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# The aid of X-Ray powder diffraction to the characterisation and treatment of asbestos containing materials

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ABSTRACT. — Fibrous asbestos minerals have been identified as priority substances for risk reduction and pollution prevention. Because of the well known hazardous effects associated with past occupational exposures to asbestos and because of its widespread use in commerce, exposures to asbestos present a general health hazard. Therefore, the identification and quantification of asbestos in bulk materials which are still sited in a number of plants and public buildings is fundamental for a proper plan of social prevention and general intervention. X-ray powder diffraction is considered an effective tool of investigation of bulk materials with a high statistical significance. The combined Rietveld-RIR method, has been successfully applied for the quantitative determination in the past of asbestos in bulk materials such as insulating cements, mixed asbestos-slag wools, contaminated soils, and others.

The University of Jussieu in Paris has been partly evacuated because the framework of the building is composed of an asbestos rich material. This paper reports the description of the characterisation of that material and a possible plan of intervention for the treatment of the hazardous material *in situ*.

RIASSUNTO. — È noto già da parecchio tempo che i materiali fibrosi classificati genericamente come asbesti (appartenenti alla famiglia del serpentino nella varietà *crisotilo* ed alla famiglia dell'anfibolo varietà *riebeckite* e *grunerite*) sono potenziali agenti cancerogeni. Data la presenza a tutt'oggi sul territorio di milioni di $m^2$ di prodotti quali il cemento-amianto o l'amianto fioccato contenenti percentuali variabili da 0.5 a 20% in peso di asbesto, la caratterizzazione di questi materiali per una corretta classificazione e trattamento, è condizione essenziale per qualsiasi tipo di intervento. La diffrazione di raggi X su polveri si è dimostrata, ancora una volta, strumento fondamentale per la caratterizzazione *in bulk* dei materiali. La tecnica che verrà descritta si avvale della combinazione del *metodo Rietveld* e del *metodo RIR* (Reference Intensity Ratio) per arrivare alla determinazione quantitativa di asbesto in materiali massivi contenenti fibre di lana di vetro (materiale amorfo).

Nel presente lavoro viene descritta in particolare l'applicazione per la risoluzione di un problema reale legato alla bonifica *in situ* di amianto fioccato presente all'Università di Jussieu (Parigi, Francia).

KEY WORDS: *Asbestos, chrysotile, grunerite, combined Rietveld-RIR method, loose asbestos containing materials, epoxy resin.*

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## INTRODUCTION

Asbestos minerals are classified in amphibole species (*anthophyllite, grunerite* or *amosite, riebeckite* or *crocidolite* and *tremolite*) and serpentine species: *lizardite* (Rucklidge and Zussman, 1965), *antigorite* (Kunze, 1961), *chrysotile* (Whittaker, 1956), and *carlosturanite* (Mellini *et al.*, 1985).

Asbestos fibers have been utilised since antiquity: anthophyllite for example was used in pottery in Finland in 2500 B.C., clothes made of asbestos were described by Carlemagne in 800 A.D. and Marco Polo in 1250 A.D. (Liddell, 1991). Major industrial use of asbestos began in 1878 with mining of chrysotile in Quebec (Canada) followed by crocidolite mining in 1910 and amosite mining in 1916 in South Africa. After more than 30 years, toxicity of asbestos was recognised: Doll in 1995 in fact observed increased mortality from lung cancer in British asbestos workers (Bhandari and Dhariyal, 1990). In 1972, in USA it was established a Permissible Exposure Limit (PEL) for asbestos of 2.0 fibres/ml (f/ml) as an 8-hour-time-weighted average (TWA) and in 1986 PEL was reduced to 0.2 f/ml. Currently there is ongoing concern about the health risks associated with exposure to asbestos since exposure to asbestos fibres is associated with diseases like *asbestosis, mesothelioma, lung cancer*. Concerning the hazardous effects, it is well known that surface features and physical shape of the asbestos particles play a key role in the interaction with the organism cell interface (Hochella, 1993; Langer *et al.*, 1972; Johan *et al.*, 1976; Thomassin *et al.*, 1976; Jurand *et al.*, 1983). According to Hume and Rimstidt (1992) the dissolution reaction of chrysotile in the pH range of lung fluids is:

$$Mg_3Si_2O_5(OH)_4 + 6H^+ \rightarrow 3Mg^{2+} + 2H_4SiO_4 + H_2O.$$

From this, they predicted that a 1 μm diameter chrysotile fibre would completely dissolve in the human lung in 9.0(4.5) months. Therefore, because the onset of disease is generally long (20 years), at least the first step in disease generation should be relatively quickly (Hocella, 1993; Pott, 1987). From epidemiological and *in vivo* studies, it is clear that crocidolite fibres have a remarkably longer lifetime in the lung tissue than chrysotile (Jones *et al.*, 1989) and thus more pathogenic than chrysotile since they seem relatively chemically stable in lung-like environments (Harington, 1962; Kane, 1993; Stanton *et al.*, 1981). In addition, amphibole asbestos is more hazardous than chrysotile because it contains iron. In fact, hydroxyls and super-oxide free radicals as generated by catalysis on fibre surfaces and the toxic hydroxyl radicals are generated in the presence of free oxygen. The mechanism involves the reduction of oxygen via the oxidation of iron on the mineral surface and the basic equation is (Fenton, 1984):

$$H_2O_2 + Fe^{2+} \rightarrow OH^{\bullet} + OH^- + Fe^{3+}$$

Regarding the physical shape, the activity among the different fiber types increases with decreasing diameter and increasing fibre length (Ross *et al.*, 1993). Stanton *et al.* (1981) postulated that the optimum morphology for the induction of intrapleural cancer is a diameter of $\leq 0.25$ μm and a length $>8$ μm.

