-bearing membrane filters. Sections of these filters were then mounted on either the phthalate-oxalate mounting medium or immersion oil and examined. As shown in Fig. 6, carbon emphasizes the difference in texture of the filter and chrysotile, improving the detection of small chrysotile fibers. In polarized light and crossed polars, a thin deposit of carbon increases the apparent birefringence of chrysotile. A polarized light examination is essential for distinguishing chrysotile from other fibrous materials in samples prepared by this method. A similar textural emphasis but not apparent increase in birefringence was achieved with evaporated aluminum.

Fraser [6] reports that the conical shape of the pores in Type AA cellulose ester filters restricts the penetration of particles to a depth of 15 to 20 μm , and Mercer [7], to a depth of 50 μm . In a discussion of a paper by Thomas [8], Green [9] reported that the penetration depends on several other factors, including air flow through the filter.

If the carbon evaporation method is to be of value, it is necessary that the particles detected at $\times 450$ be located on or very near the surface of the filter. To determine the depth of penetration, photomicrographs were taken at $\times 450$ of the same area on a carbon-coated chrysotile sample with ordinary and polarized light at two positions of brightness (90 deg apart—crossed polars) and compared. The particles detected with ordinary light were on and within 2 μm of the surface and with polarized light and crossed polars, on and throughout the filter.



FIG. 6—Chrysotile collected on cellulose ester membrane (CEM) filter and coated with vacuum-deposited carbon.



FIG. 7—Chrysotile on CEM filter—carbon coated: (A) location after coating and (B) location after transposing.

No particles located more than 2 μm below the surface of the filter were found.

Particle Retention on Membrane Filters

During the optical examination of the carbon- and aluminum-coated chrysotile samples collected on membrane filters, the particles frequently moved. For example, Fig. 7 shows the transposition of a carbon-coated chrysotile particle that occurred when the sample was prepared for microscopic examination.

The retentive power of membrane filters has been attributed to electrostatic charges developed by the passage of air through the filter. It was of interest to establish whether the evaporated coatings or sublimed iodine dissipates these charges, resulting in the particles being more sensitive to handling, or if particle movement also occurs on noncoated filters but is not recognized.

Filters containing chrysotile in the as-deposited state and after staining with sublimed iodine were examined in reflected light. A comparison of photomicrographs taken of the same areas before and after gently tapping the back of a slide supporting the stained and unstained chrysotile sections showed several particles were removed. Similar experiments were made using a gentle flow of air for about 1 s across the surface of the filter sections. The amount of stained and unstained chrysotile removed from the filters appeared to be about the same.

Filters containing somewhat large amounts of chrysotile were divided, and half of each filter was subjected to various mechanical treatments. The other half was the control sample. To emphasize differences, the treatments were more severe than dust-bearing filters would normally receive during transportation and handling. Some of the chrysotile-bearing filters were carbon coated to determine the effect of carbon. The effect of an evaporated coating of aluminum was assumed to be similar to that of carbon and therefore was not analyzed.

The same physical treatment for the carbon-coated and carbon-free filters was ensured by placing halves of each filter in a small plastic container (5 cm diameter by 1 cm) and rolled on the floor about 4 ft (1.2 m). Some filter halves were cut with scissors and others were dropped five times in a container about 4 ft (1.2 m). The filter sections were free to move in the con-

ainers. The magnesium contents of the control and treated samples shown in Table 3 were determined by atomic absorption. These data indicate the carbon deposit has a tendency to secure the chrysotile to the filter and emphasize the importance of careful handling.

Electron Microprobe Analyses

The dust samples collected in water and on membrane filters were analyzed to identify chrysotile and to distinguish between resin-encapsulated and free chrysotile on a quantitative basis. Free chrysotile particles refer only to a free surface exposed to the electron beam; the particle could be partially encapsulated. The methods and results are discussed below.

