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DIRECT-READING MEASUREMENT OF FIBER LENGTH/DIAMETER DISTRIBUTIONS

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ABSTRACT: A new technique for classifying fibers according to length has opened up opportunities for improving fiber measurement technology. The classification principle is dielectrophoresis, which moves conductive fibers at a velocity proportional to the square of their length. Even non-conductive fibers can be made sufficiently conductive by increasing the humidity in the classifier. A device based on this principle has been constructed. It can classify fibers from a broad distribution into length distributions with CVs in the range of 0.2-0.3 and with mean lengths selectable from about 4 µm to >50 µm. To complement this device, diameter classification readily can be achieved by gravitational or inertial techniques in combination with length classification. An inertial classifier was used to produce relatively monodisperse diameter fibers in the aerodynamic diameter range of 3 - 7 µm. This device was used to calibrate the response of the Aerodynamic Particle Sizer (APS) so that real-time diameter distributions were obtained for each length distribution from the dielectrophoretic length classifier. By varying the voltage on the length classifier, a length/diameter distribution can be produced in a matter of minutes

KEYWORDS: fiber, fiber measurement, fiber classification, fiber size distribution, dielectrophoresis

INTRODUCTION

Fiber measurement has traditionally been carried out using sample collection and subsequent analysis by microscopy. This approach has been taken by necessity in the absence of real time measurement techniques. Sample collection is prone to biases due to inertial, gravitational and electrostatic effects. Sample preparation is a source of bias that typically causes a reduction of measured fibers. Light microscopy has limited sensitivity to small diameter fibers; scanning electron microscopy has improved sensitivity and allows elemental analysis of individual fibers, though with increased cost. Transmission electron microscopy allows detection of all fiber sizes, permits measurement of fiber elemental composition and crystal structure, though with a further increase in cost. All forms of microscopy are limited in precision due to small number of fibers analyzed. Under optimum conditions, a sample can be collected, prepared, and analyzed by light microscopy

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in less than an hour, while typical SEM and TEM analysis times are typically longer than a day, especially when a good estimate of the size distribution is desired. These various drawbacks limit the type of measurement and research that can be carried out on fiber behavior and toxicity.

A direct reading instrument, the fibrous aerosol monitor (FAM) was developed in the late 1970s[1] and is still commercially available (Model FM-7400, MIE, Inc. Bedford MA). It aligns individual airborne fibers in an electrostatic field and measures their light scattering patterns at 90°. Theoretical calculations have indicated that this approach can be used to extract size information regarding the fibers.[2] Scanning electron microscope measurements on 0.85 µm diameter glass fibers indicated that the FAM gave comparable length distributions [3]. Further measurements are needed to confirm this capability for a range of fiber diameters and types.

There are clearly gaps in the technology of accurate, real-time measurement of fiber length and diameter. Measurement of compact particles was in a similar state in the early 1970s. Development of the differential mobility analyzer, inertial classifiers, and the aerodynamic particle sizer allowed direct reading measurement of submicrometer and larger particles, respectively [4,5]. These measurement instruments produced an explosion in research and understanding of airborne particles in a range of environments, including atmospheric, workplace and clean room situations. These instruments also allowed development and testing of simpler devices for measurement and classification of particles.

Size classification of fibers by length and diameter is difficult. Early attempts to size segregate fibers from a liquid suspension met with very moderate success. The primary impediment is the ability to separate fibers according to length. Diameter separation appears likely once length separation can be effected, because several studies have shown that gravitational and inertial behavior depends primarily on fiber diameter and only weakly on length. Recently, a technique was developed using fiber charging to separate monodisperse diameter fibers by length [6]. This appeared to be successful, but no follow-up work has been performed. We have taken the suggestion of Lipowicz and Yeh [7] that fibers can be classified by length using dielectrophoresis and developed a differential fiber length classifier. The principles, implementation and application of this technique are described below.