Concerning the cytoxicity, a number of *in vitro* and *in vivo* studies were carried out in the past but still a general picture is not clearly drawn. Because macrophages are avidly phagocytic, mineral particles are rapidly internalised, and macrophages release a diverse array of products (inflammatory activity) into their extracellular environment and stimulate oxidant production (Nathan, 1987). Alternatively, macrophages and neutrophils produce a number of enzymes, oxidants, chemo-attractants and cytokines (particularly TNF-a) which may: a) deactivate minerals; b) activate cell defense mechanisms; c) cause cell injury and abnormal cell function of epithelial cells, fibroblasts, and mesothelial cells, that is, they can initiate or take part to the disease process (Fenton, 1984; Weitzmann and Graceffa, 1984).

The need of a characterisation and accurate

quantitative analysis of asbestos containing materials is of key importance for a number of reasons: (*a*) classification of the material typology and definition of the hazardousness degree for the application of new restrictive laws imposed by the environmental agencies (Veblen and Wylie, 1993); (*b*) classification of the material typology for a proper plan of intervention and disposal; (*c*) understanding of the interaction asbestos-organism in biological studies.

The application of the Rietveld method to poorly crystalline mixtures containing disordered phases such as chrysotile is not possible. The Rietveld method is successful for the extraction of quantitative data when the structure of the crystalline phases in the mixture is ordered or has a limited extent of disorder. The structure of chrysotile is described as a cylindrical lattice, and has a large density of structure disorder. This causes reflections along well defined crystallographic directions to be broadened. In such conditions, the integrated intensity of the modulated bands as simulated in the Rietveld-type refinements are underestimated with respect to the observed profiles, and therefore the value of the phase fraction obtained from the Rietveld refinement leads to an incorrect amount of chrysotile. Gualtieri and Artioli (1995) developed a reliable structure model for the disorder in chrysotile. Without such a model, the application of the Rietveld method (Rietveld, 1969; Bish and Post, 1993; Young, 1993) is not possible. However, the developed model cannot fully account for the broadening of the diffraction peaks and therefore an internal normalisation by RIR (Reference Intensity Ratio) (Davis *et al.*, 1990) is required. The combined Rietveld-RIR method offers a number of advantages: (*a*) it is based on a rigorous model for the structure of chrysotile; (*b*) the profile-fitting technique allows control and evaluation of all microstructural, instrumental, and textural parameters of the sample, so that the proposed structure model can be adopted to chrysotiles with different degree of disorder; (*c*) the phase fractions extracted by the Rietveld refinements are rescaled on the basis of the absolute weight of corundum originally added to the mixture as an internal standard (internal renormalization); (d) the technique is of straightforward application and advantageous in terms of sample preparation and time requirements.

Since 1994, large areas of the University of Jussieu in Paris have been evacuated because it was unambiguously discovered that the framework of the building is composed of an asbestos rich material. This paper reports the description of the characterisation of the asbestos rich material collected at the building of the University of Jussieu in Paris (France) and a possible plan of intervention for the treatment of the hazardous material *in situ*.

## METHODS

### *Theoretical background and previous studies.*

Chrysotile is described in terms of a cylindrical lattice (Wicks and O'Hanley, 1988; Titulaer *et al.*, 1993). The description of the structure symmetry in the various chrysotile types is rather complex due both to the curvature of the lattice and to the statistical stacking of the layers. Since there is no univocal model for the chrysotile structure and its polytypes, Gualtieri and Artioli (1995) developed a structure model capable to satisfactorily describe the diffraction effects in the powder spectrum. The model was developed using the powder diffraction spectrum of the well characterised chrysotile from Jeffrey Mine, Quebec Canada. The structure was refined in space group *C2/m*, and the effect of layer curvature was modelled by a broadening parameter on the crystallographic direction *b*. Fig. 1 (after Gualtieri and Artioli, 1995) shows the calibration curves of the expected versus observed chrysotile content using the results of the Rietveld refinement alone (a), and using the RIR corrected values (b). In both cases the samples having different matrix composition lie on the same curve, indicating that the matrix effects have been perfectly accounted for.