Method 1

Method 1 detects free chrysotile and chrysotile encapsulated in thin coatings of resin in water-collected dust samples. The dust samples concentrated by air evaporation, water-dispersed chrysotile, and silica-coated chrysotile¹ reference samples were placed on polished copper specimen mounts, air-dried, and

¹ Calidria Asbestos RG-244, Union Carbide Corp., Niagara Falls, N.Y.

carbon coated. Standard reference specimens were also prepared by compacting them when dry under pressure and carbon coating.

The intensities of the MgK_{α} and SiK_{α} X-rays generated in the reference specimens were measured, and ratios of these intensities representing the relative amounts of magnesium and silicon were established. A similar method could be used to analyze samples collected on membrane filters. Whether the samples were prepared from a wet or dry state did not affect the results.

The electron beam was positioned on 1000 dust particles by scanning a randomly selected area. Each particle was analyzed for magnesium and silicon, and the quanta of MgK_{α} and SiK_{α} generated were measured. Derived relative amounts of magnesium and silicon were compared with similar data from standards and the chrysotile particles were identified. Whenever chrysotile was detected, the shape of the particle and any associated orange cathodoluminescence, characteristic of the resin phase, were noted. Frequently, electron microprobe images were taken.

The images in Fig. 8 are representative of most of the chrysotile-bearing particles detected. The same or nearly the same locations of the white areas on the electron images, distinctive of the resin phase, and the areas enriched in magnesium and