MEASUREMENT PRINCIPLES

Conductive fibers placed in an electric field can be readily polarized and aligned parallel to the field. The degree of conductivity required for this polarization is relatively small, since it only requires the motion of a few charges over the length of the fiber. This polarization was observed in the fibrous aerosol monitor for asbestos fibers, even though asbestos is normally considered a good insulator. Glass fibers on the other hand did not always polarize so easily. Relative humidity levels on the order of 50% were required to align fibers in early versions of the FAM. Later versions used an increased voltage to align glass fibers at lower humidity levels. The higher humidity caused water condensation on the surface of the fibers that allowed sufficient current to flow, resulting in fiber alignment.

Conductive fibers placed in a gradient electric field will first align parallel to the field, as indicated above, and then move toward the region of higher field intensity. This technique was described by Lipowicz and Yeh and shown to work for aluminum fibers in liquid suspension [7]. The velocity of fibers in a gradient electric field was described by the equation

$$\mathrm{v} = \frac{\mathrm{K_m}\,\varepsilon_0}{36\eta}\mathrm{DL}\left\{\mathrm{g}(\beta)\left(\frac{\alpha}{\alpha-1}-\mathrm{f}(\beta)\right)\right\}^{-1}\nabla\left(\mathrm{E}^2\right) \tag{1}$$

where D and L are the fiber length and diameter, β is the aspect ratio, f and g are complex functions of β, E is the electric field, and the rest of the parameters are physical constants. The net result of this equation for conductive fibers in a specific classifier is that the velocity is proportional to fiber length squared and to voltage squared.

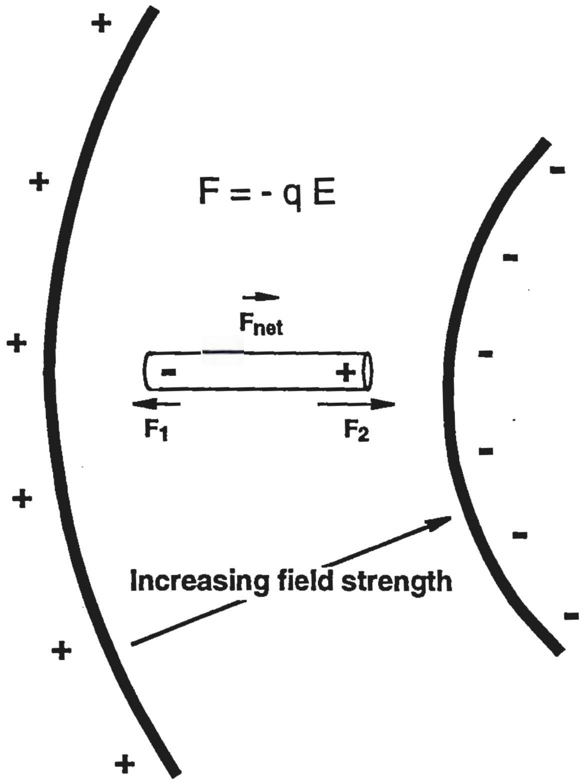

FIG. 1--*An overview of the fiber length classifier configuration with a fiber placed in the annular space between two concentric cylinders*

The dielectrophoresis technique was applied to airborne chrysotile fibers and shown to work for fibers in the length range of about 5 to 50 µm [8]. The classifier section consisted of two concentric tubes with a space of about 3 mm through which the fibers passed (Figure 1). Longer fibers deposited at the upper end of the inner tube, while shorter fibers deposited at progressively longer distances down the tube. Fibers deposited at several distances on the inner tube were removed and analyzed by TEM.

The voltage applied between the tubes was originally a sine wave, but was then changed to a square wave (50 - 100 Hz), with the peak voltages ranging from 0 to about ±7000 V. The square wave produced twice the rms electric field as the sine wave with the same peak voltage. The peak voltage was limited by corona or discharge breakdown in the classifier. The alternating voltage was used to reduce the likelihood that charged particle motion (electrophoresis) would dominate the fiber behavior. Han et el. showed that fibers with low charge levels will oscillate about the trajectory they would have taken if no charge were present [9]. Since it was relatively straightforward to reduce the charge level to Boltzmann equilibrium using a ^{85}Kr source, the use of an alternating electric field ensured proper classification of fibers. Balancing the ac voltage to give zero average voltage was also found to be important to obtain optimum resolution.