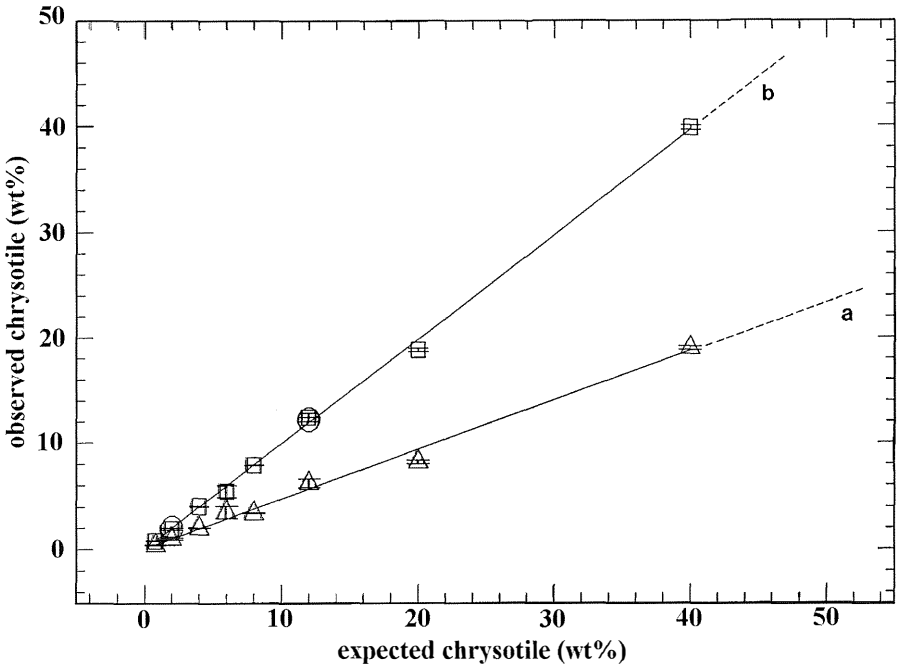



Fig. 1 – The calibration curves of expected vs observed chrysotile content (wt%) for the reference mixtures: (*a*) using the weights from the Rietveld refinements. The empty triangles are the ternary mixtures CH1-CH8; (*b*) using the weights from the Rietveld refinements and RIR-renormalization. The empty squares are the ternary mixtures CH1-CH8, the empty circles are the CH9 and CH10 multicomponent mixtures (After Gualtieri and Artioli, 1995).

*Materials and experiments.*

Two samples from the building of the University of Jussieu in Paris (France) were analysed. The specimen labeled PARIS1 was sampled from a ceiling of the building; the sample labeled PARIS2 was sampled from a wall of the building. The samples were considered representative of the construction material of the building. A qualitative investigation by X-ray powder diffraction revealed that the sample PARIS1 is composed mainly of amorphous material (rock or slag wool) with minor amounts of serpentine asbestos (chrysotile), calcite and brucite. The sample PARIS2 is as well composed mainly of amorphous material (rock or slag wool) with minor amounts of grunerite, calcite and quartz. For the quantitative analysis, the powders were added 10%wt NIST standard corundum to carry out the Rietveld-RIR. Data were collected using a Philips Bragg-Brentano diffractometer with a curved pirolitic graphite monochromator on the diffracted beam, in the range 5-100 °2θ with steps of 0.02 °2θ, 10 s/step, 0.5° divergence slit, 0.1 mm receiving slit, and 0.5° antiscatter slit. Data sets were refined by the Rietveld method using GSAS (Larson and von Dreele, 1999). In GSAS, the structure factors were calculated by using the formal atomic scattering factors. The background was successfully fitted with a Chebyshev function with 16 coefficients. The peak profiles were

