GEN 55 (REV 6-74)

teroffice Memorandum

(Name and Location)	DATE
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Use of SEM/XES to Identify Asbestos in Insulation

PLAINTIFF'S EXHIBIT CEL-609

SUMMARY

Scanning Electron Microscopy and X-ray Energy Spectroscopy were used to identify asbestos in samples of insulation. SEM/XES is a rapid, fairly simple method of identifying asbestos fibers as small as fractions of a micrometer in diameter. Asbestos minerals can be distinguished by morphology and relative Fe, Mg, Ca, Al and Si content as measured by XES.

Uniterms

Asbestos
Insulation
X-ray
SEM

INTRODUCTION

At the request of Mr. E. S. Ramey, I have examined five samples of insulation with Scanning Electron Microscopy and X-ray Energy Spectroscopy (SEM/XES) techniques. The purpose was to determine if asbestos was present, and to find out whether SEM/XES methods could detect asbestos easily in such materials.

CONCLUSIONS

Asbestos of two or three different types was easily detected by SEM/XES methods in three of the five samples. Results matched expectations as to which samples contained asbestos. Both the appearance in the SEM and the elemental composition determined by XES are necessary to identify asbestos.

RESULTS AND DISCUSSION

Qualitative observations on the appearance and a summary of elemental compositions are given for each sample in the Table. This data has been reported in ESR-291-76 (1). The three asbestos-containing samples (3-5) appear to be mixtures of asbestos with a material resembling glass fibers or glass wool in composition (but not in appearance). Sample 2 appears to be mostly glass-like fibers and particles. This is probably a raw mineral rather than a fused product like glass. Sample 1 consists of thin plates or flakes of a silica-alumina containing some K and Na.

Photomicrographs and X-ray spectra are shown in Figures 1-5. A brief discussion and interpretation of these data for each sample follows:

Sample 1 (Figure 1) - The flakes seen in the photomicrographs range in size from a few μm to about 50 μm. Many of them are partially transparent. This puts an upper limit of about 50 nm on their thickness (1, pp 12-13). The XES spectrum is also shown in the figure.

Sample 2 (Figure 2) - Small agglomerates of fine particulate matter are interspersed with ribbon-like fibers. These flat fibers are about 10-30 μm in width. The photomicrograph taken with a 1 KeV beam (very low electron penetration) shows that the surface of these fibers is also covered with fine particulate material. As the XES spectrum shows, the elemental compositions of the two forms are virtually identical.

Sample 3 (Figure 3) - This material consists of long rods on the order of 1 μm in diameter, and clumps of short fibers perhaps 1/2 to 1/10 this diameter. Due to the very small size of the particles, it was not possible to perform an XES analysis on an area which included only the long rods (X-ray spatial resolution is a few microns). In the spectrum shown for the rod-like fiber, the "background" due to the particulate matter has been mathematically subtracted by subtracting a normalized particulate spectrum from the fiber spectrum. The normalization factor was calculated by assuming the asbestos contained no Ca. This is probably not true. It is obvious, however, that the two materials are quite different, especially in Fe content.

Sample 4 (Figure 4) - The sample has agglomerates of fine particulate matter, bundles of fine fibers, and a few broken individual fibers or small bundles of fibers. The bundles are somewhat bent and twisted, and are covered with particulate debris. The large bundles are on the order of 10-20 μm in diameter. The XES spectra of the fibers and the particulate matter are quite different. There is some particulate matter contribution to the fiber spectrum shown. The high Mg content and sinuous fiber form strongly suggested the crysotile form of asbestos (2, 3). Since this form also contains fair amounts of Ca and Al along with Si, no correction as in sample 3 was attempted.

Sample 5 - This material is similar in appearance to sample 4, except that the fiber bundles are very straight and about twice the size of those in Figure 4. The clumps of particulate matter are also much larger than in sample 4. XES spectra of the particles and the fiber bundles are also different from sample 4. Neither spectrum is corrected for the small amounts of the other phase which are seen to be present on close examination of the areas analyzed. The high Fe/Mg ratio as well as the appearance suggest amosite or crocodilite as the asbestos type present.

Criteria for asbestos identification by SEM/XES methods are suggested by the above results. Visually, the appearance of long or short rod-like fibers individually, in bundles, or in fuzzy agglomerates suggests the presence of an asbestos-type mineral. Identification can be confirmed rapidly by XES methods in the SEM, looking in particular for the Mg, Fe, and Si characteristic of all the asbestos minerals. This is a fairly rapid and simple process in the SEM requiring a few minutes per suspected fiber.

