Reprinted from ENVIRONMENTAL RESEARCH, Volume 4, Number 2, April 1971
Copyright © 1971 by Academic Press, Inc. *Printed in U. S. A.*

PLAINTIFF'S EXHIBIT

CAP-1477

ENVIRONMENTAL RESEARCH 4, 141–145 (1971)

Electron Diffraction Patterns of U.I.C.C. Asbestos Samples

M. I. SKIKNE, J. H. TALBOT, AND R. E. G. RENDALL

Pneumoconiosis Research Unit of the South African Medical Research Council at the South African Institute for Medical Research, Johannesburg, Republic of South Africa

Received December 9, 1970

The U.I.C.C. standard samples of amosite, anthophyllite, crocidolite, chrysotile A, and chrysotile B have been studied by electron diffraction. The effect of fibre orientation on the electron diffraction patterns was studied using a double-tilt stage. Rotation did not have a marked effect on the general pattern of weak and strong spectra. It has been shown that electron diffraction method may be used to identify single fibres of the samples.

The International Union against Cancer (U.I.C.C.) has sponsored the preparation and distribution of standard reference samples of each class of asbestos (Timbrell, Gilson, and Webster, 1968). These include one sample each of the three amphiboles (Speil and Leineweber, 1968) amosite, anthophyllite, and crocidolite, and two samples of chrysotile designated chrysotile A (Rhodesian) and chrysotile B (Canadian). These samples have been studied by electron diffraction in order to obtain data for the identification of fibres sampled from the atmosphere and from biological tissues (Gross, Detreville, and Haller, 1970; Parsons, 1968; Thomson, Kaschula, and MacDonald, 1963; Thompson, 1965; Thompson and Graves, 1966). These studies are described here.

METHOD

Several specimens of each sample were prepared by suspending a small mass of the fibres in a very dilute solution of collodion in amyl acetate. The fibres were dispersed by mechanical agitation of the suspension. Films of the collodion-containing asbestos fibres were formed by placing a drop of the suspension on a water surface. Sample areas of the films were mounted on electron microscope specimen grids.

The electron microscope used was the AEI series EM 801 which has a selected area diffraction facility. Selected area diffraction patterns of circular areas 1 μm in diameter were taken. In the case of the amphiboles individual fibres were selected, but in the case of chrysotile it was only rarely possible to find individual fibres as most of the fibres occurred in open bundles.

The effect of fibre orientation on the electron diffraction pattern was studied by means of the double-tilt stage. Fibres were rotated about axes both parallel and perpendicular to the fibre axis and electron diffraction patterns recorded at various orientations. Angles about both axes up to 30° from the untilted position were used.

ASARCO ALV 0006355

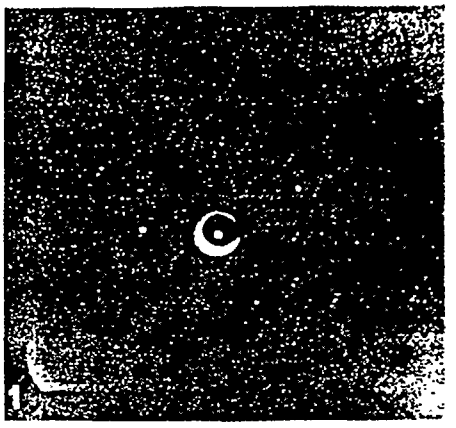

FIG. 1. Diffraction pattern of crocidolite.

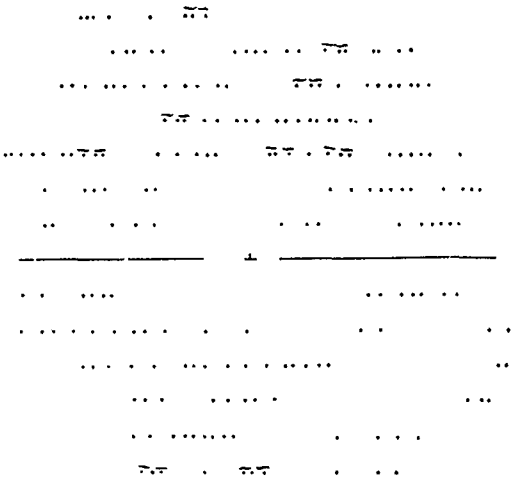

FIG. 2. Spot pattern of crocidolite. Made from a 4 × enlargement of a diffraction pattern.