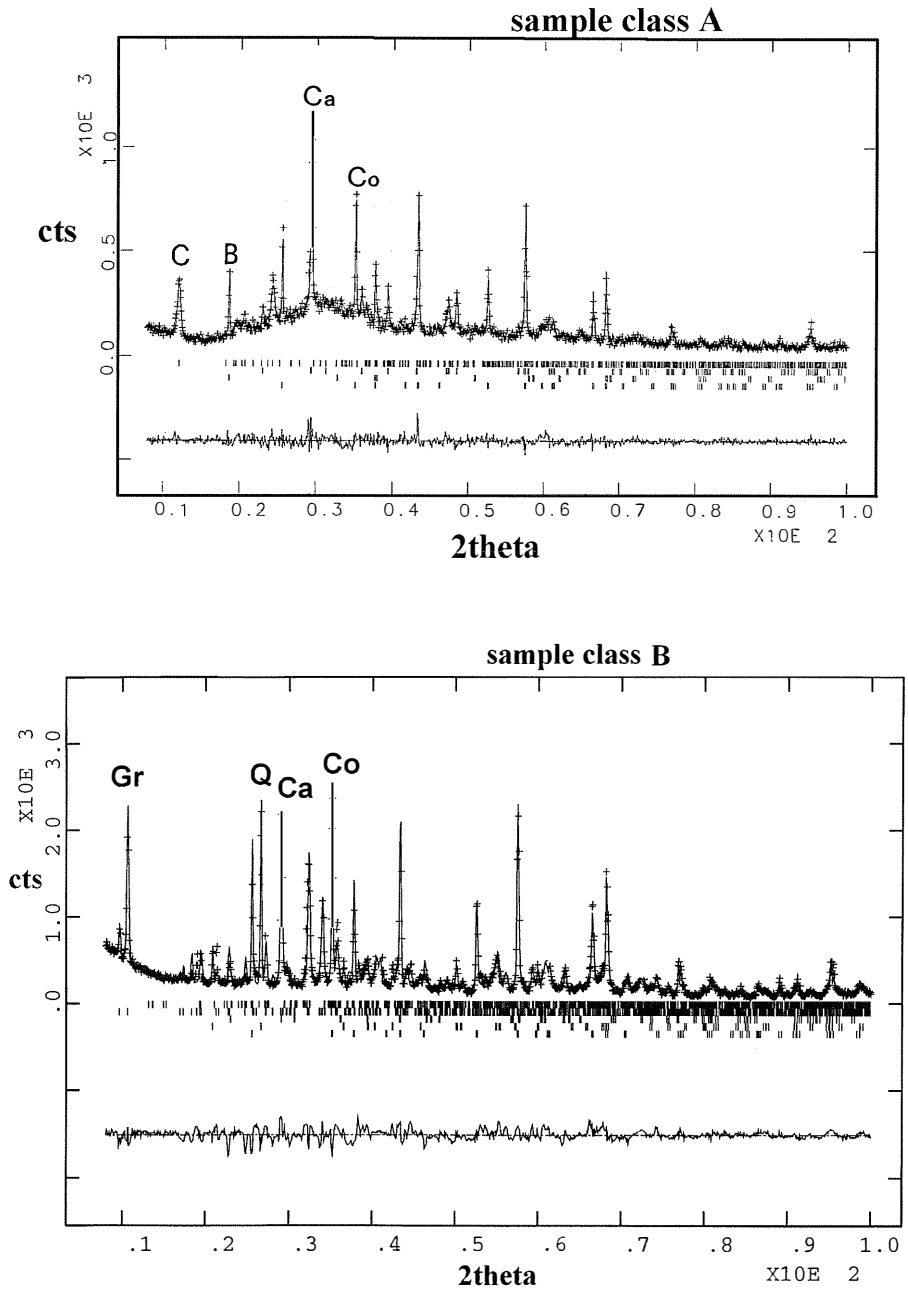



Fig. 2 – The observed and calculated patterns, and difference curve of the refinement of samples PARIS1 (*a*) and PARIS2 (*b*).

modelled using a pseudo-Voight function with one gaussian and one lorenzian coefficient. The lattice constants, the phase fraction, and coefficients corresponding to sample displacement and asymmetry were also refined. The total number of parameters refined at the same time in the last stages of the refinements for each pattern was 40.

The SEM (Scanning Electron Microscopy) analysis of the sample was performed on specimens of ca. 0.7×0.7×0.7 cm mounted on an Al holder and fixed with an Ag paste. Dehydration was performed under an IR lamp for 15 min and coating was performed using Au. A Philips XL 40/604 automated instrument was utilised with a vacuum of ca. $10^{-7}$ torr. In order to give a statistical significance to the observations, 20 frames were collected for each specimen. Frames were collected using a beam size of 5 mm and an intensity of 25 kV.

## RESULTS AND DISCUSSION

Fig. 2*a* and 2*b* show the observed (crosses), calculated (continuous line) and difference curve (bottom line) of the refined patterns. A good agreement between the calculated and observed patterns is evident at low angle as well as at high angle. The agreement factors for the Rietveld refinements are: Rwp=9% and $\chi^2$=2.1 for PARIS1 and Rwp=11% and $\chi^2$=3.1 for PARIS2, respectively. The results of the quantitative analysis of the two samples are reported in Table 1.

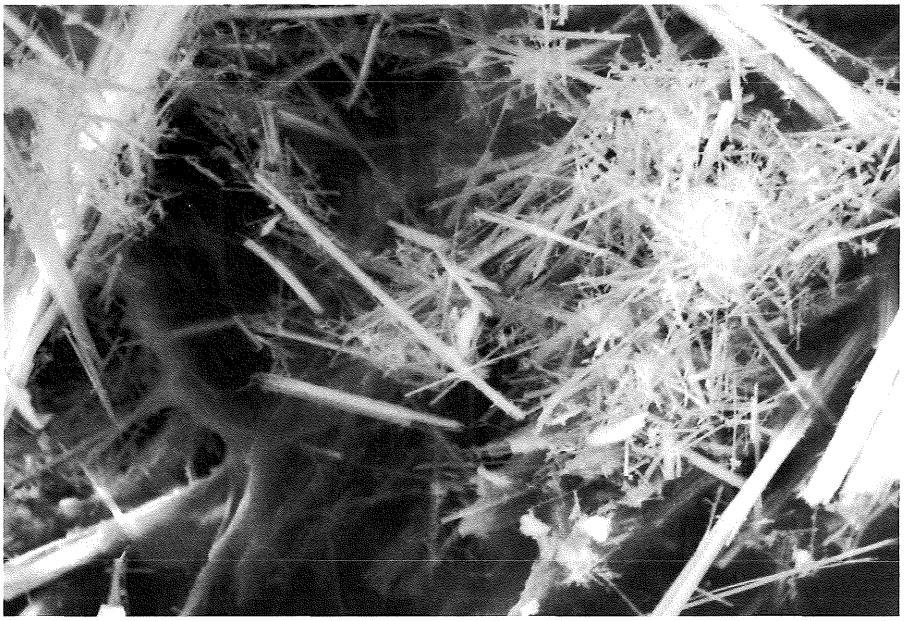
Fig. 3 shows the raw samples from the wall (a) and ceiling (b) of the University. PARIS1 is composed of an intergrowth of free glass fibers (slag wool) and asbestos fibers plus some grains of crystalline material (mainly calcite). PARIS2 is composed of amphibole asbestos fibres interdispersed in a matrix of glass fibres. Qualitatively fibres looking rigid and brittle are glassy wool, fibres which are rolled with an *hair-like* shape are asbestos fibres.

