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Measurement of Asbestos and Other Fibers

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Introduction

The term fiber has been applied to a wide variety of particles having an elongated shape, i.e., one particle dimension significantly greater than the other two. Because of this elongation, fibers can have aerodynamic and other properties quite different from more compact particles. Certain fibers have several unique properties that make them not only useful from a commercial standpoint, but also important from a health standpoint. Asbestos, for instance, includes six commercial fibrous minerals that have high tensile strength, chemical resistance, and heat resistance. These properties have made asbestos useful in a variety of products, including friction materials, high-temperature insulating materials, acoustic insulating materials, fire proof cloth and rope, and floor tiles. While the bulk materials in these products consist primarily of macroscopic-sized fibers, many of them can release long, thin fibers into the air.

A variety of materials can be considered fibers from an aerosol behavior standpoint. Besides asbestos, other mineral fibers exist in nature. Several materials, including glass and mineral slags, have been melted and spun into fibers. Ceramic materials have similarly been spun into fibers, as well as grown by chemical and vapor crystallization. Carbon and graphite fibers are produced commercially for high-strength products. Organic fibers, such as cotton, wood, and other cellulosic materials, are widely present in the environment, both from commercially produced materials as well as natural sources. Besides cylindrical particles that have relatively high strength, chains of particles also may behave as fibers and can serve as models for some aerodynamic properties. Some organic materials can be crystallized into well-defined fibrous shapes and can be used to test theories of fiber aerodynamic behavior.

Asbestos fiber aerosols have been closely associated with several diseases, such as asbestosis (a fibrosis of the lung), mesothelioma (cancer of the lining around the lung), and lung cancer (NIOSH 1976*a*). Thus, the fibers that can enter the respiratory system are of greatest concern. In addition, the high rate of

1. Mention of product or company names does not constitute endorsement by the National Institute for Occupational Safety and Health.

prompted the statement that there "is ce for a threshold or a 'safe' level of os exposure" (NIOSH 1990*a*). The sness of the diseases has driven mea- ent technology to provide maximum ity and accuracy for measuring asbes- ols. Other airborne fibers may have or more of the same physical and chem- properties as asbestos. In some cases, xposures and/or animal studies sug- the disease potential of these fibers. there is concern regarding the health fibers other than asbestos. While the al properties of mineral fibers have a store of knowledge about physical logic properties, it is the health con- have largely driven the technology ing and quantifying airborne con- s of fibers. Thus, much of the fiber esearch and measurement capability to the ability of microscopic-sized to enter and deposit in the human ory system.

dimensions of fibers in aerosols can a wide range. For asbestos, diameters s small as 0.025 μm (Langer, Mackler, ley 1974), while lengths can be sev- ndred micrometers. The dimension tions depend on the fiber type as well w the fibers were comminuted from material. The magnitude of disparity en length and diameter often makes it lt to make accurate size distribution ments. Several protocols, using types of microscopes, have been to deal with fiber distribution ment. Other types of instruments, ily using light scattering properties, been developed to characterize fibers. er, these instruments usually provide an approximate indication of fiber ns.

that the following discussion, except herwise indicated, will deal largely surement of aerosolized fibers, gen- ble only with a microscope, and not the macroscopic or bulk properties of eous material. Since asbestos has been st intensely studied type of fiber, many ts will relate to this material. Many issues regarding asbestos mineralogy, health effects, and measurement techniques are discussed in a review by Walton (1982); further reviews in Environmental Health Perspectives are introduced by Langer, Mackler, and Pooley (1974) and Dement (1990). Additional topics are presented in the books by Selikoff and Hammond (1979), Rajhans and Sullivan (1981), Michaels and Chissick (1979), Chissick and Derricott (1983), and Holt (1987). Similar reviews have been carried out for man-made fibers (NIOSH 1976*a*, 1977; IARC 1988).

FIBER SHAPE

The behavior of fibers suspended in a gas is a function of the fiber dimensions. Assuming either a cylindrical or prolate spheroidal shape, these dimensions can be defined by two parameters, length and diameter. A third parameter β is often invoked to indicate the fibrosity or aspect ratio, i.e., the ratio of the length to the diameter. However, often, real fibers meet neither the ideal cylindrical assumption nor the prolate spheroid shape assumption. Glass or mineral fibers are often nearly cylindrical, but even these fibers frequently display curvature along their length as well as bulbous or jagged ends. Asbestos fibers are formed from a unique crystal habitat in which the bulk mineral has slip planes in two directions, but only rarely in the third. This results in a propensity to produce particles that can split longitudinally to produce thinner and thinner fibers, ultimately resulting in fibrils about 0.025–0.05 μm diameter. Thus, while some asbestos fibers exhibit a nearly ideal cylindrical shape, others may have various combinations and degrees of splayed ends, curvature, splitting, noncircular circumference, etc. For instance, the magnetically aligned chrysotile fibers in Fig. 25-1 show many of these characteristics. In spite of these possible variations in shape, fibers are still most often characterized just by length and diameter.

Distributions of natural fibers are rarely monodisperse in diameter and even more rarely in length. This has made it difficult to provide adequate calibration for instruments

FIGURE 25-1. Scanning Electron Micrograph (1500×) of an Aqueous Sample of Magnetically Aligned UICC Canadian Chrysotile Collected on a 0.1 μm Pore Size Filter. (Source: Timbrell 1973.)

that attempt to measure fibers as well as to perform measurements of fiber toxicity as a function of fiber dimension. Distributions of fibers can often be described by a two-dimensional (length L and diameter W) lognormal distribution (Cheng 1986; Schneider, Holst, and Skotte 1983), i.e., ln L and ln W are each distributed normally. The probability density function is given by

$$f(L, W) = \frac{1}{2\pi\sigma_W\sigma_L\sqrt{1-\tau^2}\,LW} \times \exp\left[-\frac{A^2 + B^2 - 2\tau AB}{2(1-\tau^2)}\right] \quad (25\text{-}1)$$

where

$$A = (\ln W - \mu_W)/\sigma_W$$

$$B = (\ln L - \mu_L)/\sigma_L$$

μ_i and σ_i^2 are the mean and variance, respectively, of the natural logarithm of L and W and τ is the correlation between ln L and ln W. The five parameters μ_L, μ_W, σ_L, σ_W, and τ are needed to define completely a two-dimensional size distribution. The two-dimensional lognormal size distribution has the properties that the marginal and the conditional distributions are lognormal (Holst and Schneider 1985). The former property indicates that the length and diameter distributions are each, separately, lognormal. The latter indicates that functions of length and diameter of the form kW^pL^q, where k, p, and q are constants, are also lognormal. Such functions include the aspect ratio, surface area, volume, and aerodynamic diameter. Deviations from lognormality can sometimes be attributed to artifacts in sampling or analysis or to multiple aerosol generation sources.

Many fiber distributions reported in the literature include the length and diameter means and variances but, unfortunately, not τ. However, if the original data are reported in a table as a function of both length and diameter, the correlation term can be estimated (Cheng 1986). Most fiber distributions have positive τ, suggesting that diameter often increases with length.

There have been measurements of a variety of fibrous aerosols. Table 25-1 lists the results of some examples. Some of these materials have been generated for toxicity studies, some have been measured in environmental studies, while others have been generated as calibration materials.

FIBER BEHAVIOR

Translational Motion

As with other aerosol particles, fiber dimensions can cover a relatively wide range; the smaller fibers are affected primarily by diffusional forces, while the larger ones are primarily affected by flow shear, inertial, and gravitational forces. Fiber behavior has been observed and theoretically calculated for several fibrous shapes, including prolate ellipsoids, cylinders, and chains of spheres. The motion of these various shapes, generally, differs only slightly.

Fiber behavior differs, depending upon whether the major axis is oriented parallel to or perpendicular to the direction of the motion relative to the surrounding gas (Fig. 25-2a and b). The drag on a fiber is greatest when it is oriented perpendicular to the flow of the surrounding gas. Fiber behavior is

TABLE 25-1 Examples of Measured Fiber Size Distributions

Material	Diameter (μm)	σ_g	Length (μm)	σ_g	MMAD (μm)	σ_g	Measurement Technique
Chromoglycic acid[1]	0.205	1.58	2.09	1.83			SEM
					0.65	1.88	Cascade impactor
Soot cane silicate[2]	0.3–1.5*		3.5–65*				TEM
Caffeine[3]	1.13	1.08	5.55	1.12			SEM
					2.1	1.1	Sedimentation
Ceramic fibers[4]							
Sample a	0.5		10.1				TEM
Sample b	0.66		8.3				TEM
Sample c	0.98		22.8				TEM
Chrysotile[5]							
Preform ring	0.13	2.15	1.6	2.7			TEM
Fart dressing	0.08	1.92	1.0	2.4			TEM
Core press	0.13	1.94	1.5	2.2			TEM
Crocidolite[6]							
Mining/Mill**	0.08–0.10	1.86–2.08	0.98–1.25	2.30–2.55			TEM
Manufacturing	0.04	1.58	0.54	2.32			TEM
Fibrous glass[7]							
Code 100	0.12	1.8†	2.7	2.2†			TEM
Code 110	1.8	1.7†	26	2.0†			TEM
Iron oxide chains[8]							
	0.059	1.1	1††	2.0			TEM
					0.32	1.11	Centrifuge

Source: 1. Chan and Gonda (1989); 2. Boeniger et al. (1988); 3. Vaughan (1990); 4. Rood (1988); 5. Rood and Scott (1989); 5. Pinkerton et al. (1983); 6. Hwang and Gibbs (1981); 7. Timbrell (1974); 8. Kaspar and Shaw (1983)

* These values represent the range of particle sizes rather than the median diameters

** These values represent the range of several measurements that produced similar results

† Estimated from data in reference

†† Estimated from mean chain length of 22 primary particles

often described in terms of a combination of the two orientations. While the difference in drag between the two orientations is typically about 15–30%, it can be difficult to determine the contribution of each orientation in experimental systems. At low Reynolds number (Re_p), fiber orientation will be stable (disregarding Brownian rotation) and not change due to translational motion, e.g., during gravitational settling (Gallily 1971). In addition, fibers settling in still air will not settle exactly in the direction of the gravitational force, but will drift somewhat due to orientation (Weiss, Cohen, and Gallily 1978). Larger fibers, with Re_p greater than about 0.01, will settle with their major axis oriented perpendicular to the direction of motion (Fig. 25-2a). With increasing Re_p ($Re_p > 100$), longer fibers ($\beta > 20$) are unstable in the perpendicular orientation, but there is an increasing trend toward instability (Clift, Grace, and Weber 1978, 154).