Distinguishing one asbestos mineral from another is more difficult. There are some ten asbestos forms, some of which differ only subtly in morphology and elemental composition. The relative amounts of Mg, Fe, Al, and Ca are particularly useful in distinguishing these minerals. A ternary diagram in which X-ray intensities of Mg, (Fe + Mn), and (Na + Ca + Al) are the basis of the three axes has been reported (3). Since these quantities are highly dependent on instrumental conditions

and sample preparation, each laboratory would have to produce its own compositional diagram using known materials. Fortunately, most of the asbestos used commercially is chrysotile, which is easily identified by its serpentine strands and high Mg/Fe ratio.

Sample preparation required simply picking up some of the material on double-sided tape affixed to an SEM stub, then coating the surface lightly with carbon in a vacuum evaporator. The carbon coating was kept thin to improve X-ray detectability. The resulting rather low conductivity limited high magnification resolution somewhat.

Instrumental conditions were those routinely employed in our laboratory, i.e. a beam energy of 20 KeV, secondary electron photomicrographs, and 30° sample tilt.

Peter B. DeGroot

PBD:eem

REFERENCES

1. Hayat, M. A., ed., "Principles and Techniques of Scanning Electron Microscopy," Vol I, Van Nostrand Reinhold, New York, (1975).

2. Walker, C. W., G. G. Paulson, and R. E. Ferrell, Proc. of the 32nd Annual Meeting of the Electron Microprobe Society of America. 1974. 524-25.

3. McCrone, W. C. and J. G. Delly, The Particle Atlas Vol III. Ann Arbor Science Publishers, Inc., Ann Arbor MI. 1975. 627-28.

TABLE I

CHARACTERIZATION OF INSULATION SAMPLES BY APPEARANCE IN THE SEM AND BY ELEMENTAL COMPOSITION

Sample No.	Form of Material	Identification of Material	Elements Found by XES		
			Major	Minor	Trace
1	Thin flakes	?	Si, Al	K, Na	Fe
2	Long flat fibers	?	Ca, Si	Al	Fe, Cr ?
	Fine particles	?			
3	Long round very fine fibers	Asbestos (Amosite and/or Anthrophyllite?)	Fe, Si	Mg, Mn, Al	--
	Clumps of short, fine fibers	?	Ca, Si	Al	--
4	Bundles of long, fine fibers	Asbestos (Chrysotile)	Si, Ca, Mg	Al, Fe	--
	Fine particles	?	Si, Ca	Na, Al	S, Fe
5	Bundles of long, fine fibers	Asbestos (Amosite or Crocodolite)	Fe, Si	Mg	Al
	Clumps of very fine particles	?	Si, Ca	--	Na