As it was expected the content of asbestos phases is larger than the maximum amount allowed by law. Thus, the whole building was reasonably evacuated in 1994 and a drastic plan of intervention is demanded. Among the various solutions, the neutralisation of the asbestos containing material can be performed *in situ* by spraying an epoxy resin on walls and ceiling which is capable to penetrate and cement the fibres, preventing its dispersion in air. Such intervention was described in detail in Gualtieri (1998) and Gualtieri (1999).

TABLE 1

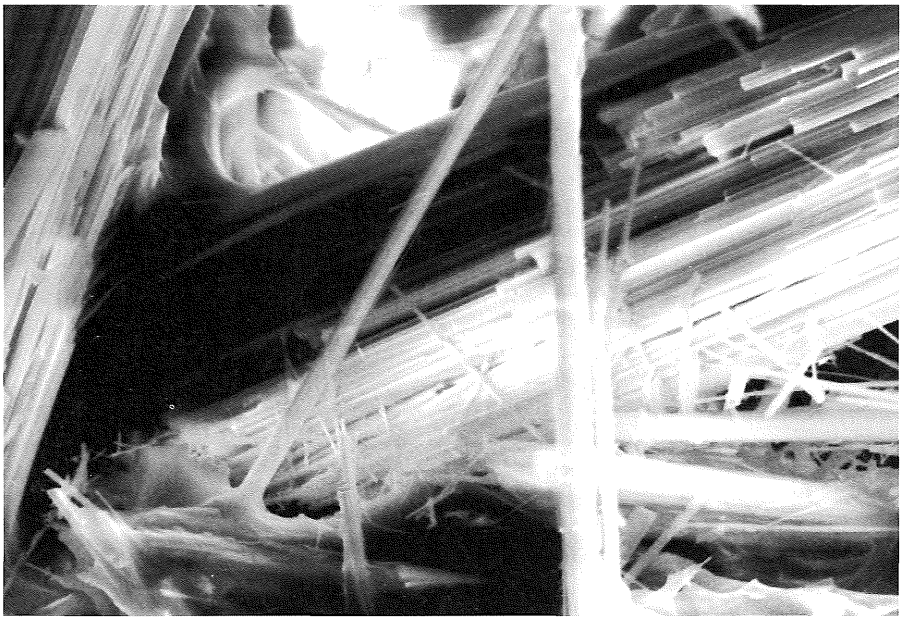
*Results of the quantitative analysis of the samples PARIS1 and PARIS2.*

| phase | Paris1 | Paris 2 |
|---|---|---|
| *Chrysotile* | 6.3(6) | – |
| *Grunerite* | – | 9.5(9) |
| *Brucite* | 0.6(2) | – |
| *Calcite* | 6.7(5) | 5.0(6) |
| *Quartz* | – | 0.5(2) |
| *Glass* | 86(1) | 85(1) |