The aerodynamic diameter d_a of a prolate spheroid has been calculated from

$$d_a = d_f \sqrt{\frac{\rho_f \beta}{\rho_0 \chi}} \tag{25-2}$$

by using the numerical shape factor χ of a prolate ellipsoid of revolution (Fuchs 1964, 37); d_f is the physical fiber diameter, ρ_f is the fiber density and ρ_0 is unit density. A cylinder with the same diameter and length as a prolate ellipsoid has 3/2 times greater volume and mass. Therefore, for cylinders with the same axial dimensions, the right-hand side of Eq. 25-2 must be multiplied by $(3/2)^{1/3}$ or $(3/2)^{1/2}$ to obtain the equivalent-volume diameter or equivalent-weight diameter, respectively

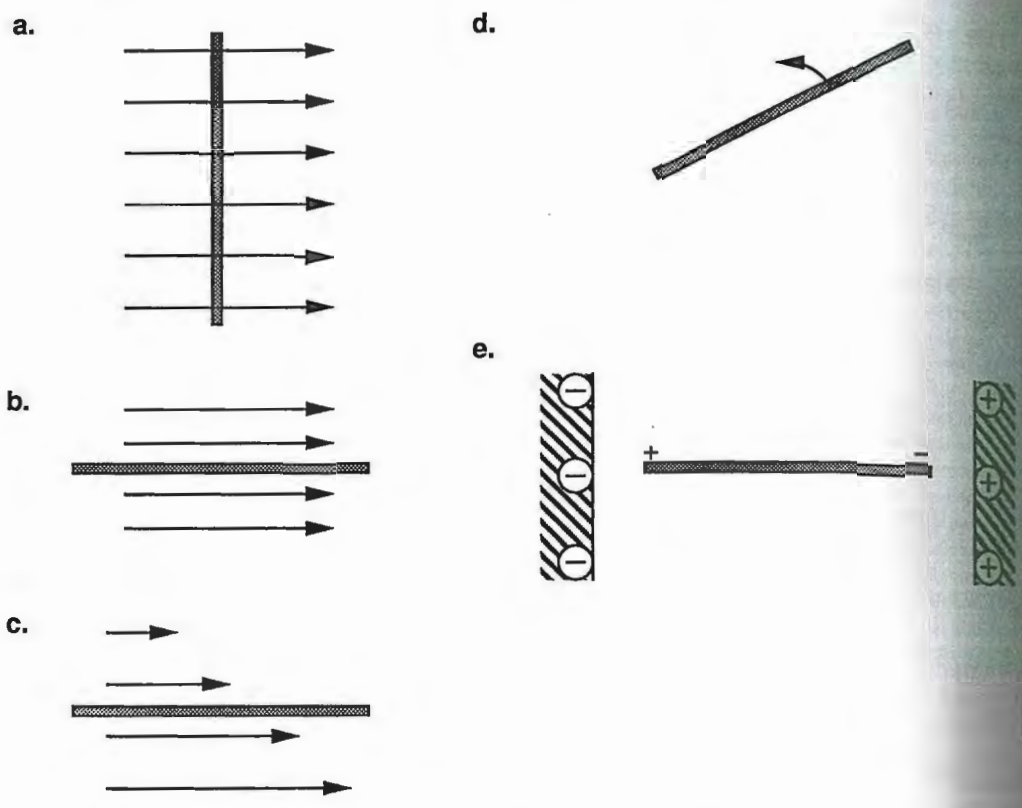

FIGURE 25-2. Fiber Alignment in Various Force Fields. (a) Fiber Aligned Perpendicular to Relative Gas Motion. This is the Preferred Orientation During Gravitational Settling and Acceleration at $0.01 < Re < 100$ in the Absence of Other Forces. (b) Fiber Parallel to Relative Gas Motion. Fiber Motion is Often Treated as a Combination of Cases a and b. (c) Fiber is Readily Oriented Parallel to, or at Some Small Angle to, the Direction of Shear Flow in the Suspending Gas Medium. (d) Small Fibers Governed by Diffusional Forces may Exhibit Completely Random Orientation. (e) Conductive Fibers are Aligned Parallel to an Electric Field. Many Fibers are also Aligned in a Magnetic Field, Usually Parallel to the Field Lines, Though They may be Aligned Perpendicular to the Field or, for Some Materials, at Some Intermediate Angle.

(Griffiths and Vaughan 1986). For motion parallel and perpendicular to the fiber major axis, the respective shape factors $\chi_{\parallel}$ and $\chi_{\perp}$ are (Stöber 1972; Kasper 1982):

$$\chi_{\parallel} = \frac{4(\beta^2-1)}{3}\Bigg/\left\{\frac{2\beta^2-1}{\sqrt{\beta^2-1}}\ln(\beta+\sqrt{\beta^2-1})-\beta\right\} \tag{25-3}$$

$$\chi_{\perp} = \frac{8(\beta^2-1)}{3}\Bigg/\left\{\frac{2\beta^2-3}{\sqrt{\beta^2-1}}\ln(\beta+\sqrt{\beta^2-1})+\beta\right\} \tag{25-4}$$

(see Fig. 25-2a and b). Note that a dynamic shape factor χ_d is also defined that is related to the numerical shape factor for prolate spheroids by $\chi = \chi_d \beta^{-1/3}$. The dynamic shape factor is applied when the equivalent-volume diameter of the particle is used rather than the physical diameter.

An alternate approach for directly calculating the aerodynamic diameter of cylinders (Cox 1970) gives similar results:

$$d_{a,\parallel} = d_f\sqrt{\frac{9\rho_f}{4\rho_0}\left[\ln(2\beta)-0.807\right]} \tag{25-5}$$

and

$$d_{a,\perp} = d_f \sqrt{\frac{9\rho_f}{8\rho_0}\,[\ln(2\beta) + 0.193]} \qquad (25\text{-}6)$$

Others have also provided formulae for prolate ellipsoids and cylinders (Gonda and Khalik 1985; Prodi et al. 1982).

If fibers are not preferentially oriented by a drag force or any other alignment force, the orientation may be completely random. Then, a single average shape factor $\bar{\chi}$ that is a function of the two shape factors noted above may be used, i.e.,

$$\frac{1}{\bar{\chi}} = \frac{1}{3\chi_{\parallel}} + \frac{2}{3\chi_{\perp}} \qquad (25\text{-}7)$$

In the presence of air gradients, the fiber will experience a torque until the fiber is oriented parallel to the direction of the shear force (Fig. 25-2c). Thus, a fiber settling in a horizontal laminar flow will tend to be oriented horizontally (parallel to the shear). However, the fiber will experience a periodic instability and perform a "flip". This instability is a function of fiber dimensions as well as the flow gradients. Under such conditions, the aerodynamic diameter is not strictly an inherent property of the particle and depends on the experimental conditions of measurement (Gallily and Eisner 1979).

Inertial separation is commonly used for particle separation and sizing, e.g., in impactors and cyclones. In such systems where flow conditions are rapidly changing, the fiber mechanics are governed by initial orientation and flow relaxation time besides the usual parameters observed for spherical particles (Gallily et al. 1986). For instance, fibers with large rotational inertia (especially long fibers) may not orient completely or may over-rotate in passing through a nozzle. Fiber behavior under such a situation may be only approximately defined by the Stokes number or other nondimensionalized parameters.

The experimental measurement of fiber deposition has been carried out in horizontal elutriators (Gallily and Eisner 1979; Griffiths and Vaughan 1986; Iles 1990), centrifuges (Stöber, Flachsbart, and Hochrainer 1970; Martonen and Johnson 1990), impactors (Burke and Esmen 1978; Prodi et al. 1982), and cyclones (Fairchild et al. 1976; Iles 1990) for a variety of fiber types.

The extended shape also means that interception during translational motion plays a larger role in fiber deposition than for compact particles. However, the alignment of fibers by shear flow often reduces the effect of length on interception.

Rotational Motion

The rotational mobility B_r of a high aspect ratio ellipsoid can be approximated by (Lilienfeld 1985)

$$B_r = \frac{3[2\ln(2\beta - 1)]}{2\pi\eta L^3} \qquad (25\text{-}8)$$

where η is the viscosity of the gas. Note that the rotational mobility is a strong inverse function of fiber length. Similarly, the rotational diffusion coefficient D_r for fibers is also a strong function of fiber length:

$$D_r = \frac{3kT}{\pi\eta\beta L^3}(\ln 2\beta - \delta) \qquad (25\text{-}9)$$

where k is the Boltzmann constant, T is the temperature and δ is 1.4 for aspect ratios β larger than ten. Rotational mobility can be estimated by measuring the rate of relaxation after removal of an electrostatic alignment force (Cheng et al. 1991).

Behavior in the Transition Regime

Under molecular bombardment, fibers can exhibit both rotational diffusion as well as translational diffusion. Such fibers are likely to be randomly oriented (Fig. 25-2d). As for fibers in the Stokes regime, it is often convenient to separate the translational motion of fibers into motion in which the major axis is parallel to the direction of motion and another in which the major axis is perpendicular to the translational motion. The diffusion of fibers is described by the diffusion coefficient

D_f (cm^2/s):

$$D_f = BkT = \frac{kT}{f} = \frac{kTC_f}{f^0} \qquad (25\text{-}10$$

where B is the fiber mobility (dyn cm/s), and f^0 is the drag per unit velocity of the fiber in the continuum regime and f is the drag per unit velocity of the fiber corrected for slip by the fiber slip correction factor C_f. A theory for the slip correction factors for nonspherical particles is described by Dahneke (1973a, b, c; 1982).

Fiber diffusional behavior is usually treated as a modification of spherical particle diffusion using particle shape factors (Asgharian, Yu, and Gradon 1988). This approach has agreed well with the experimental diffusion coefficient measurement of fibers with mean diameters of 0.24–0.38 μm (Gentry et al. 1983). Diffusional coefficients of much smaller fibers have also been measured (Gentry, Spurny, and Soulen 1988), which show higher diffusion coefficients than expected.

As with the stagnant flow conditions for fibers in the continuum regime, fibers are expected to be randomly oriented unless affected by shear flow or other forces. Again, the longer the fiber, the more likely it is to be oriented by such forces.

Several studies have estimated the effects of various deposition mechanisms (diffusion, impaction, interception) to determine overall particle deposition in filters (Fu, Cheng, and Shaw 1990) and lung airways (Asgharian and Yu 1989; Balashazy, Martonen, and Hofmann 1990).

Charging

Theories for unipolar diffusion charging (Laframboise and Chang 1977) and bipolar diffusion charging (Wen, Reischl, and Kasper 1984) of fibers have been developed. Unipolar charging of fibers causes the charge of long, thin fibers to increase dramatically, though the electrical mobility of such fibers changes more slowly with aspect ratio (Yu, Wang, and Gentry 1987). Such a variation of mobility with fiber aspect ratios may allow the separation of fibers of different lengths.