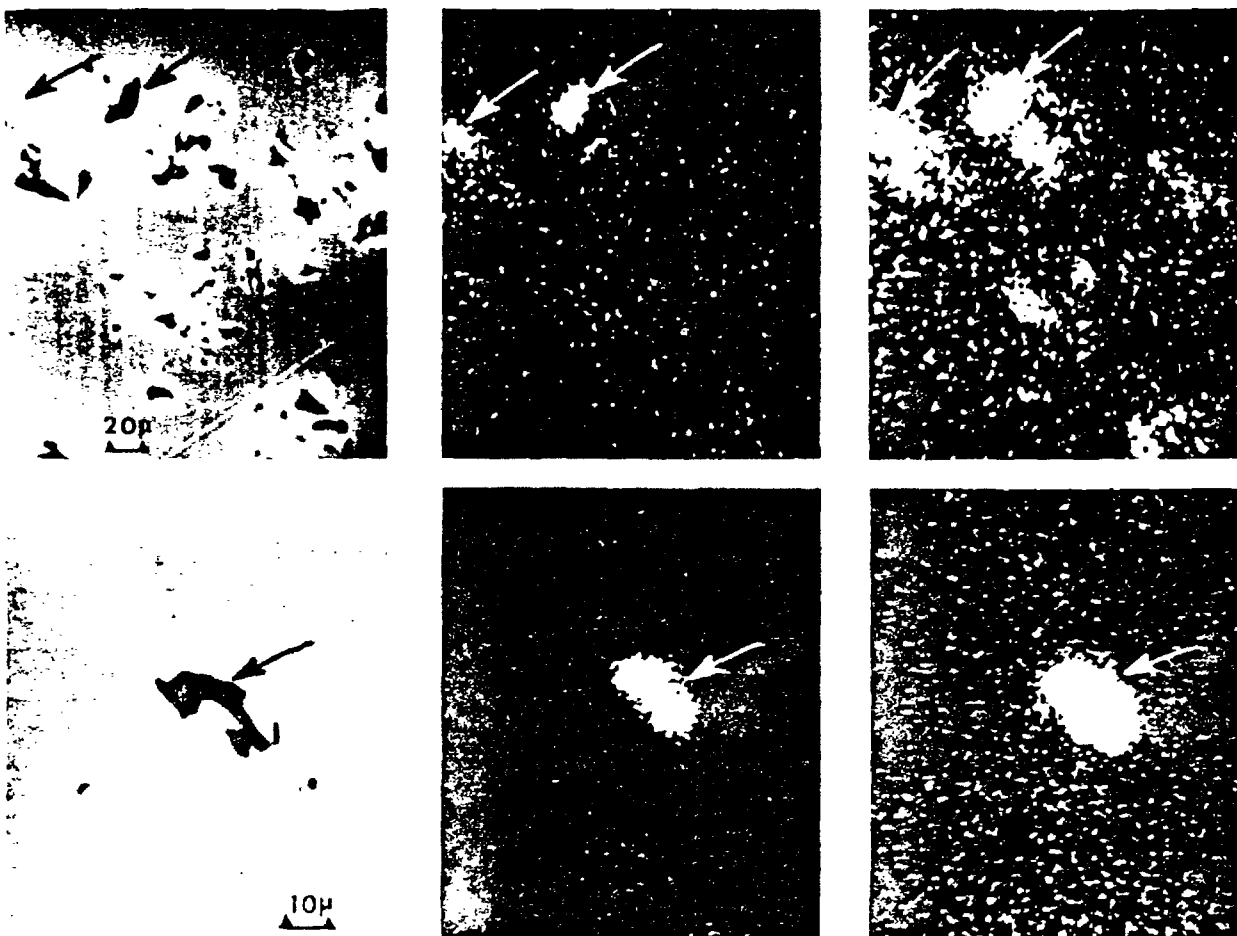


FIG. 8—Chrysotile particles in dust samples from 4% chrysotile-polyester plaque (top) collected during grinding, $\times 325$ and (bottom) collected after grinding, $\times 800$: (left to right, electron image, magnesium, and silicon. Chrysotile particles are emphasized with arrows.

TABLE 3—Magnesium contents of control and treated samples.

Sample Treatment	Uncoated		Carbon-Coated	
	% Mg	% Mg Removed	% Mg	% Mg Removed
Reference Sample	0.085	...	0.056	...
After rolling 4 ft (1.2 m)	0.039	54.1	0.051	8.9
Reference Sample	0.12	...	0.18	...
After cutting with Scissors	0.09	25	0.14	12.5
Reference Sample	0.062	...	...	...
After dropping 4 ft (1.2 m) (5 times)	0.042	32.3	...	...

silicon on the X-ray scanning images, characteristic of chrysotile, show the close association of chrysotile and resin.

The chrysotile particles detected during these analyses were usually equidimensional or lamellar rather than fibrous, as a result of grinding. For example, see Fig. 8, especially the magnesium and silicon X-ray scanning images.

Figure 9 is typical of the dust sample collected from the 10% chrysotile, polyester, glass, and calcite plaque. The phases indicated by numbers on the electron scanning image were identified as follows: No. 1, glass; No. 2, chrysotile, resin, and calcite conglomerate; and No. 3, calcite. The numbers of chrysotile particles per 1000 dust particles detected by this method are presented in Table 2.

One set of the samples analyzed by this method was collected in water for 5 min during grinding and the second set, for 2 h

after grinding. The maximum number of fibrous chrysotile particles detected, either free or encapsulated in a thin resin coating, was 2. Throughout these analyses, it was difficult to find fibrous chrysotile particles. For example, 28 nonfibrous but no fibrous, free, or encapsulated chrysotile particles were detected in a dust sample from a 10% chrysotile-bearing plaque.

Method 2

Method 2 was developed to detect only free chrysotile or chrysotile with a surface exposed to the electron beam in dust samples collected on membrane filters. It could also be applied to dust samples collected by other methods.

A standard sample of pure chrysotile, Calcidria asbestos RG-114[®], and a dust sample containing only resin-encapsulated chrysotile were collected on membrane filters. Sections of these two reference filters and the dust-bearing filters to be analyzed were secured to a specimen mount and carbon coated. The coating was deposited sufficiently thick to prevent the detection of the MgK_{α} and SiK_{α} X-rays generated in the encapsulated chrysotile standard but thin enough for easy detection when produced in the free chrysotile standard.

In a variation of this method, the carbon coating thickness was adjusted to detect only the SiK_{α} X-rays from the encapsulated chrysotile standard. The intensity of the SiK_{α} X-rays is three to four times greater than that of the MgK_{α} X-rays

• Union Carbide Corp., Niagara Falls, N.Y.