*a*

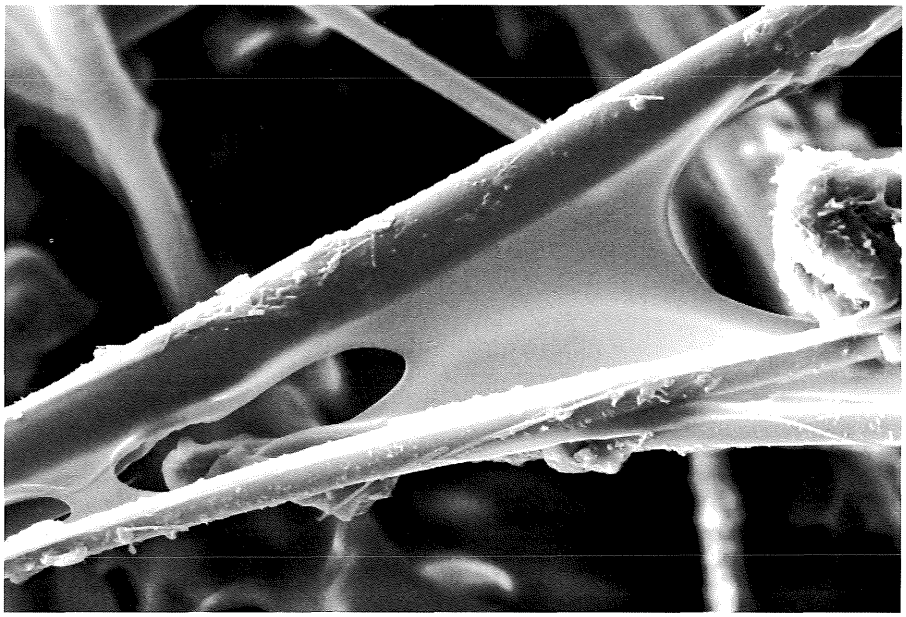
----- = 5 μm



*b*

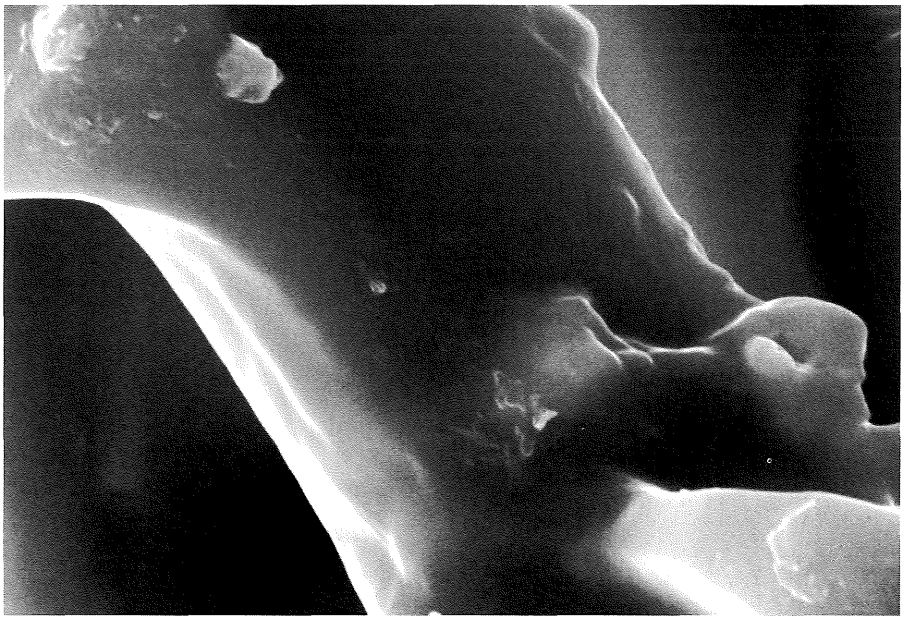
----- = 2 μm

Fig. 3 – SEM image of the untreated specimen sampled at the University of Jussieu Paris. Sample (*a*) is PARIS1, sample (*b*) is PARIS2.



*a*

----- = 5 μm



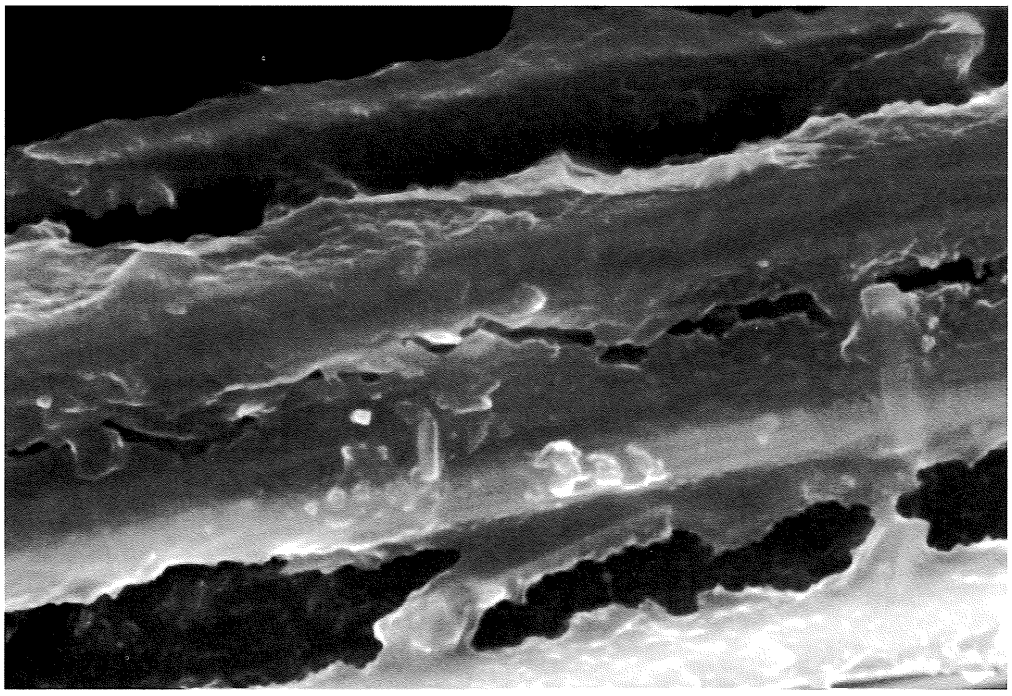
*b*

---------- = 1 μm

Fig. 4 – SEM magnified images of PARIS1 after the interaction with the epoxy resin at a different magnification showing a clear change in the microstructure of the material.