Electric Field Effects

A fiber may be aligned in an electric field by an induced dipole in the fiber. This requires that charges in the fiber be separated so that the polarity is opposite to that of the surrounding electric field as shown in Fig. 25-2e. The charge separation from conduction is usually greater than that from polarization of the material. For charge separation to occur, the fiber must be sufficiently conductive so that the charges can migrate the length of the fiber in a reasonable time. Aerosol particles, even those consisting of a normally nonconducting material, can often be considered conductive because of their low capacitance and small dimensions (Fuchs 1964; Lilienfeld 1985). Surface impurities can also contribute to a particle's conductivity. Thus, an electric field of sufficient strength (1000–5000 V/cm) can overcome diffusional randomization and flow shear forces to align most types of fibers, including relatively nonconductive ones. For instance, electrostatically aligned zinc oxide fibers were used to modulate microwave radiation (Tolles, Sanders, and Fritz 1974).

When fibers and compact particles of the same aerodynamic diameter are charged under the same conditions, the fibers may have higher mobility than compact particles. Field studies of work environments suggested that fibers carried a charge proportional to fiber length (Johnston, Vincent, and Jones 1985). Other studies indicated that unipolar, charged particles can be separated according to aspect ratio (Griffiths, Kenny, and Chase 1985; Yu, Wang, and Gentry 1987).

Dielectrophoresis has also been investigated for separating fibers of different lengths (Lipowicz and Yeh 1989). Uncharged, conductive fibers may be separated according to length in a nonuniform electric field. Since the electrical mobility of charged fibers is generally higher than the dielectric mobility, such a separation must be carried out on fibers with low charge, in an ac electric field, or both.

An electrostatic enhancement of fiber deposition in lungs (conductive tubing) has been observed (Jones, Johnston, and Vincent [illegible]). Calculations support such an enhancement of sedimenting charged fibers (Chen and Yu 1990).

Magnetic Field Effects

If a suspension of fibers in a liquid or gas is subjected to a magnetic field, fibers with sufficient magnetic susceptibility will align at some angle to the field. Usually this angle is either 0° or 90°; some amphibole asbestos samples have fibers aligned at both angles. Timbrell (1975) developed a technique for preparing permanently aligned samples by allowing a suspension of fibers in 0.5% celloidin/amyl acetate to dry in a 5–10,000 G magnetic field. Several fiber types have been aligned by Timbrell (1972, 1973), including carbon fibers and the various types of asbestos. Fibers of silicon carbide, silicon nitride, and tungsten-cored boron did not align in similar fields.

Figures 25-3–25-5 contain the images of fibers magnetically aligned on a slide surface with light scattering patterns from magnetically aligned liquid suspensions of the same types of fibers. The direction of the magnetic field is shown in the figures. The scattering pattern has the main laser beam in the center, with the plane of scattering radiating in opposite directions. In Fig. 25-3, monodisperse diameter carbon fibers are all aligned parallel to the field so that a well-defined scattering pattern perpendicular to the field is produced. The crocidolite fibers in Fig. 25-4 are aligned the same way, but are not monodisperse. In other cases, the fibers are aligned perpendicular or both perpendicular and parallel to the magnetic field (e.g., Fig. 25-5), the latter resulting in two planes of scattering. A synthetic fluoroamphibole was observed to align at $\pm 65°$ to the magnetic field direction. The degree and direction of alignment has not been adequately explained; however, it appears to be more a function of the mineralogical source of the material rather than of the primary crystal structure.

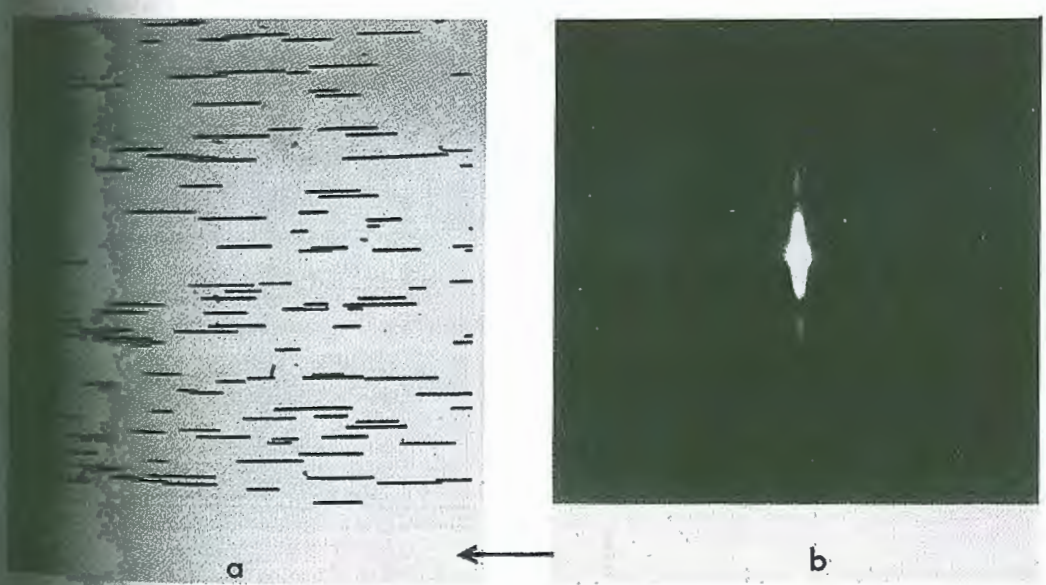

FIGURE 25-3. (a) Phase Contrast Microscope (PCM) Image of Magnetically Aligned Carbon Fibers Suspended in Celloidin on a Glass Slide. (b) Light Scattering Pattern from the Same Fibers in Aqueous Suspension. The Direction of the Magnetic Field is Indicated by the Arrow. Note the Monodisperse Diameter of the Fibers, Reflected in the Sharply Defined Scattering Pattern. (Source: Timbrell 1973.)

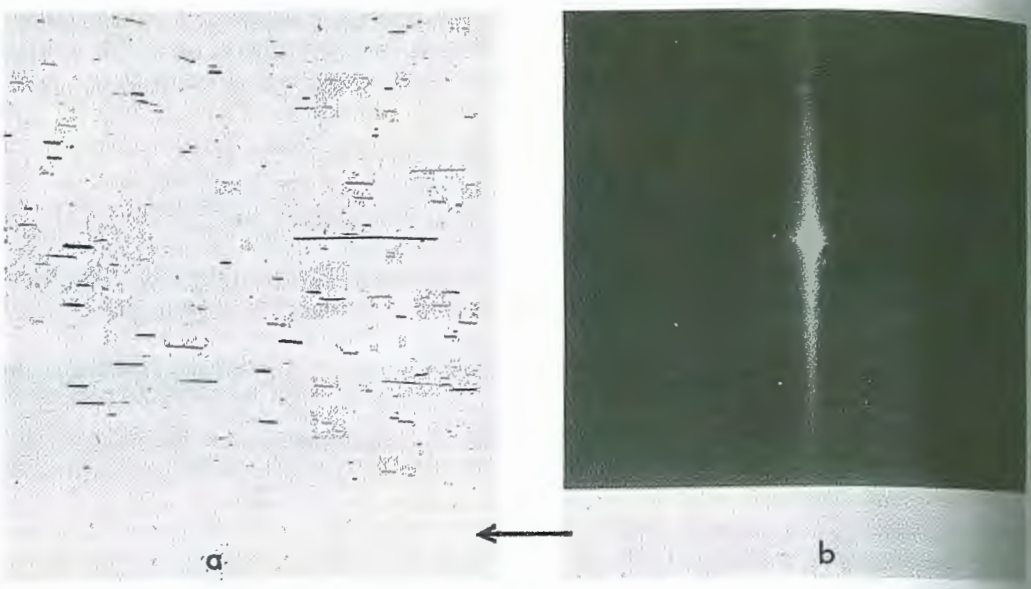

FIGURE 25-4. (a) PCM Image of Magnetically Aligned UICC Crocidolite Fibers Suspended in Celloidin on a Glass Slide. (b) Light Scattering Pattern from the Same Fibers in Aqueous suspension. The Direction of the Magnetic Field is Indicated by the Arrow. (Source: Timbrell 1973.)

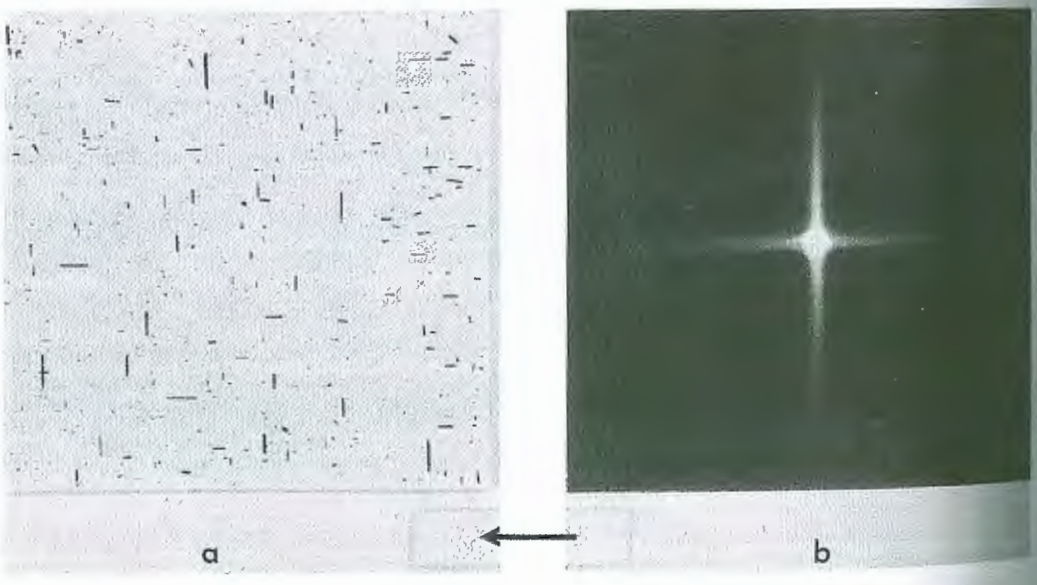

FIGURE 25-5. (a) PCM Image of Magnetically Aligned UICC Amosite Fibers Suspended in Celloidin on a Glass Slide. (b) Light Scattering Pattern from the Same Fibers in Aqueous Suspension. The Direction of the Magnetic Field is Indicated by the Arrow. (Source: Timbrell 1973.)

Thus, Ugandan tremolite was observed to align perpendicular to the magnetic field, while Zululand tremolite aligned parallel to the field (Timbrell 1973).