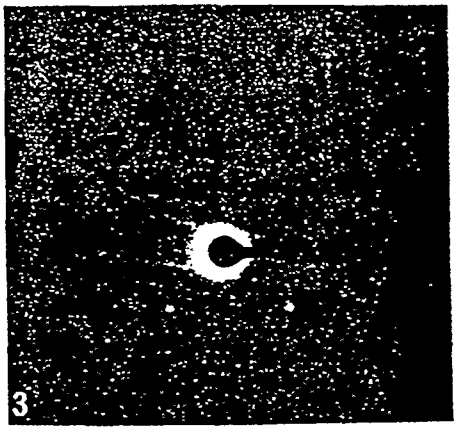

FIG. 3. Diffraction pattern of amosite.

ASARCO ALV 0006356

RESULTS

All five of the U.I.C.C. samples yielded single fibre diffraction patterns consisting of straight parallel rows of diffraction spectra. The spacing between rows corresponds to a period of 530 pm in the crystal and this is the same for all five samples. The crocidolite pattern has spectra with alternating long and short spaces corresponding to periods of 1.66 and 2.16 nm, respectively (Figs. 1 and 2). In the cases of amosite and anthophyllite the spectra in each row are equally spaced corresponding to a period of 1.80 nm (Figs. 3 and 4). Crocidolite may therefore be distinguished readily from amosite and anthophyllite.

Most of the fibres in the anthophyllite sample showed a distinctive pattern of

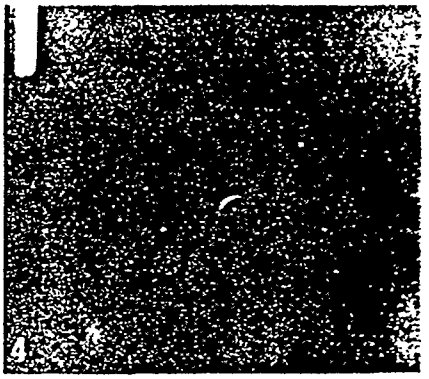

FIG. 4. Diffraction pattern of anthophyllite.

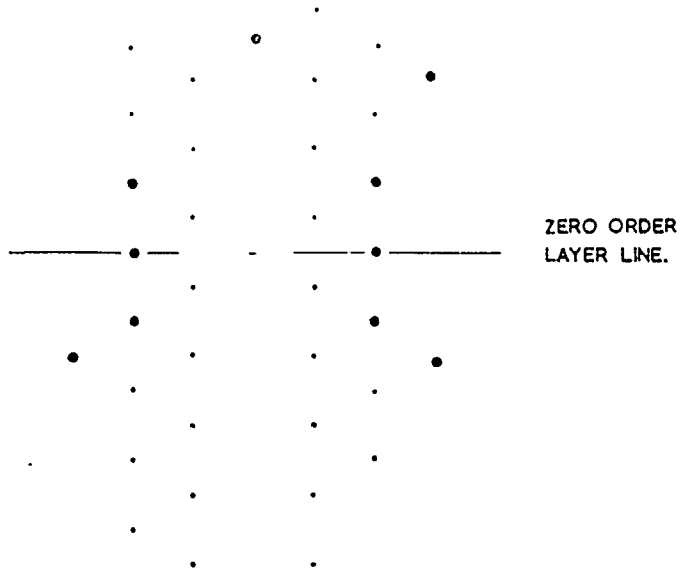

FIG. 5. Spot pattern of anthophyllite. Note intense second-order spots. Made as in Fig. 2.