As discussed by Harington (1962), hemolitic activity of chrysotile asbestos is blocked by chelators, particularly those with affinity for Mg. There are a number of groups that may modify the surface chemistry of chrysotile fibers to reduce they cytoxicity. Although the monoionic polymer polyvinyl-2-pyridine N-oxide (PVPNO) does not passivate chrysotile, some ecidic, water soluble polymers, such as carboxymethylcellulose (CMC) are effective (Schnitzer, 1974). Brown *et al.* (1990) published a report in which they describe the modification of the surfaces of amosite fibers (mostly cummingtonite-grunerite) with octyldimethyl-chlorosilane ($C_8$) or octadecyl-methylchlorosilane ($C_{18}$) through *in vivo* studies showing that fibers coated with $C_{18}$ chains were dramatically less active at producing tumors. Besides, Pott *et al.* (1989) reports that actinolite with 2-polyvinylpyridine-N-oxide hydrogen bonded to its surface produced fewer mesotheliomas with a longer latency period than actinolite without polymer. For these reasons the utilisation of an epoxy resins should also benefit for the neutralisation of the surface activity of asbestos. The investigated specimen were treated by epoxy resin (see description in Gualtieri 1999) and observed by SEM after the treatment in order to put into evidence the differences in the morphology and microstructure of the materials *before* and *after* the interaction with the epoxy resin.

Fig. 4*a,b* are magnified image of PARIS1 after the treatment with the epoxy resin. It is evident a change in the micro-structure of the material which is now a diphasic material where the fibres and the crystals are now cemented by the resin. Fibres are thus captured in a solid matrix which inhibits the dispersion in air medium. The same applies for sample PARIS2 (fig. 5).



---------- = 2 μm

Fig. 5 – SEM magnified images of PARIS2 after the interaction with the epoxy resin.

It is possible to say that a proper *in situ* intervention using an epoxy resin modifies the nature of the asbestos containing materials which do not release free fibres in air and thus restore a safe environment to the scientific community of the University. A plan of intervention on an industrial scale has been proposed by AXSON Technologies, a France enterprise which produces the epoxy resin for the treatment. The purpose is still under discussion by the France Government.

## CONCLUSIONS

In the present work we successfully applied the combined Rietveld-RIR for the quantification of chrysotile asbestos in bulk materials from the main building of the University of Jussieu (Paris, France). The addition of an internal standard (RIR) is necessary to overcome the matrix (glassy, clay, heterogeneous) effects and better account for the structural disorder of chrysotile. The importance of the study is paramount for either intervention plans and biological studies. It was demonstrated that a proper intervention *in situ* with an epoxy resin modifies the nature of the asbestos materials which do not release free fibres in air and thus restoring a safe environment. In the specific case, quantitative characterisation of the material is necessary for the development of the proper composition of the epoxy resin since composition is terms of viscosity and solvent addition is strictly dependent upon the mineralogical composition.

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## REFERENCES

BHANDARI, R.K. and DHARIYAL, K.D. (1990) — *Asbestos and associated health hazards*, Central Building Res. Inst. Roorkee, Oxford and IBH Pub. Co. PVT. LTD New Delhi, 54 pp.

BISH, L.B. and POST, J.E. (1993) — *Quantitative mineralogical analysis using the Rietveld full-pattern fitting method.* Am. Min., **78**, 932-940.

BROWN, R.C., CATHEW, P., HOSKINS, J.A., SARA, E., and SIMPSON, C.F. (1990) — *Surface modification can affect the carcinogenicity of asbestos.* Carcinogenesis, **11**, 1883-1885.

DAVIS, B.L., KATH, R., and SPILDE, M. (1990) — *The reference Intensity ratio: Its measurement and significance.* Powder Diff., **5**, 76-78.

FENTON, H.J.H. (1984) — *Oxidation of tartaric acid in presence of iron.* J. Chem. Soc., **65**, 899-910.

GUALTIERI A.F. and ARTIOLI G. (1995) — *Quantitative determination of chrysotile asbestos in bulk materials by combined Rietveld and RIR methods*, Powder Diffraction, **10(4)**, 269-277.

GUALTIERI A.F. (1998) — *A solution to the asbestos problem: the in situ impregnation by an epoxy resin.* Reunion «Le traitment de l'amiante in situ» Press Club de France, Paris, 29 giugno 1998.

GUALTIERI A.F. (1999) — *A solution for the full impregnation of asbestos: the use of an epoxy polymer resin*, J. App. Polymer Science, submitted.

HARINGTON, J.S. (1962) — *Natural occurrence of oils containing 3,4-benzpyrene and related substances in asbestos.* Nature, **193**, 43-45.

HOCHELLA, M.F. (1993) — *Surface chemistry, structure, and reactivity of hazardous mineral dusts.* in *Health effects of mineral dusts.* G.D. Guthrie and B.T. Mossman eds. Min. Soc. of Am. **28**, 275-308.

HUME, L.A., and RIMSTIDT, J.D. (1992) — *The biodurability of chrysotile asbestos.* Am. Min., **77**, 1125-1128.

JOHAN, Z.A., GONI, J., SARCIA, C., BONNAUD, G., and BIGNON, J. (1976) — *Influences de certains acides organiques sur la stabilite du reseau du chrysotile.* 6th Cong. Int. Geoch. Organique Technip. Ed. Paris, 883-903.