Light Scattering

If a flashlight beam were shone on a glass rod such that the light beam was perpendicular to the rod axis, one would expect the refracted and scattered light to be dispersed into a plane perpendicular to the rod's axis. Light scattered and refracted from microscopic fibers produces a similarly unique pattern. Such scattering patterns from magnetically aligned fibers in an aqueous suspension are given in Figs. 25-3–25-5. If the fiber is not perpendicular to the light beam, the light scattering pattern is not as conveniently constrained to a plane, becoming a cone of light. The details of light scattering from infinitely long cylinders, ellipsoids, and several other regular elongated shapes can be described by the Mie theory (Kerker 1969; Van de Hulst 1957). As with scattering from spherical and compact particles, the scattering from fibers with diameters larger than the light wavelength is concentrated in the forward direction. The scattering from smaller-diameter fibers is less, but more uniform in all directions around the fiber axis. In addition, the scattered light tends to be polarized in the direction parallel to the fiber axis.

The unique, planar scattering pattern for right-angle illumination has been the basis of several useful fiber detection techniques. As noted above, the fiber must be held perpendicular to the light-beam axis to obtain the characteristic fiber pattern. A fiber collected on a surface such as a glass slide will generally be parallel to the slide surface, thus allowing a beam perpendicular to the slide surface to produce the narrow, planar scattering pattern from a single fiber. To obtain a characteristic scattering pattern from a group of fibers, they must be aligned using some force, such as magnetic, electric, or shear flow.

Figures 25-3–25-5 show the scattering patterns for several types of fibers. Note the well-defined scattering pattern for monodisperse carbon fibers (Fig. 25-3) while the broad distribution of diameters for the other fiber types produces a more diffuse pattern.

LABORATORY FIBER GENERATION

Fibers are more difficult to generate than compact particles because of their tendency to intertwine when in contact with each other. This tendency is the basis for some commercial properties of fibers, e.g., the formation of rope and felt. Various types of fibers can be generated in various concentration ranges for instrument calibration, analytical method validation, and quality assurance, and toxicology studies. Various generation mechanisms have been used to produce well-dispersed fibers.

Nebulization of liquid suspensions has been used for generating relatively short fibers at low concentrations. Fibers larger than the nebulized droplet diameter may not be generated efficiently and, if the concentration is too high, more than one fiber may be present in a droplet, resulting in fiber agglomerates.

Several researchers have used high speed chopping of a packed fiber plug. Timbrell, Hyett, and Skidmore (1968) developed a version of this approach using a household coffee grinder. A specially ground reference material, Union Internationale Contre le Cancer (UICC) asbestos (Timbrell, Gilson, and Webster 1968), was packed uniformly in a syringe body and pushed slowly into the rotating blades of the grinder. The consumption rate of asbestos was 0.6–1.0 g/h, though not all of this was generated as an aerosol. The dispersion appeared largely to be single fibers, with relatively few clumps or flocculates, though no size distribution was reported. A generator with similar operating principle and dimensions was constructed using more durable materials, including tungsten carbide blades and a stainless steel body (Fairchild et al. 1976). This device was used for both chrysotile and fibrous glass. Although there were relatively few clumps of chrysotile by number, the mass in these clumps accounted for the largest fraction of the mass

distribution. Airborne concentrations of 6–8 mg/m^3 were achieved, though the feed rate could be lowered to one-fourth or increased to ten times the value used for this measurement. Fibrous glass aerosols were more successfully generated with this device (Fairchild et al. 1978).

Fluidized-bed generators have also been used for creating fibrous aerosols. Fluidized beds consist of two phases: a powder phase, containing one or more components, and an air phase passing through the powder. The powder is made to act like a boiling fluid by passing air through it or by applying vibrational or acoustic energy. The powder may consist solely of the dust to be generated or it may consist of the dust plus particles that are too large to be carried away by the airflow. These powder particles separate the dust particles from each other and tend to break apart agglomerates.

A two-component fluidized bed was used for inhalation exposure experiments with fibrous glass (Carpenter et al. 1981) and crocidolite (Griffis et al. 1983). The bed consisted of a stainless steel powder mixed with the fibers as a slurry and then dried. Air passing through the bed fluidized the bed and released the fibers, initially at a high rate, then decreasing exponentially. A similar air-fluidized bed with bronze powder as the fluidizing powder was used for generating multiple filter samples of chrysotile for a quality assurance program (Baron and Deye 1987).

Charge must be reduced on generated aerosols to produce uniform air concentrations and consistent measurements. This can be accomplished by charge neutralizers (see Chapter 19) or by modifying the generation conditions. It was found that fluidized beds produced highly charged fibers when operated with dry air; the charge level dropped about tenfold when the relative humidity of the air was increased to about 15% (Baron and Deye 1989*a*).

A two-component fluidized-bed generator with a screw feed system to refresh the bed continually with premixed powder was found to produce a constant output concentration (Tanaka and Akiyama 1984). This generator, using glass beads as the large-particle fluidizing component, was found to produce a constant output concentration 6 mg/m^3 fibrous glass (from a glass fiber filter) for one week (Tanaka and Akiyama 1987). A similar system using stainless steel beads and asbestos fibers was developed by Sussman, Gearhart, and Lippmann (1985).

A one-component fluidized bed developed by Spurny (Spurny, Boose, and Hochrainer 1975; Spurny 1980) used vibrational energy to assist bed fluidization. The output of the bed for several types of asbestos was constant with time and the fiber size was controlled by the vibration frequency and amplitude. A fluidized-bed generator of this type, using mechanical vibration, is commercially available (PALAS Co., Karlsruhe).

Chains of iron oxide particles have been generated using a laminar carbon monoxide flame (Kasper, Shon, and Shaw 1980). This generation system created a high concentration of relatively monodisperse ($\sigma_g \approx 1.4$), nearly spherical particles that, under appropriate conditions, agglomerated into chains with little branching. Similar chains have been observed in other flame systems. An example of particle chain size distribution is noted in Table 25-1. These chains can be used for investigating fiber diffusion, alignment, and aerodynamic size, especially in the transition regime (Kasper and Shaw 1983).

FIBER HEALTH EFFECTS

While asbestos fibers have many useful commercial properties, there has been much concern regarding their ability to cause disease. There are three primary diseases that have been attributed to asbestos fiber exposure: asbestosis (a fibrosis or scarring of the lung tissue), mesothelioma (a cancer of the pleura or peritoneum), and lung cancer. Other cancers, e.g., of the gastrointestinal tract, also have been attributed to asbestos exposure. In spite of an abundance of research into disease mechanisms of asbestos fibers, the etiologies of these diseases are still not well understood. Fiber shape appears to play a major role although other properties, such as fiber chem-

and solubility in body fluids, clearly, are important.

iber diameter must play a role in disease, the aerodynamic properties resulting in ratory system deposition are strongly ndent on diameter (Timbrell 1982). In eral, fibers must be smaller than about μm diameter to reach the thoracic region thinner still to reach the air exchange ons of the respiratory system (Stöber, sbart, and Hochrainer 1970). It has ypothesized that short fibers have ss disease potential because macro- ges in the lung can engulf these particles move them from the lung with relative Longer fibers cannot be completely en- by these cells and, therefore, tend to ain in the lung much longer (Holt 1987). rell (1982) found that clearance occurred ers up to 17 μm long. Besides shape, the ical properties of fibers also appear to role. Some fibers, especially glass, have found to dissolve in lung tissue over an ded period, reducing their potential for e (Johnson, Griffiths, and Hill 1984; unn, and Hesterberg 1990). Chrysotile longer than 5 μm were found to in- in number in lung tissue, apparently a longitudinal splitting of the fibers lmann et al. 1986). *In vitro* studies have gested that also the surface properties of ers can affect cell toxicity (Light and Wei 977).

An extrapolation of these properties indi- that long (especially those > 5 μm thin (< 3 μm diameter) insoluble fibers e significant disease potential. Sev- archers have postulated more specific nges as causing the various diseases 978; Lippmann 1988; Timbrell 1990).

REGULATIONS

otentially severe health effects of asbes- exposure have prompted several re- and health research organizations to sh regulations and guidelines for lling the airborne concentrations of asbestos fibers. Since the health-based data indicate that disease at current exposure concentrations is primarily related to fiber number, most regulatory air concentration measurements are based on asbestos fiber number concentration rather than on mass concentration. In the United States, for example, the Occupational Safety and Health Administration (OSHA) provides regulations for exposure to hazardous agents in industrial and other workplace settings. The OSHA regulations require that workers are not to be exposed to more than 0.2 asbestos fibers/cm^3 averaged over an 8 h period or more than 1.0 fiber/cm^3 over 30 min as measured using the filter collection/phase contrast microscope (PCM) method (Occupational Safety and Health Administration 1986). The Mine Safety and Health Administration (MSHA) regulates exposures in mines and mills, and limits miner exposure to 2.0 fibers/cm^3 for an 8 h average and 10 fibers/cm^3 for a 15 min average (Mine Safety and Health Administration 1988).

The Environmental Protection Agency (EPA) regulates the environmental levels of pollutants. Apart from prohibiting visible emissions, EPA has not implemented limits for environmental concentrations. However, to protect children from being exposed to asbestos in schools, the EPA has mandated procedures for removing and measuring asbestos in schools (Environmental Protection Agency 1987). The EPA has defined asbestos-containing material (ACM) as material containing more than 1% asbestos, to be measured using polarized light microscopy. After the removal of ACM in schools, the EPA requires that the airborne asbestos concentration in the cleaned area be no greater than that outside the area. The measurement is conducted using five air samples inside and five outside for the comparison. An analysis by transmission electron microscope (TEM) is required for monitoring the completion of all asbestos removal operations, except that the PCM can be used when removing small amounts of asbestos. Guidance documents describing the methods for controlling asbestos in buildings (Environmental Protection

Agency 1985*a*, 1990*a*) and for the measurement of asbestos after removal (Environmental Protection Agency 1985*b*) have been provided.

The Consumer Product Safety Commission (CPSC) provides guidance to manufacturers regarding the material content and potential hazards of commercial products. One such product to be targeted was hairdryers, prompting measurements of their emissions (Geraci et al. 1979). Individual state agencies set regulations that are often more stringent than those of the national agencies (Abbot 1990).

The National Institute for Occupational Safety and Health (NIOSH) recommends health standards to OSHA and MSHA. NIOSH (1990*a*) has indicated that there is no safe airborne concentration of any asbestos fibers. Based on this, NIOSH urged that the goal be to eliminate, or reduce to the lowest possible levels, exposures to asbestos fibers and recommended an exposure guideline of 0.1 fibers/cm^3, based on practical limitations of PCM measurements.

Regulation of other fibers, e.g., fibrous glass and mineral wool, has generally dealt with these materials as nuisance dust. However, this may change since fibers other than asbestos have been demonstrated to have disease potential in humans and animals. For instance, erionite (a fibrous zeolite) has been associated with human mesotheliomas (Baris 1980) and several man-made fibers have produced disease in animal exposure studies (Pott et al. 1987; Smith et al. 1987).