ASARCO ALV 0006357

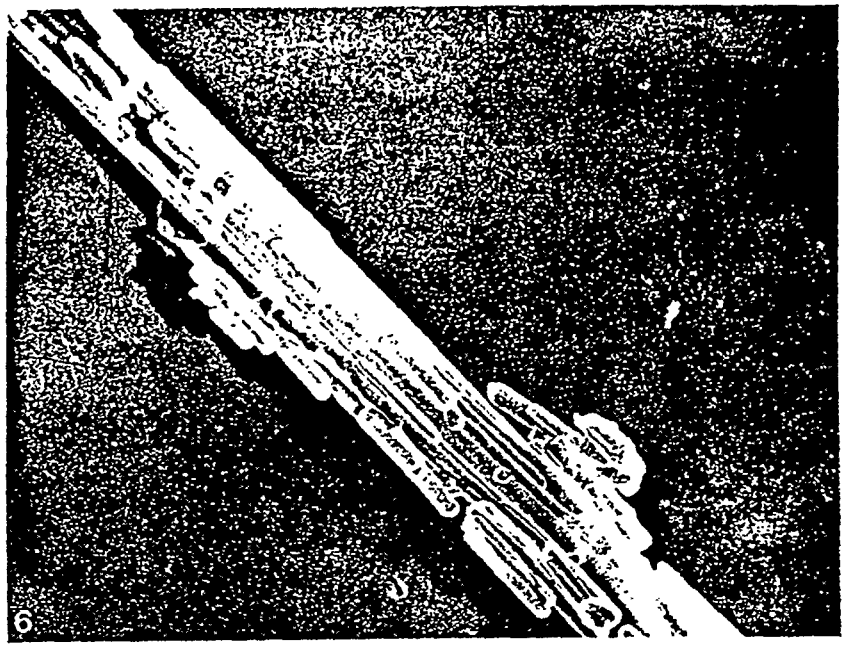

FIG. 6. Transmission replica of chrysotile. ×66,000.

strong spectra which is shown in Fig. 5. In some cases this general pattern was slightly modified, presumably due to absorption in the fibre.

The anthophyllite sample was observed to contain considerable quantities of a sheet silicate, probably mica or talc.

Diffraction patterns of amosite fibres were found to be more variable and no such distinctive pattern of strong spectra was found as in the case of anthophyllite. Thus to distinguish between amosite and anthophyllite we have chosen the criterion of the array of the intense spots in the diffraction patterns. Figures 3–5 illustrate this point.

The effect of orientation on the diffraction pattern was investigated by means of a double-tilt stage. It was found that rotation about either the fibre axis or an axis perpendicular to the fibre axis produced significant changes in the relative intensities of the various spectra but the general patterns already described were preserved. The fibres produce distinctive diffraction patterns whatever their orientation.

Chrysotile fibres are distinctive, both in their appearance in the electron microscope and in their electron diffraction patterns. The fibres are usually curved and occur in bundles having the appearance of fibres in the process of splitting off from a large composite fibre. The diffraction patterns are characterized by arcing of the spectra and in some cases rings. Typical electron micrographs and diffraction patterns are shown in Figs. 6 and 7. The patterns produced by both chrysotile samples are similar and this indicates that the minerals are probably the same.

ASARCO ALV 0006358

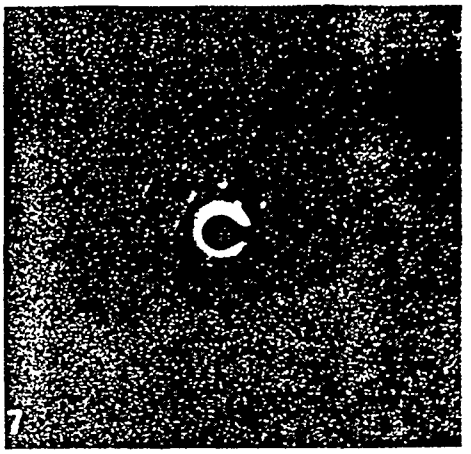

FIG. 7. Diffraction pattern of chrysotile.

CONCLUSION

This study of the electron diffraction patterns of the U.I.C.C. samples has shown that in most cases it is possible to identify individual fibres of amosite, anthophyllite, crocidolite, and chrysotile. The patterns of the three amphiboles bear a close resemblance to each other but in general may be distinguished by differences in the spacing of spots and the pattern of intensity variations within the layer lines. Chrysotile has a distinctive appearance and electron diffraction pattern.

REFERENCES

GROSS, P., DETREVILLE, R. T., AND HALLER, M. N. (1970). Asbestos versus nonasbestos fibres. *Arch. Environ. Health* 20, 571–578.

PARSONS, D. F. (1968). The examination of mineral deposits in pathological tissues by electron diffraction. *Int. Rev. Exp. Pathol.* 6, 1–54.

SPEIL, S., AND LEINEWEBER, J. P. (1965). "Asbestos Minerals in Modern Technology." Johns-Manville Research Center, Manville, N. J. 29 pp.

THOMSON, J. G. (1965). Asbestos and the urban dweller. *Ann. N. Y. Acad. Sci.* 132, 196–214.

THOMSON, J. G., AND GRAVES, W. M. (1966). Asbestos as an urban air contaminant. *Arch. Pathol.* 81, 458–464.

THOMSON, J. G., KASCHULA, R. O., AND MACDONALD, R. R. (1963). Asbestos as a modern urban hazard. *S. Afr. Med. J.* 37, 77–81.

TIMBRELL, V., GILSON, J. C., AND WEBSTER, I. (1968). UICC standard reference samples of asbestos. *Int. J. Cancer* 3, 406–408.

ASARCO ALV 0006359