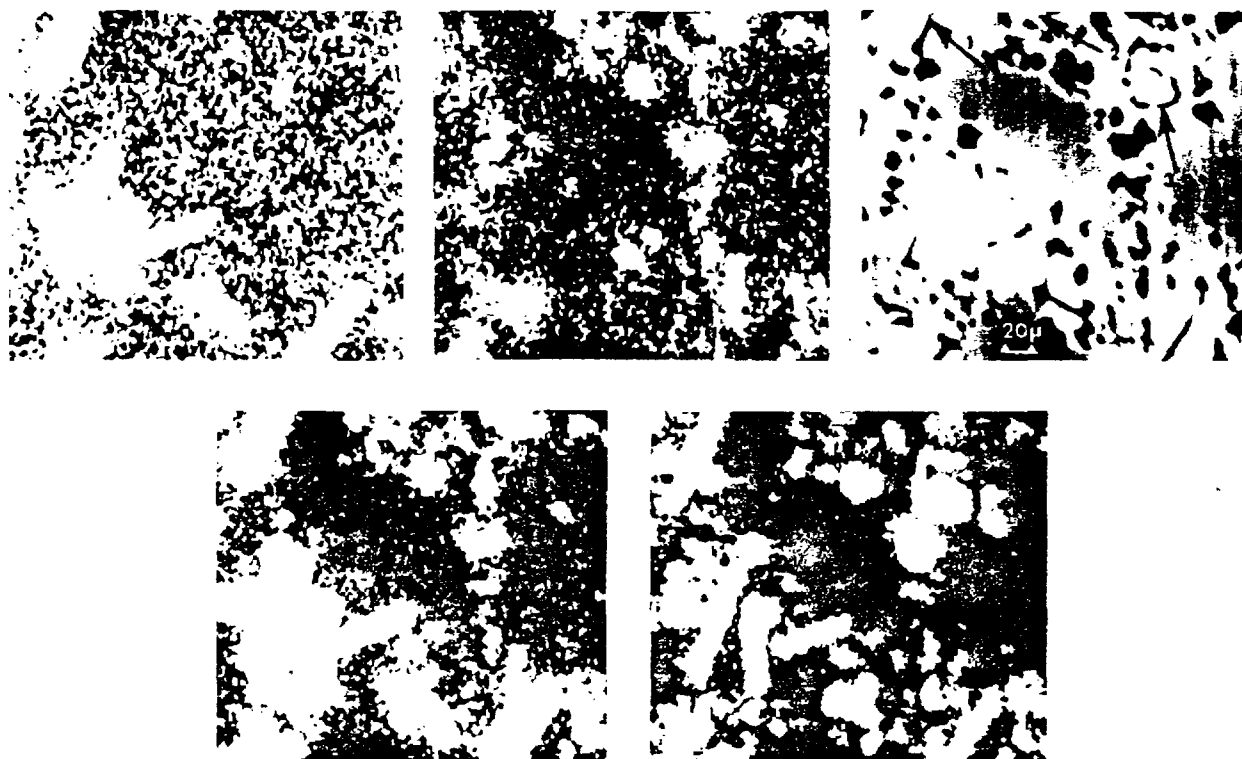


FIG. 9—Dust collected during grinding of polyester plaque containing 10% chrysotile, 10% glass, 10% sisal, and 85% calcite. (Arrow 1) a glass fiber; (Arrow 2) a chrysotile-bearing particle; (Arrow 3) calcite. (Top, left to right) electron image, magnesium, and aluminum; (bottom, left to right) calcium and silicon.

Thus, a thickness of the carbon coating can be deposited that will cut off any indications of the MgK_{α} X-rays and still permit the detection of the SiK_{α} X-rays. Such a carbon coating allows easy detection of both MgK_{α} and SiK_{α} X-rays generated from free chrysotile and thus discriminates between free and encapsulated chrysotile.

After adjustment of the carbon coating thickness, the dust-bearing membrane specimens on the same mount were analyzed. The electron beam was positioned on 1000 particles and the intensities of the MgK_{α} and SiK_{α} generated were measured. Ratios of these intensities were compared to similar ratios derived from the standard reference sample. The electron microprobe was operated at lower than normal specimen current and accelerating voltage, 50 nA and 15 kV, and the dwell time of the beam on each particle minimized to avoid melting or decomposing the filter.