ASBESTOS TERMINOLOGY

Asbestos is a term applied to several commercial minerals exploited for their useful properties, largely due to their tendency to produce long, thin fibers. The development of asbestos crystal structure occurs primarily along one crystal axis. This results in a structure consisting of fibrils (the smallest-diameter fibers, 0.025–0.05 μm diameter) bundled together. When subjected to comminution, asbestos normally breaks down into particles (fibers) that have high aspect ratios both on macroscopic and microscopic scales; the mineralogical term for this condition is asbestiform (American Society for Testing Materials 1982). Other minerals with the same chemistry and crystal structure but without the unequal crystal development tend to produce more regular particles (termed cleavage fragments), though some of these particles may also be elongated. Although these cleavage fragments have a length/width distribution different from the asbestiform fibers (Virta et al. 1983), individual elongated cleavage fragments are often indistinguishable from asbestiform fibers when measured with commonly available techniques.

The asbestiform minerals that have been regulated include the following six: chrysotile (serpentine), amosite (cummingtonite–grunerite), crocidolite (glaucophane–riebeckite), tremolite asbestos, actinolite asbestos, and anthophyllite asbestos. The nonasbestiform mineral names for the first three materials are provided in parentheses. The latter three have the term asbestos attached because nonasbestiform varieties of these minerals have the same name. The latter five types of asbestos are classified as amphiboles. There are other asbestiform or fibrous minerals, but these are relatively rare and, except for attapulgite and wollastonite, have not been exploited commercially (Zumwalde and Dement 1977).

Health-related regulations, based on available microscope measurement methods, have specified asbestos fibers as particles with the elemental composition (from X-ray analysis) and crystal structure (from electron diffraction) appropriate to asbestos and with a length greater than 5 μm and an aspect ratio greater than 3:1 (Occupational Safety and Health Administration 1986; NIOSH 1990*a*). Thus, the health-related regulatory definitions of asbestos fibers are based on measurements that may include particles that are cleavage fragments and not necessarily asbestiform. This definition is in contrast to the mineralogical definition relating to the asbestiform crystal structure, which often produces particle distributions with higher mean aspect ratios (Kelse and Thompson 1990; Wylie 1979).

term "asbestos structure" is used for ting asbestos air concentration under sbestos Hazard Emergency Relief Act RA) regulations (Environmental Pro- Agency 1987). An asbestos structure is rticle (fiber, bundle, cluster, or matrix) ting of or containing an observed fiber ment. This fiber segment must be longer 0.5 μm, with an aspect ratio of 5:1 or ater, and be identified as asbestos by ele- analysis and electron diffraction. This on is used to provide a sensitive hod for assessing cleanliness after asbes- oval has been completed.

UREMENT TECHNIQUES

re are two main classes of measurement ques for fibers: microscopic observation idual fibers and light-scattering-based ruments. Other instruments described in book also can detect fiber aerosols, but, hey are not specific for fibers, will not sidered here. Microscopic techniques the collection of samples, most often amples, that are returned to the labor- for preparation and analysis by a roscopist. Four principal types of micro- es are used for fiber detection and ana- the phase contrast light microscope CM), the polarized light microscope LM), the scanning electron microscope EM), and the transmission electron micro- (TEM). A review of microscope tech- ques for workplace and environmental stos measurements is given by Chatfield 986). Descriptions of various light and ctron microscopic techniques as well as tures of many different types of fibers particles are given in the seven-volume *icle Atlas* (McCrone and Delly 1973).

ple analysis can take place at various s of complexity, e.g., counting fibers with- cribed size limits, determining the fiber tribution, and measurement of the ete size distribution, as well as identify- ividual fiber types. The first is usually ed to establish compliance with regula- and may require some qualitative ana- well as a simple counting of fibers. The latter types of analysis are usually reserved for research studies or environmental assessments where the fiber sources are unknown.

Sample preparation is extremely important for microscopic analysis, since the view of the fibers and other particles is largely two-dimensional. Thus, the particles must be uniformly distributed at optimum concentration or loading over the sample surface. If the loading is too high, fibers and particles will overlap and be difficult to analyze; if the loading is too low, it will take too long to find a useful number of fibers (Iles and Johnston 1983; Peck, Serocki, and Dicker 1986).

The Environmental Protection Agency (1989) has evaluated asbestos sample preparation techniques. Most analyses of fibrous aerosol samples are performed on filter samples that are prepared without disturbing the location of the fibers on the filter surface (direct-transfer sample preparation). This approach has been taken because asbestos fibers can break up when suspended in a liquid, increasing the fiber number. When sampling other fiber types that do not break apart, or when sampling problems dictate the dilution of the collected sample, a liquid suspension and redeposition of the fibers may be performed (indirect-transfer sample preparation). Indirect sample preparation has the advantages of allowing the removal of some interfering particles as well as providing a more uniformly deposited and optimally loaded sample.

Bulk sample analysis by polarized light microscopy is often used in conjunction with air sampling to find potential sources of airborne fibers. X-ray diffraction (Abell 1984) and other bulk analysis techniques have also been used for asbestos and other fibers; however, these techniques are not discussed here since they are not specific for fibers and usually are not sufficiently sensitive for aerosol sample analysis.

Phase Contrast Microscope (PCM)

The phase contrast microscope (PCM) technique uses an optical microscope selected for the detection of small-diameter fibers. It is

primarily used to measure fiber number concentrations, but generally cannot be used to distinguish between different types of fibers. PCM-based methods are inexpensive and readily available, lending themselves to measuring asbestos fiber concentrations in places known to contain asbestos fibers. PCM only provides an index of asbestos fiber concentration since it cannot be used to detect those fibers thinner than about 0.25 μm. The exact limit of detection is proportional to the difference in refractive index between the fiber and the surrounding medium (Rooker, Vaughan, and Le Guen 1982). For high refractive index fibers, such as crocidolite, fibers somewhat thinner than 0.25 μm may be detected (Kenny, Rood, and Blight 1987). While phase contrast optics improves the ability to detect thin fibers, it also reduces the resolution to approximately 0.6 μm diameter; i.e., thin fibers are detected but appear wider than they really are. Thus, the PCM is not useful for measuring the diameters of thin fibers.

Several fiber detection methods have been published based on the PCM (Asbestos International Association 1979; Carter, Taylor, and Baron 1989*a*; National Health and Medical Research Council 1976; Leidel et al. 1979; World Health Organization 1981).

Sampling

Current sampling methods for asbestos fibers employ a 25 mm sampling cassette with a 50 mm long cylindrical, conductive cowl attached to the inlet. The cowl is primarily intended to reduce contact contamination by personnel who might be wearing the sampler. The collection medium is a 0.45, 0.8, or 1.2 μm pore size, mixed cellulose ester, membrane filter. The smaller the pore size, the greater the pressure drop across the filter and the greater the fraction of fibers depositing on the top surface of the filter. Thus, 0.45 μm filters are used for high-volume area samples analyzed by TEM, while the others are used for personal samples (taken with a battery-powered pump) analyzed by PCM. Sampling periods are chosen to obtain optimum fiber loading on the filter, usually 100–1300 fiber/mm^2. Fiber loadings above this range result in undercounting, while samples below this range tend to result in overcounting (Cherrie, Jones, and Johnston 1986). Non-fibrous particulate present at high concentrations will obscure the fibers and cause undercounting.

Much of the sampling for asbestos fibers is performed to establish compliance with a regulatory standard or exposure guideline. The number of samples required to show compliance or noncompliance have been outlined and is based on the number of samples as well as on the confidence limits around each analysis result (Leidel, Busch, and Lynch 1977).

As with other types of sampling, unit (or 100%) sampling efficiency is needed to obtain samples that represent the environment. Since the sampler diameter and flow rate are constrained by method specifications and sample size requirements, respectively, sampling is rarely conducted isokinetically. For asbestos fibers, this is generally not a problem since most of the fibers posing a health risk have a small enough Stokes number that sampling efficiency is close to 100%. Sampling in stagnant air and at several air velocities, no differences in fiber concentration were found at flow rates up to 16 l/min (Johnston, Jones, and Vincent 1982). However, for fibers with larger median diameters (e.g., > 3 μm), such as glass, graphite, and cellulose fibers, significant undersampling or oversampling may occur under anisokinetic conditions. Measurements of < 5 μm aerodynamic diameter particles under anisokinetic conditions suggest that the 25 mm cowled sampler is accurate within about $\pm 10\%$ at wind speeds up to 0.2 m/s, while at 0.5 m/s it may undersample or oversample as much as $\pm 25\%$ (Fletcher et al. 1989).

Besides optimum loading, the filter deposit must be uniform, since only a small portion of the filter is observed during analysis. Various sampling forces, e.g., electrostatic, inertial, gravitational, can affect the trajectory of fibers entering the sampler and depositing on the filter. Anisokinetic sampling allows some fibers to impact or settle on the cowl surface and results in noticeable distortion of the filter deposit uniformity. Cornett et al. (1989)

ted that a significant portion of glass is collected on the inner walls of the not on the filter. Breysse et al. (1990) oped a technique for determining the tion of fibers on the cowl on the filter. ther these cowl-deposited fibers repre- sses" and should be included in the lysis has yet to be established.

surements of asbestos fibers produced dustrial settings suggest that these fibers more highly charged than particles with a pact shape and, thus, are more mobile in electric field (Johnston, Vincent, and Jones 5). The sampling cassette may carry a rge, in spite of being conductive, since it trically isolated in many sampling ions. An electric field produced by a ed cassette can produce biased sampling well as a nonuniform deposition of fibers the filter (Baron and Deye 1989*a*, *b*; Vincent, and Jones 1985).

cle Preparation

t sample preparation technique is used M analysis. The filter is made optically and a liquid or resin with a refractive lose to that of the filter is used to fill e between the filter and a glass cover cover slip is required by the design of croscope objective. Several techniques been used for clearing filters and sur- ng the sampled particles in a medium ctive index 1.48. The three most com- ly used techniques for sample prepar- in recent years are the dimethyl e–diethyl oxalate (DMP–DEO) od (Leidel et al. 1979), the acetone– tin method (Carter, Taylor, and Baron) and the dimethyl formamide -Euparal method (Le Guen and in 1981).

ples prepared by the DMP–DEO od are temporary, since the filter breaks the mixture crystallizes after about s. The acetone–triacetin method has ntage of providing samples lasting roximately six months to two years n-Taylor and Ogden 1986), as well as uick and easy to perform (Baron and ord 1986). The relative permanence of the samples makes it easier to perform sample recounts for quality assurance programs. This method also provides slightly better sample clarity than the DMP–DEO method. The DMF–Euparal prepared samples are stable for many years, since Euparal is a resin. Although having a slightly higher refractive index, these preparations also have the best clarity of the three (Le Guen and Galvin 1981). The DMF mixture has a higher toxicity than the other materials and the preparation should be conducted in a well-ventilated area (NIOSH 1990*b*).