FIGURE 1

SECONDARY ELECTRON PHOTOMICROGRAPHS AND XES SPECTRUM
INSULATION SAMPLE NO. 1

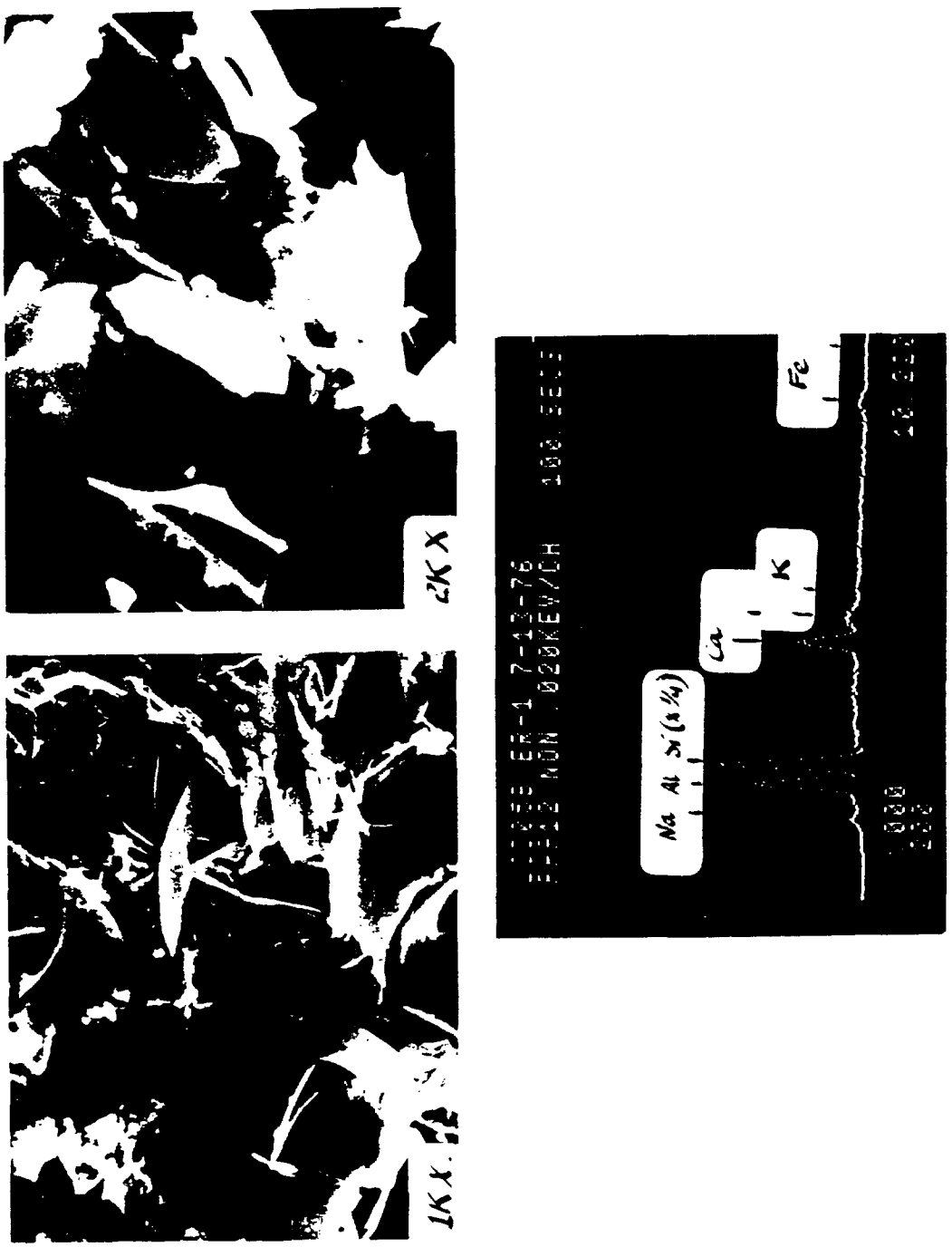
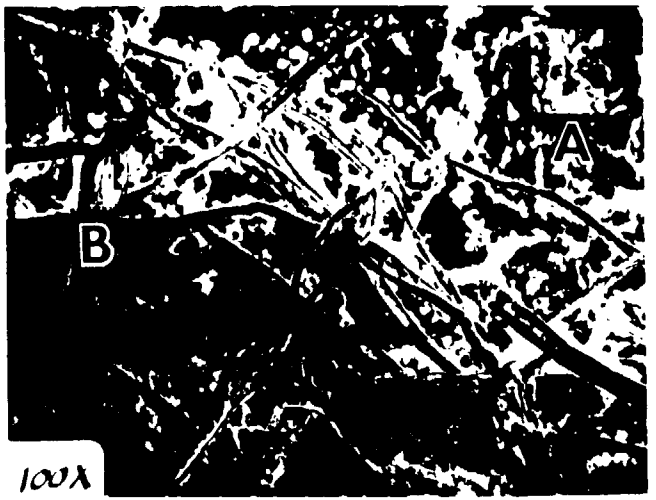
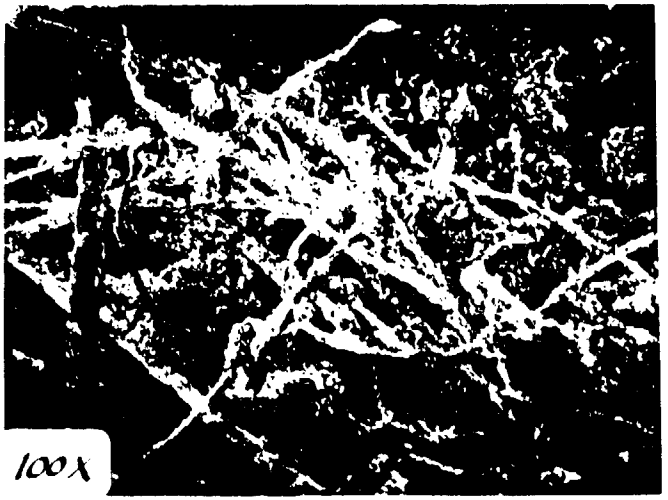