JONES, A.D., VINCENT, J.H., MCINTOSH, C., MACMILLAN, C.H., and ADDISON, J. (1989) — *The effect of fiber durability on the hazard potential of inhaled chrysotile asbestos fibers.* Exper. Path., **37**, 98-102.

JURAND, M.C., BIGNON, J., SEBASTIEN, P., and GONI, J. (1983) — *Leaching of chrysotile asbestos in human lungs.* J. Colloid. Interf. Sci.», **95**, 1-9.

KANE, A.B. (1993) — *Epidemiology and pathology of asbestos-related diseases*, in *Health effects of mineral dusts.* G.D. Guthrie and B.T. Mossman eds. Min. Soc. of Am. **28**, 347-359.

KUNZE, G. (1961) — *Antigorit. Strukturtheoretische Grundlangen aun ihre praktische bedetung für die*

*weiters serpentin-forschung*, Fortschr. Min. **39**, 206-324.

LANGER, A.M., RUBIN, I.B., SELIKOFF, I.J., and POOLEY, F.D. (1972) — *Chemical characterization of asbestos boby cores by electron microprobe analysis.* J. Hystochem. Cytochem., **20**, 723-734.

LARSON A.C. and VON DREELE R.B. (1999) *GSAS: General structure analysis system.* Report LAUR 86-748. Loa Alamos National Laboratory, NM 130 pp.

LIDDELL, D. (1991) — *Exposure to mineral fibers and human health: historical backgroud.* In Mineral fibers and health, D. Liddell and K. Miller Eds. Boca Raton CRC Press Florida Cap 1.

MELLINI, M., FERRARIS, G. and COMPAGNONI, R. (1985) — Carlosturanite*: HRTEM evidence of a polysomatic series including serpentine.* Am. Min., **70**, 773-781.

NATHAN, C.F. (1987) — *Secretory products of macrophages.* J. Clin. Invest., **79**, 319-326.

POTT, F. (1987) — *Problems in defining carcinogenic fibres.* Ann. Occup. Hygiene», **31**, 799-802.

POTT, F., ROLLER, M., ZIEM, U., REIFFER, F.J., BELLMANN, B., ROSENBRUCH, M., and HUTH, F. (1989) — *Carcinogenicity studies of natural and man-made fibers with the intraperitoneal test in rats.* in *Non occupational exposure to mineral fibers.* Bignon, J. Ed. IARC Sci. Publ. No 90, Lyon France, 173-179.

RIETVELD, H.M. (1969) — *A profile refinement for nuclear and magnetic structures.* J. App. Cryst., **2**, 65-71.

ROSS, M., NOLAN R.P., LANGER, A.M., COOPER, W.C. (1993) — *Health effects of mineral dusts other than asbestos.* in «Health effects of mineral dusts». G.D. Guthrie and B.T. Mossman eds. Min. Soc. of Am., **28,** pp 361-401.

RUCKLIDGE, J.C. and ZUSSMAN, J. (1965) — *The crystal structure of the serpentine mineral lizardite* $Mg_3Si_2O_5(OH)_4$, Acta Cryst., **19**, 381-389.

SCHNITZER, R.J. (1974) — *Modification of biological surface activity of particles.* Environ. Health Oers., **9**, 261-266.

STANTON, M.F., LAYARD, M., TEGERIS, A., MILLER, E., MAY, M., MORGAN, E., SMITH, A. (1981) — *Relation of particle dimension to carcinogenicity of amphibole asbestos and other fibrous minerals.* J. Nat'l Cancer Inst., **67**, 965-975.

THOMASSIN, H., GONI, J., BAILLIF, P., TOURAY, J.C. (1976) — *Etude par spectrométrie ESCA des premiers stades de la lixiviation du chrysotile en milieu acide organique.* C.R. Acad. Sci. Paris, **283**, 131-134.

TITULAER, M.K., VAN MILTEBURG, J.C., JANSEN, J.B., and GEUS, J.W. (1993) — *Characterization of tubular chrysotile by thermoporometry, nitrogen sorption, drifts and TEM.* Clays and Clay Min., **4**, 496-513.

VEBLEN, D.R. and WYLIE, A.G. (1993) — *Mineralogy of amphiboles and 1:1 layer silicates.* in *Health effects of mineral dusts.* G.D. Guthrie and B.T. Mossman eds. Min. Soc. of Am. **28**, 61-137.

WEITZMAN, S.A., and GRACEFFA, P. (1984) — *Asbestos catalyzes hydroxyls and superoxide radicals generation from hydrogen peroxide.* Arch. Biochem. Biophys., **228**, 373-376.

WHITTAKER, E.J.W. (1956) — *The structure of chrysotile. II. Clinochrysotile.* Acta Cryst., **9**, 855-861.

WICKS, F.J., and O'HANLEY, D.S. (1988) — *Serpentine minerals: structures and petrology.* in *Hydrous Phyllosilicates* S.W. Bailey ed. Min. Soc. of Am. **19**, 91-188.

YOUNG, R.A. (1993). *The Rietveld method.* Oxford University Press, 235 pp.