Some materials, such as fibrous glass, may have a refractive index too close to that of the prepared filter to allow good contrast. An alternate preparation technique involves collapsing the filter and etching the surface with a low-temperature oxygen-plasma (Rendall and Schoeman 1985). Since the fibers are surrounded by air, resulting in a larger difference in the refractive index, higher and presumably more accurate counts can be obtained.

Fiber Counting Procedures

Typical counting rules are listed in NIOSH Method 7400 (Carter, Taylor, and Baron 1989*b*). Particles longer than 5 μm, with an aspect ratio greater than 3:1 are counted. Other commonly used counting rules differ slightly in that neither fibers with diameters greater than 3 μm nor fibers attached to particles with diameters greater than 3 μm are counted.

The graticule depicted in Fig. 25-6 defines the area observed in the microscope field in which fibers are counted (Walton and Beckett 1977). This graticule is adapted specifically for each microscope so that it will present a 100 μm diameter field to the microscopist. A fiber within the graticule field is counted as one, and a fiber with one end within the field is counted as one-half fiber; all others are not counted (see the examples in the figure). Fields are blindly chosen (to prevent biased field selection) from areas on the filter surface, generally along a radius of the filter. A minimum of 20 fields is evaluated. Measurement continues until 100 fibers have been counted,

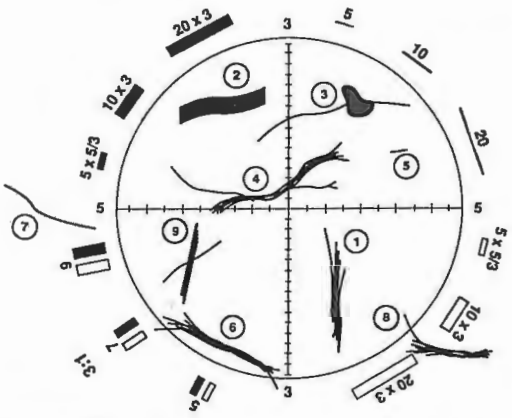

FIGURE 25-6. Walton–Beckett Light Microscope Graticule for Counting Fibers. Particles 1, 2, 3, and 4 are counted as one fiber; 5, 6, and 7 are not counted; 8 is counted as one-half fiber; and 9 is counted as two fibers.

or 100 fields have been evaluated, whichever comes first.

The fiber count observed on each filter sample is corrected for background counts performed on blank filters. The airborne concentration C (fibers/cm^3) is calculated, based on the number of fibers F observed in N microscope fields:

$$C = \frac{(F/N\,A_g)\,A_f}{1000\,V} \qquad (25\text{-}11)$$

where each graticule field A_g has a nominal area of 7.85×10^{-3} mm^2, the sampling filter has a collection area A_f (approximately 385 mm^2 for a 25 mm diameter filter) and the sampled volume V is measured in liters. F is adjusted for blank filter counts.

An alternate approach to counting fibers, especially at low concentrations, is to count voids, that is, the number of fields that contain no fibers (Attfield and Beckett 1983). If the fibers are distributed on the filter according to the Poisson distribution (uniformly random), the number of void fields can be directly related to the fiber concentration per field (c_f) on the filter:

$$c_f = \ln\,(n/E) \qquad (25\text{-}12)$$

where n is the number of fields observed and E is the number of void fields. Sahle and Larsson (1989) found that for lightly loaded samples, this approach is faster than conventional counting by almost a factor of two and also may be more precise.

Measurement Accuracy

The accuracy of various fiber counting techniques is poor when compared to other analytical methods. For instance, most methods in the NIOSH Manual of Analytical Methods state an overall accuracy (combined variability and bias) of better than 25% (NIOSH 1984). This requires that the variability of a method be less than about 12%. Under optimum analysis conditions (uniform sample deposit, no background dust interference, optimum loading), a relative standard deviation of 0.10 (or 10%) is predicted for the fiber counting method. Thus, the accuracy (including bias and variability) can be no better than this level.

In fact, other sources of variability and bias can occur. Some of these are due to the small sample size observed as part of the measurement procedure. For instance, fibers may be nonuniformly distributed in the sample due to inertial, electrostatic, or other sampling influences. Since the analysis assumes a uniform distribution on the filter, taking a small portion for microscopic analysis can, therefore, result in significant variations in the reported concentration. In addition, one microscopist may introduce biases relative to other microscopists due to decisions regarding which particles to count as fibers. When comparisons are made between groups of microscopists, these biases may appear as an increased variability in the overall results.

Comparisons of the PCM with other microscopic techniques have been made (Marconi, Menichini, and Paoletti 1984). Because of differences in sensitivity, resolution and type of illumination, different microscopic techniques will often produce different results. However, if the analysis of fibers by a high-resolution technique, e.g., electron microscopy, is limited to those fibers visible

by PCM, good agreement can be obtained (Marconi, Menichini, and Paoletti 1984; Taylor et al. 1984).

The use of established analytical procedures for fiber count analysis is extremely important. This is the only way that results from one laboratory can be compared reliably with those from other laboratories. Microscopist training, proper equipment, and established quality control procedures are all important components of proper laboratory practice. To ensure a uniformity of application of these analytical procedures, both within-laboratory and interlaboratory sample exchanges are necessary (Abell, Shulman, and Baron 1989; Ogden et al. 1986). The usual technique for determining analytical biases, that is, the comparison with a reference method, does not work because an alternative fiber counting technique that measures the "true" fiber concentration does not exist. Thus, the final test of fiber counting accuracy is the comparison of one's results with those of a group of competent laboratories.

Several formal programs of sample exchange have been established for PCM. These include the American Industrial Hygiene Association's (AIHA) Proficiency Analytical Testing (PAT) program (Groff, Schlecht, and Shulman 1991), U.K.'s Regular Inter-Laboratory Counting Exchange (RICE) program (Crawford and Cowie 1984) and the International Asbestos Fibre Regular Interchange Counting Arrangement (AFRICA) program (Institute of Occupational Medicine, Edinburgh). For a laboratory performing PCM analyses to establish compliance with OSHA asbestos fiber exposure level regulations, regular sample exchanges with other laboratories are required.

The measurement of fiber concentrations for comparative use within a single study may not need all the components of a complete quality assurance scheme. For instance, if a study is intended only to provide relative fiber concentrations to show differences with time or location, interlaboratory exchange of samples may not be necessary. However, the use of published counting and sample preparation procedures, as well as the performance of blind repeat analyses, are important for establishing analytical confidence limits.

Application

The PCM is primarily used for fiber counting to provide an index of asbestos fiber exposure in workplaces in which asbestos is known to be present. The current U.S. regulations for determining airborne asbestos fiber exposure in industrial workplaces (Occupational Safety and Health Administration 1986), mines and mills (Mine Safety and Health Administration 1988), and, sometimes, for determining acceptable levels after asbestos abatement in schools (Environmental Protection Agency 1987) are based on PCM analysis.

Although the morphology observed under the PCM allows some discrimination between fiber types, it is not specific enough to allow positive identification of asbestos or other fibers. The PCM is used also for the measurement of man-made mineral fibers (MMMF), e.g., mineral wool and fibrous glass (NIOSH 1977). It can be used with other analytical techniques, such as PLM, SEM, or TEM, for specific fiber types. Such an approach allows compliance with regulations and guidelines as well as comparison with the epidemiological and toxicological data that have been obtained for various types of fiber exposure.

For instance, a TEM method, NIOSH Method 7402 (Carter, Taylor, and Baron 1989*b*), provides qualitative analysis for asbestos fibers. In this method, all fibers thicker than 0.25 μm (presumed optically visible) are analyzed by electron diffraction and energy dispersive X-ray analysis (EDXA). Rather than using the TEM count directly, the asbestos fiber fraction (relative to the total number of fibers) is applied to the PCM count on the same sample. This approach is taken because Taylor et al. (1984) found that the TEM count alone had higher variability than the combined TEM/PCM result.

Polarized Light Microscope (PLM)

PLM is often used to determine the percentage of asbestos or other fibres in bulk

materials that can potentially release an aerosol. The EPA (1987) has defined asbestos-containing material (ACM) as material containing more than 1% asbestos. PLM permits the identification of fiber type by an observation of the fiber morphology, light diffraction, and optical rotation properties relative to the surrounding medium. Under carefully controlled conditions, an estimate of percentage of asbestos may be obtained.

Several PLM techniques are used for identifying fiber type as well as quantifying the percentage of fibrous material (usually asbestos) in a sample (Klinger et al. 1989; Middleton 1979; McCrone, McCrone, and Delly 1978). These techniques depend on the refractive index and other optical properties of individual particles. Many of these PLM techniques require a visual observation of color in the fiber and become less reliable for fibers thinner than about 1 μm (Vaughan, Rooker, and Le Guen 1981).

Sampling

Several procedures have been suggested for obtaining representative bulk samples of ACM in a fashion that prevents unnecessary exposure to asbestos aerosol (Jankovic 1985). The material should be wetted or sealed during sample removal. A small coring device, such as a cork borer, can be used to obtain a sample from the full depth of the material. At least three samples per 1000 ft^3 of ACM should be taken. The sample should be placed in a well-sealed, rugged container. Finally, the sampled area should be repaired or sealed to minimize further fiber release.

Sample Preparation and Analysis

Sample preparation for a PLM analysis involves grinding the material to the optimum particle size range (1–15 μm diameter) and dispersing the particles in a liquid of known refractive index on a glass slide. Some ACM, such as vinyl asbestos floor tiles, may require dissolution or ashing of the matrix material so that the fibers are separated and are visible in the microscope. Before and after preparation, the sample is observed with a stereomicroscope at 10–100× magnification to evaluate sample uniformity and observe whether fibrous material is present.

Some materials that interfere with accurate fiber identification either by their similarity or by covering up the fibers can be removed by physical treatment of the sample. For instance, organic materials, such as cellulose fibers or diesel soot can be removed by low-temperature oxygen-plasma ashing (Baron and Platek 1990). Leather fibers and chrysotile have a similar appearance and refractive index. The leather can be removed by ashing at 400°C (Churchyard and Copeland 1988).

Morphology of fibers can give some indication of fiber type. For instance, it assists in differentiation of chrysotile (curly fibers) from the amphibole asbestos (straight fibers, especially when shorter than 50 μm). Asbestos fibers often have frayed or split ends. Glass or mineral wool fibers are typically straight or slightly curved with fractured or sometimes bulbous ends; diameters are usually in the 5–15 μm range. Many plant fibers are flattened and twisted, with diameters 5–20 μm. Note that it is not recommended to base identification solely on morphology.

Crossed polarizing filters in the microscope can be used to indicate whether a fiber is isotropic (isometric or amorphous) or anisotropic (uniaxial or biaxial crystal structure). A measurement of the angle at which these fibers disappear (the extinction angles, a function of the crystal structure) narrows the possible identity of the fibers.