FIGURE 2

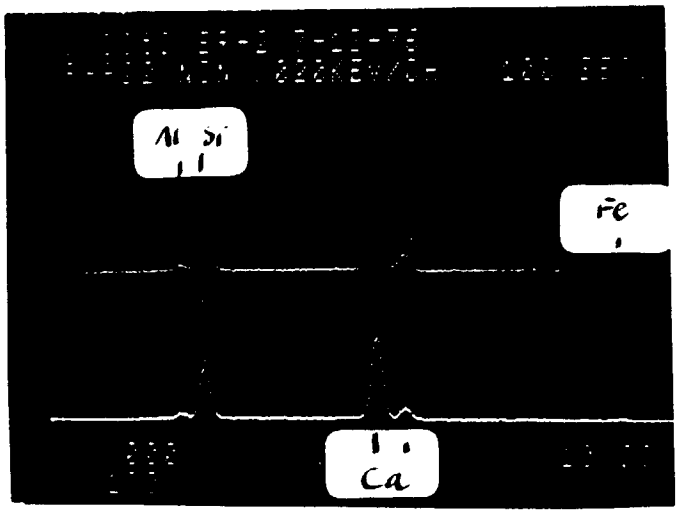
SECONDARY ELECTRON PHOTOMICROGRAPHS AND XES SPECTRA
INSULATION SAMPLE NO. 2



20 KeV Beam



1 KeV Beam

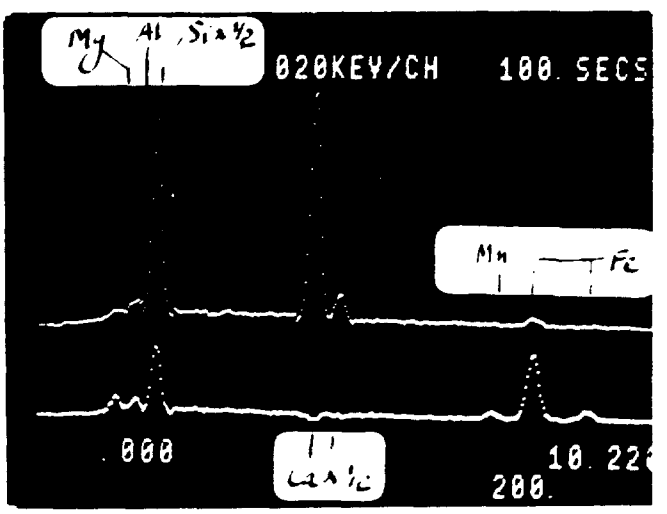


XES Spectrum, Area A Above

XES Spectrum, Area B Above

FIGURE 3

SECONDARY ELECTRON PHOTOMICROGRAPH AND XES SPECTRUM
INSULATION SAMPLE NO. 3



Top - XES Spectrum, Area B
Bottom - XES Spectrum, Area A

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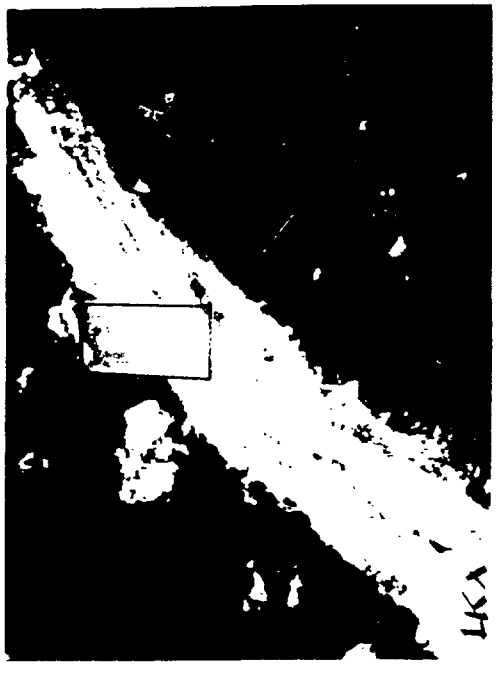
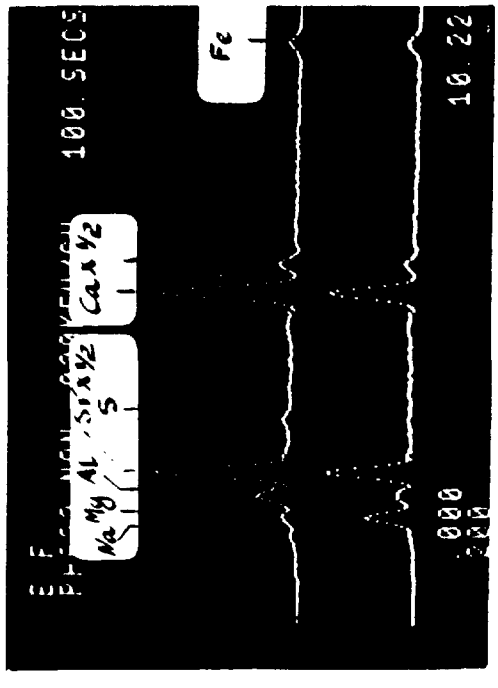
FIGURE 4