Images typical of the free chrysotile standard prepared by this method are shown in Fig. 10, and a chrysotile particle associated with a trace of calcite in a dust sample, is shown in Fig. 11.

The number of free chrysotile particles detected per 1000 dust particles is shown in Table 2. The free fibrous chrysotile particles range from 0 to 2 per 1000 dust particles and the nonfibrous from 0 to 7.

Summary

Dust samples generated by grinding polyester and epoxy resin plaques containing up to 18% chrysotile asbestos were collected by settling in water and on membrane filters. Samples were examined optically and with the electron microprobe. The dust-bearing membrane filters were also analyzed by the NIOSH phase contrast method.

Staining procedures for identifying and increasing the optical detectability of chrysotile asbestos are presented. These procedures using iodine in solution or as a vapor are specific for the hydrated magnesia surface and distinguish chrysotile from all other commonly encountered fibrous materials. They can be applied to membrane filters but require the use of immersion oil to clear the filter.

Extensive optical examination of the samples collected in water, although not a quantitative measure of airborne chrysotile dust levels, shows clearly that free fibrous chrysotile is rare. There appears to be a trend with chrysotile content: no fibers at 0.8% chrysotile in the resin, a few at 2% chrysotile, and a low level at 4–18% chrysotile. The membrane filter samples analyzed by the NIOSH phase contrast method show similar trends.

Vacuum deposition of carbon and aluminum improves the detection of chrysotile by emphasizing the textural differences between chrysotile and the supporting membrane filter. Carbon deposition also increased the apparent birefringence of the chrysotile.

Studies were designed to compare the membrane filter retention of stained, carbon-coated, and as-deposited chrysotile. Results established that staining by sublimed iodine has no effect, whereas vacuum-deposited carbon has a tendency to secure the chrysotile to the filter. Movement of carbon-coated chrysotile on the filters was difficult to avoid but easily detected during the optical examinations. As-deposited chrysotile on membrane filters is probably more easily moved during handling than generally recognized, but, because of difficulties associated with its detection, overlooked.

An electron microprobe method was applied to water-collected

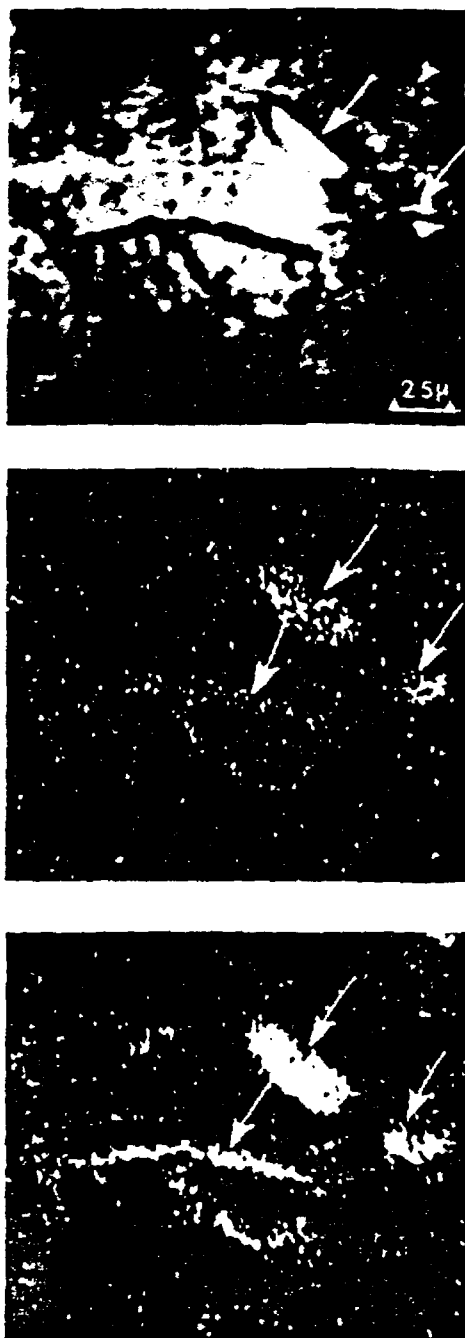


FIG. 10—Chrysotile collected on CEM filter (Method 2). Chrysotile particles are emphasized with arrows: (top) electron image, (middle) magnesium, and (bottom) silicon.