When a transparent fiber has a larger refractive index than its surrounding medium, the bright halo (Becke line) around that fiber appears to move into it as the microscope focus is raised; when the fiber has a smaller refractive index, the Becke line moves out of it. This technique can be used to bracket the fiber refractive index.

Dispersion, or refractive index change with wavelength, of a fiber can be used for identification. When particles are placed in a liquid whose dispersion is different from that of the particle, the particle may exhibit a color caused by the refraction of light. This technique requires the use of special "dispersion

staining" optics. By using several refractive index liquids in series, the refractive index and the dispersion of the fiber can be established and compared with those of standard materials or published data (McCrone 1980).

Once the sample has been prepared in the appropriate refractive index liquid, specific fiber types, e.g., asbestos, can be identified and the percentage of fibers estimated. Two approaches are typically used: visual comparison with prepared reference slides or pictures and point counting. When attempting to estimate whether a material is ACM (i.e., $> 1\%$ asbestos), the visual comparison technique is adequate when more than about 10% of the particles observed are asbestos. Point counting is used for these lower concentration samples to provide higher accuracy (Environmental Protection Agency 1990*b*). It involves observing 400 randomly selected "points" (identified with a reticle crosshair) in the sample. The number of points containing asbestos is divided by the total number of points observed to give the percentage of asbestos. A combination of these approaches balances the analysis time and accuracy of the results (Webber et al. 1990).

PLM also can be used for qualitative analysis of air sample filters by collapsing the filter and using low-temperature plasma etching of the surface to expose the fibers. Various refractive index liquids can then be placed on the etched surface to surround the fibers, allowing techniques noted above to be used (Vaughan, Rooker, and Le Guen 1981). The smallest fibers that can be identified by this method are about 1 μm in diameter.

Accuracy

PLM analysis is primarily used for qualitative identification of fiber type. Accurate identification of asbestos and other fibers requires a proper training in the crystallographic properties of particles as well as training and familiarization with the PLM. As with fiber counting, a laboratory quality assurance program is necessary to ensure consistently accurate results. The National Voluntary Laboratory Accreditation Program (NVLAP) operated by the National Institute for Standards and Technology (NIST) inspects laboratories for proper practice as well as provides unknown samples four times a year to check their performance in fiber identification. Under a predecessor to this program, approximately 350 laboratories correctly classified 98.5% of the samples as asbestos and correctly identified the specific asbestos types in approximately 97% of the samples. A blind test of 51 laboratories resulted in 97.5% correct classifications and 79.1% correct identifications (Parris and Starner 1986).

PLM has been cast in a quantitative measurement role by the EPA requirement of determining whether a school building material is ACM. Many variables, including particle size, density, and shape, are not adequately controlled or measured in the analysis and contribute to errors in the percentage mass estimate. Thus, PLM analysis is at best a semi-quantitative technique. Performance evaluation of laboratories' ability to estimate the percentage of asbestos is also planned under the NVLAP program.

Scanning Electron Microscope (SEM)

The SEM is manufactured in a variety of models, ranging from inexpensive devices that have relatively low resolution and can only display particle images, to more sophisticated devices that rival the TEM in resolution and can also provide elemental analysis of particles. Thus, the SEM provides analyses intermediate between those of the light microscope and the TEM, giving elemental analysis and higher-resolution images than the light microscope. Under routine fiber counting conditions, fibers thinner than about 0.1 μm are not generally visible with the SEM, nor can the internal structure of fibers be seen. Additionally, a definitive determination of fiber type by electron diffraction is not available with the SEM (Middleton 1982; Steen et al. 1983). However, in the "photographic mode", a high-quality SEM can be used to detect small fibers quite effectively. The photographs can be used to measure fiber size distributions (Platek et al. 1985).

Sample Collection and Preparation

A sample that is to be used directly in the SEM must have the fibers exposed on the sampling substrate. Samples are typically either collected on the smooth-surfaced polycarbonate capillary pore filter or deposited on a smooth substrate by impaction or electrostatic precipitation. Samples that are taken with a membrane filter can also be used if the filter is collapsed and the filter material is etched away by low-temperature plasma ashing. The sample must generally be coated with a conductive layer to reduce charge accumulation from the electron beam in the SEM. Gold and carbon are common coating materials. Gold allows a thinner layer to be effective, resulting in somewhat higher resolution, but interferes with EDXA of the fibers. Once a sample is coated, it can be placed directly in the SEM for analysis. Relatively large samples, each up to several cm in diameter, can be analyzed.

Application

For the most part, the SEM has been used for fiber detection and analysis as an intermediate between TEM and PCM analysis. Generally, the SEM has not been recommended for measuring asbestos, especially in environmental situations, because of the inability to positively identify fibers by electron diffraction as well as the lack of instrument standardization. For occupational exposures, where the fibers present are generally of a known type, PCM has been preferred as the less expensive and more available method. A SEM method was developed by the Asbestos International Association (1984). A SEM method was recommended by the World Health Organization for sizing man-made mineral fibers (World Health Organization 1985).

However, the SEM does permit the microscopist (1) to obtain higher resolution and sensitivity than the light microscope, (2) to obtain qualitative information about fiber type from elemental analysis, and (3) to reduce sample preparation and analysis costs vs. the TEM. In research studies of known fiber types when analysis time is not important, photographic images from the SEM can be analyzed to obtain size distributions including all fiber sizes (Platek et al. 1985). The SEM field of view is larger than that for the TEM and, thus, long fibers may be sized more accurately.

Transmission Electron Microscopy (TEM)

TEM allows the most definitive analysis of individual fibers: particle shape can be observed and measured, elemental composition can be determined by EDXA, and crystal structure can be deduced from electron diffraction patterns (Langer, Mackler, and Pooley 1974). TEM has sufficiently high resolution and sensitivity to allow observation of the smallest fiber sizes. However, TEM analysis is the most expensive one described in this chapter because of instrument cost and complexity, high operator expertise, and complex sample preparation. Furthermore, quantitative fiber concentration measurements have relatively poor reproducibility (Environmental Protection Agency 1985*b*).

Sampling

Currently, post-asbestos abatement clearance monitoring is perhaps the most common use of TEM analysis. The measurement is intended to indicate that a location is acceptably clean so that it can be reoccupied. The EPA (1987) AHERA regulations require the use of 25 mm conductively cowled cassettes with 0.45 μm pore size, mixed cellulose ester filters. Polycarbonate capillary pore filters also have been used in the past; however, they have fallen into disfavor because of erratic contamination of the filter medium with asbestos fibers (Powers 1986).

Sampling requirements for TEM analysis are similar to those of other microscopic analyses, although the high magnification afforded by TEM allows a somewhat higher filter loading than that for PCM. For direct-transfer sample analysis, the sample must be optimally loaded, the sampling efficiency should be as close to 100% as possible, and the filter deposit uniform. For indirect-transfer sample analysis, only the sampling effici-

ency is important. Mixed cellulose ester filters are commonly used, but may not be optimal because short, thin fibers can deposit deep within the filter and not be completely transferred to the TEM sample grid. Other filter materials have also been used, e.g., vinyl copolymer. Note that vinyl and polycarbonate filters tend to retain more nonuniform surface charges than cellulosic filters because of their lower conductivity. These charges may cause nonuniform particle deposition.

The AHERA regulations also require the use of "aggressive sampling". This is a procedure in which high-velocity air blowers (e.g., leaf blowers) are used to release dust particles from the surfaces in a room so that they can be sampled. At the same time, fans keep the dust suspended and mixed with the air. An ac line operated pump is used to sample at flow rates up to 10 l/min. The sampling cassette is placed at a height of 1.5–2 m from the floor in a downward-facing position. Sampling times are typically 2–10 h.

Sample Preparation

Sample preparation, always an important part of any microscopy, is especially important for the TEM. The potentially high levels of magnification dictate that the sample be relatively small (the grid holding the sample is 3 mm diameter), thin (the electron beam must be able to penetrate the particles to perform electron diffraction), and evenly dispersed (the grid should accurately represent the entire sample). Two general approaches have been taken in preparing samples: direct transfer (the carbon film contains the particles in the same location and orientation as when they were originally deposited on the filter or substrate) and indirect transfer (collected particles are dispersed as a liquid suspension and redeposited on a filter for grid preparation) (Environmental Protection Agency 1989). Many of the following procedures are described in the EPA (1987) AHERA method.

As with other measurement techniques, the purpose of the analysis is important in determining the approach to take. Sometimes, the fibers may be fragile (agglomerate chains, asbestos fibers) and minimal manipulation of the sample is necessary to prevent breakup. In such cases, direct transfer of the particulate to the electron microscope grid is preferred. The direct-transfer technique may have difficulty including all the particles collected on a filter (especially membrane filters) in the carbon membrane/grid sample due to varying particle thickness and collection below the filter surface. This technique is the one most widely adopted for environmental asbestos measurements.

For other analyses where number concentration is not important or the fibers are more sturdy, an indirect-transfer technique may have advantages. The collected sample is resuspended in a liquid, mixed (usually by ultrasonication) and an appropriate-sized aliquot is redeposited on a capillary pore filter for preparation of the carbon film. The indirect-transfer method homogenizes the sample and ensures that the final preparation has the appropriate concentration as well as good uniformity. This approach may cause an apparent increase in particle concentration because resuspension tends to separate particles, especially fibers, that were attached in the aerosol phase. One study found 15.5 times as many fibers with the indirect approach as with the direct approach (Huang and Wang 1983). However, it has been suggested that this increase is primarily due to shorter (< 1 μm) fibers being separated from larger particles (Chatfield 1983) and that the number of long (> 5 μm) fibers is not changed significantly as long as the ultrasonication is gentle (Chatfield 1986).

Complete separation of fibers into fibrils by extensive ultrasonication was used as a technique for asbestos fiber mass determination since it eliminated the variability of fiber diameter, gave a more uniform dispersion of fibers in the sample, and increased the number of fibers available for counting (Selikoff, Nicholson, and Langer 1972).