SECONDARY ELECTRON PHOTOMICROGRAPHS AND XES SPECTRA
INSULATION SAMPLE NO. 4



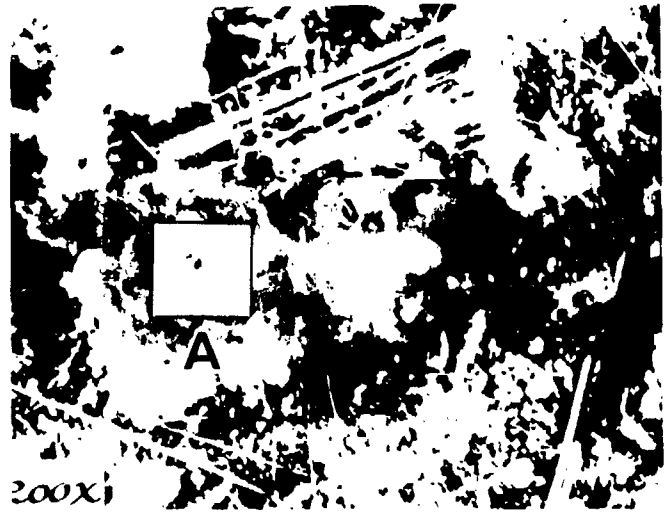
XES Spectrum of particles (out of photomicrograph field of view).

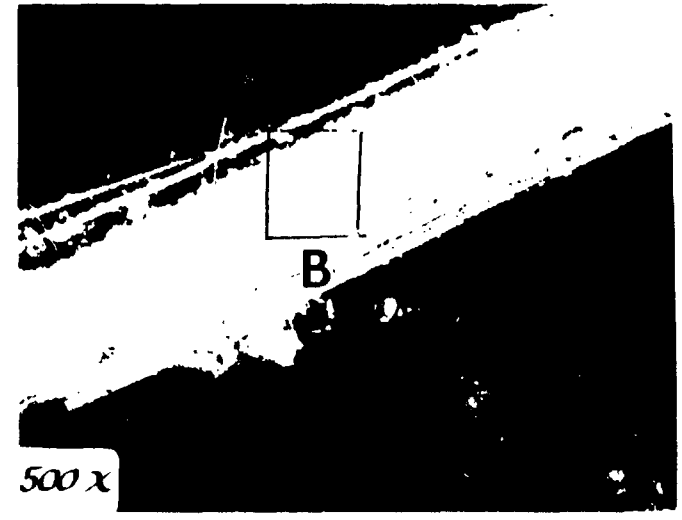


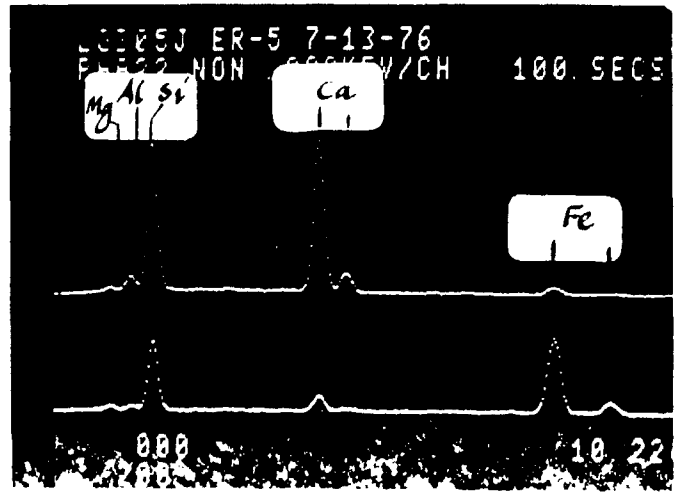
XES Spectrum of fiber area highlighted above

FIGURE 5

SECONDARY ELECTRON PHOTOMICROGRAPHS AND XES SPECTRA
INSULATION SAMPLE NO. 5







XES spectrum, area A above

XES spectrum, area B above

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