samples to measure the number of chrysotile particles per 1000 dust particles that were either encapsulated in a thin coating of resin or had a surface exposed to the electron beam. It was found that the chrysotile was usually present as somewhat equidimensional, resin-encapsulated particles that were frequently conglomerated with other materials. A total of 2 fibrous

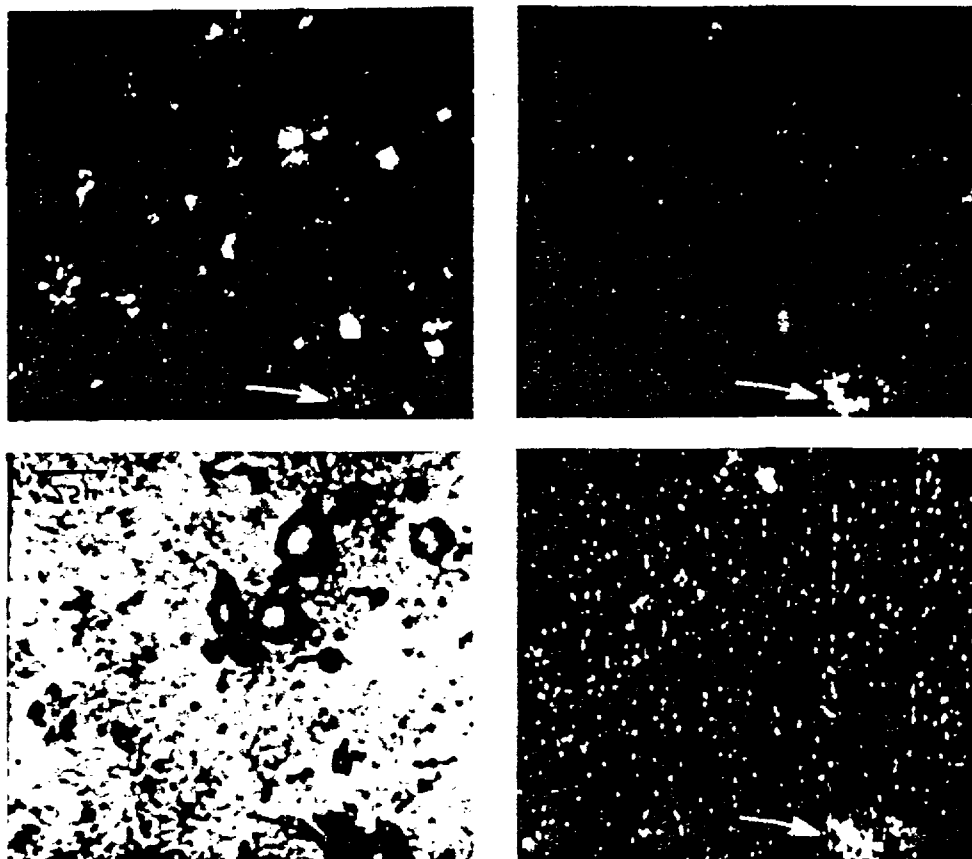


FIG. 11—Dust collected on CEM filter during grinding of polyester plaque containing 10% chrysotile, 10% sisal, 10% glass, and 35% calcite (Method 2). Chrysotile particles are emphasized with arrows. (Top left) electron image, (top right) magnesium, (bottom left) calcium, and (bottom right) silicon.

chrysotile particles was found in 4 dust samples (4000 particles counted) from plaques containing 2 to 10% asbestos.

A new method was developed to distinguish between resin-encapsulated chrysotile and chrysotile particles with a free surface exposed to the electron beam. This method was applied to count the number of chrysotile particles with a surface exposed to the electron beam in situ on membrane filters.

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