Figure 25-7 provides an indication of the major steps involved in the direct-transfer preparation of the TEM sample. The membrane filter is collapsed using vapors of a solvent such as acetone. It has been suggested that acetone vapor collapse of the filter causes

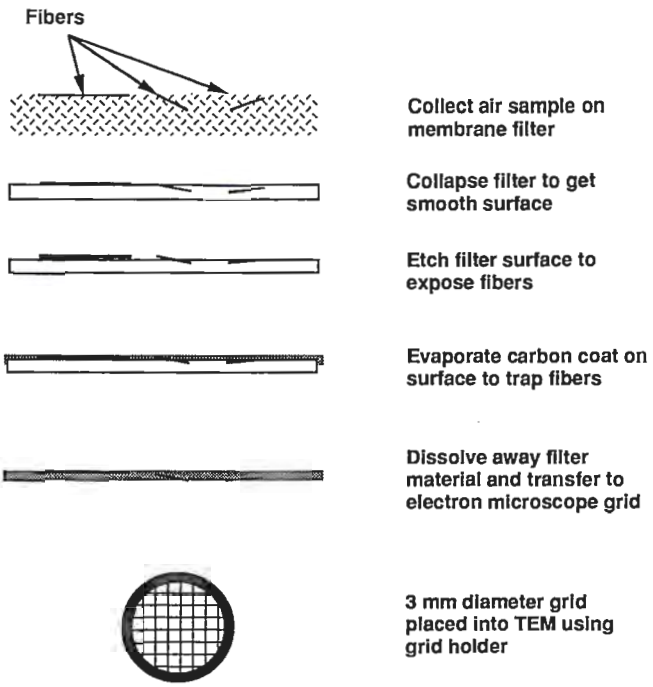

FIGURE 25-7. Principal Steps Used in Transmission Electron Microscope Sample Preparation by the Direct Transfer Method.

fibers to be washed into the body of the filter. Thus, the carbon film does not capture all these fibers for transfer to the electron microscope grid (Chatfield 1986). For this reason, the DMF technique described above for PCM samples, combined with the use of a 0.1 μm pore size filter, may provide an improved TEM sample (Burdett and Rood 1983; Chatfield 1986). To expose more of the fibers, especially those that may lie below the top surface of the collapsed filter, a low-temperature ashing step is often performed. Carbon is then vaporized onto the surface, generally from a range of angles to produce a uniform coating. The carbon film must be of the proper thickness to provide a stable film on the grid; thinner films will crack and break apart while thicker films prevent the electron beam from providing good contrast images of the fibers. Thicker films may also obscure the electron diffraction patterns of very small fibers.

A copper grid is placed on the carbon-coated surface and the filter is dissolved away using an appropriate solvent. This step is important to complete because incomplete removal of the filter degrades imaging by the TEM severely. The removal must also be gentle to prevent a breakup of the carbon film. Complete filter removal may take 4–24 h.

Several grid preparations are generally made to ensure that enough grid openings have the carbon film intact to provide an adequate area for analysis. Observing more than one grid also reduces the probability of biased results due to local variations of fiber deposition on the filter.

Analysis

The TEM operates by focusing a high-energy (10–1000 keV) electron beam on an area of the sample. The beam passes through the sample, which is usually supported on a grid by a thin, nearly transparent, carbon film. By controlling the magnetic focusing optics, the image of the sample can be observed on a phosphorescent viewing screen at various magnifications ranging from about 200× to 1,000,000×. For asbestos fibril detection,

10,000–20,000 × magnification is used. Particles absorb more of the electron beam energy than the supporting carbon film and appear as dark objects on a light background. Particle shape is often an important indicator of the type or origin of fiber. Larger asbestiform fibers can usually be distinguished by their fibrillar structure, especially at the ends where the fibrils tend to splay apart. Chrysotile fibrils, when observed at high magnification, usually display a tubular structure. These various shape characteristics are often sufficient when the presence of certain fiber types has been established. However, in samples of unknown source or composition, further qualitative analysis of each fiber is usually necessary.

Besides observing particle shape, the TEM allows the use of electron diffraction, which indicates crystal structure, and EDXA, which indicates the elemental composition of particles. Diffraction and elemental composition patterns from reference materials are used to confirm the presence of specific types of fibers. It often happens that for a given fiber, the diffraction pattern and/or the elemental composition pattern may not be completely detected or may change over the length of the fiber. This may be due to fiber thickness, interfering materials, variations in fiber composition or twisting of the crystallographic axis along the fiber length. Thus, a full confirmation of the identities of all fibers is often not possible.

Applications

The TEM is a microchemical instrument that can be used for analyzing collected samples of aerosols as well as bulk samples. It can provide the most definitive qualitative information regarding fiber type as well as quantitative information on fiber number. In addition, the fiber dimensions, composition, and number concentration information can be used to estimate the mass concentration.

Accuracy

A detailed study by Steel and Small (1985) of parameters affecting the TEM analysis accuracy showed that instrument capabilities, including mechanical stage, imaging and contrast, and diffraction, can produce order-of-magnitude errors if they are not up to the demands of asbestos analysis. By a careful comparison between several counters and microscopes using well-characterized chrysotile samples, it was also found that a careful and experienced analyst could attain accuracies of 90% in finding all asbestos fibrils in a sample. The accuracy degraded significantly for fibrils shorter than 1 μm and analysts had a 50% or less chance of finding those shorter than 0.5 μm. In an interlaboratory comparison on prepared grid samples, Turner and Steel (1991) found that the average result reported by the laboratories was 0.67 of the NIST-verified count. Forty percent of the fibers shorter than 1 μm were missed; half as many longer than 1 μm were missed.

Repeat counts by a single analyst of laboratory-generated amosite fiber samples gave a relative standard deviation of 0.27 (Taylor et al. 1984). The interlaboratory relative standard deviation for repeat counts on the same sample has been estimated to be approximately 1.0 (Environmental Protection Agency 1985*b*).

Recently, a laboratory accreditation program has been established by the National Institute for Standards and Technology under the National Voluntary Laboratory Accreditation Program (NVLAP). Data regarding laboratory performance in this program have not yet been published. Quality assurance guidelines for laboratories participating in this program are available (Berner et al. 1990).

AUTOMATED FIBER ANALYSIS TECHNIQUES

IMAGE ANALYSIS

There have been several attempts to improve the accuracy and speed and to reduce the subjectivity of microscope analysis by using automated image analysis of asbestos fiber samples (Whisnant 1975). Automated image analysis involves taking the image from a microscope, digitizing the image, and using a

computer to evaluate the number and size of objects present in the image. For fiber analysis, this may involve counting the number of fibers present or obtaining a size distribution of the fibers.

Fiber counting has been achieved with reasonable success for PCM analysis of asbestos fibers. The Manchester Asbestos Program (MAP) was developed at the Manchester University (U.K.) with support from the Health and Safety Executive (Kenny 1984). The MAP (Applied Imaging, Tyne & Wear, U.K.) operates in a semiautomated mode, with the analyst selecting fields and focusing the microscope.

Evaluations of this program indicated that it provided a good correlation with manual counting (by microscopist) in most cases. The precision of the results from MAP was better than that produced by manual counting (Baron and Shulman 1987; Kenny 1988). Some difficulties the program had included breaking fiber images apart and counting them as more than one, not detecting very thin fibers, detecting edges of large particles as fibers, and detecting chains of particles as fibers. A second version of the program was developed to deal with samples in which most of the particles were not asbestos, such as samples from abatement sites (Kenny 1988). The MAP program is used as the reference for a U.K. quality assurance program (Ogden et al. 1986).

Many image analysis systems have available software for counting larger, well-defined fibers. For instance, they work reasonably well for fibrous glass insulation or synthetic organic textile fibers. Such programs will not work as well as the MAP for asbestos fiber images because of the difficulty of detecting fibers of various diameter and curvature, some barely visible, some overlapping, in the presence of a noisy background containing particles of different shapes and sizes. However, with increased computer power and improved image analysis techniques, more accurate automated fiber counting may be possible in the future.

Image analysis systems are integrated with some SEMs, since it is possible to apply direct computer control of the electron beam. Such systems work well for compact particles and are capable of determining size distribution and elemental analysis of about 200 particles per hour (Stettler, Platek, and Groth 1983). A system based on principles similar to those of the MAP for fiber detection was developed for an SEM (Stott and Meranger 1984) but has not been commercialized.

An image analysis system can be attached to a TEM by attaching an imaging camera below the observation screen. The image can then be acquired by the computer system and analyzed in much the same way as an optical image. However, many image processing and analysis functions are integrally tied to the magnification of the image. Therefore, any change in the magnification may require extensive reprogramming of the software.

Direct-Reading Fiber Measurement

An optical particle counter has been used for the detection of chrysotile fibers in a textile plant where asbestos was the primary aerosol contaminant (Rickards 1978). Other direct-reading monitors also may be used in situations where the fibers are the major constituent in the aerosol, as in laboratory studies.

A fibrous aerosol monitor was developed for a more specific measurement of asbestos fibers in the presence of compact particles (Lilienfeld, Elterman, and Baron 1979). A commercial version of the Fibrous Aerosol Monitor (FAM-1, MIE Inc., Bedford, MA) was produced and has undergone continuous improvement, primarily in ruggedness for field use, over the past ten years. A new version with computer control has recently been produced (FM-7400, MIE Inc., Bedford, MA). This instrument detects fibers via a combined electrostatic alignment/optical scattering technique. Fibers are aligned in a plane perpendicular to a laser light beam and then oscillated back and forth within this plane. These fibers will scatter the light primarily in a direction perpendicular to the fiber's principal axis. Thus, as the fibers oscillate, pulses of light are observed at a photomulti-

plier detector, which views a direction at right angles both to the laser beam and to the fiber axis. These pulses can be related to fiber size and shape. See Chapter 17 for a further discussion of the FAM-1 operation principles.

The FAM-1 depends on light scattering patterns for the detection of fibers and is, therefore, not specific for fiber type, e.g., asbestos. The instrument is calibrated with asbestos fibers and compared with filter samples counted using NIOSH Method 7400. Several evaluations show that data from the FAM-1 correlate reasonably well with PCM-based measurements (Lilienfeld 1986), though one study found that the FAM-1 was not much better for monitoring fibers in workplaces than an optical particle counter (Iles and Shenton-Taylor 1986). Some instrumental problems noted in that study may have been due to reliability problems associated with early production instruments. Currently, the FAM-1 is frequently used to monitor control systems that are installed to reduce asbestos exposures at asbestos abatement sites.

Another direct-reading fiber monitor (Hygenius, Inc. Mississauga, Ont.) has recently been marketed that also uses electrostatic alignment and light scattering detection principles. However, the light scattering patterns are detected with a solid state imaging array rather than a photomultiplier.

OTHER MEASUREMENT TECHNIQUES

An asbestos fiber analysis system, the M-88 Fiber Analyzer (Vickers Instruments, York), was developed based on magnetic alignment/light scattering detection of asbestos fibers on filter samples. Fibers were floated from the filter surface with a solvent, magnetically aligned, and then trapped in an aligned position when the solvent evaporated. The integrated scattering from a large portion of the filter was analyzed and compared to reference standards to give a quantitative indication of fiber count. Evaluations of this instrument found that the calibration varied significantly with fiber source and size distribution (Jones and Gale 1982; Abell, Molina, and Shulman 1984). However, even these observed correlations between instrument response and fiber concentration disappeared at low filter loadings (Verrill 1982). Production of the instrument subsequently ceased.

Asbestos and other fibers have been stained with fluorescent dyes to enhance their visibility in a light microscope (Benarie 1983). While this technique may be used as a rapid screening technique, it is not sufficiently specific for asbestos to provide unequivocal identification of fiber type.

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