Several additional factors were found to be important in separating fibers by this technique. Fibers had to be sufficiently conductive. As noted with the FAM, glass fibers were not sufficiently conductive at relative humidity below 50% to allow optimum separation. At high input aerosol concentrations, a second neutralizer was sometimes used. In addition, the high voltages required to classify fibers were close to the breakdown voltage of air. Therefore, all surfaces that could exacerbate corona discharges were rounded and coated with a thin dielectric layer. In addition, surfaces on which fibers could deposit were coated with an oil and grease mixture to prevent fiber resuspension.

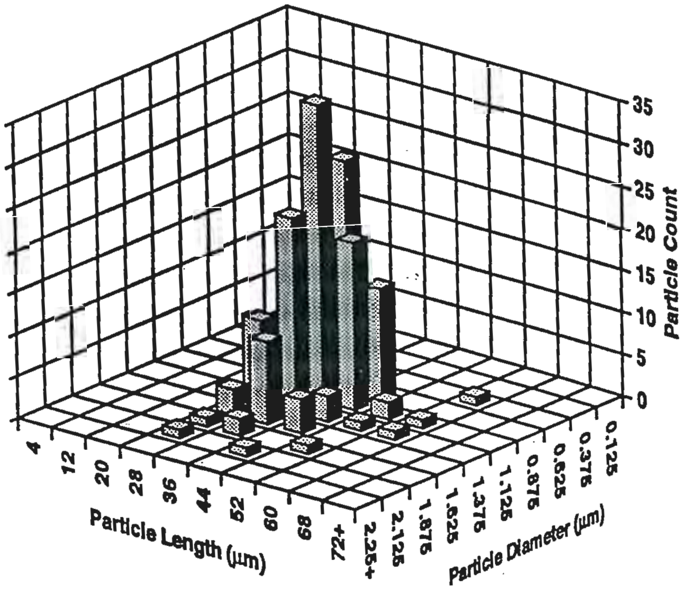

FIG 2--*A size distribution of glass fibers produced with the fiber length classifier and measured using SEM.*

The classifier developed by Baron et al. [8] was modified to operate in a differential mode so that a minor flow was extracted at the bottom of the classifier near the inner tube. This minor flow contained fibers that had been attracted to the inner tube, but had not yet deposited. This flow contained narrow length-distribution fibers, as opposed to the earlier

FIG. 2b--*A picture of glass fibers produced with the fiber length classifier*

classifier in which the exiting flow contained all fibers shorter than the length depositing on the inner tube. In the differential mode, the major flow contained fibers shortest fibers, the longest fibers were deposited on the inner tube, and the intermediate length fibers were in the minor flow.

The fibers in the minor flow were measured as a function of flow rate and voltage. Equation 1 appeared to give an adequate description of the classifier behavior. However, there are still some problems with the classifier since the spread of the classified fiber lengths is about twice that predicted. It is suspected that the region in which the minor and major flows are split may contain turbulence which reduces the resolution of the classifier. An example size distribution is shown in Figure 2a and a picture of the fibers in this distribution is shown in Figure 2b.

CLASSIFIER APPLICATIONS

A fibrous aerosol classifier can be used in two ways. Once the behavior of the classifier has been calibrated, the device can be used to measure fiber lengths. Alternatively, the output of the classifier can be used for other experiments.

The fiber length classifier has been used with several particle counters to indicate size distributions. An optical particle counter was used to indicate the concentration at each of several classifier voltage levels to indicate the fiber length distribution. However, it is often more useful to obtain both length and diameter for a distribution. Several possibilities are available. Inertial sizing has been shown to separate fibers primarily according to diameter. Therefore, with length-classified fibers, an inertial classifier can be used to produce narrow diameter distributions. If this inertial classifier is tunable, then the output of the two classifiers in series can be used to determine a size distribution. We have used a tunable virtual impactor to produce monodisperse diameter fibers. This device was developed by V.Toporkov (Novosibirsk, Russia) and marketed by GIV (Breuberg Germany). This classifier was capable of separating particles in the aerodynamic diameter range of 3 - 7 µm and producing a narrow diameter distribution with a standard deviation of about 10%. One of the problems encountered with the use of both classifiers simultaneously is that the number of fibers penetrating each classifier is on the order of 1% of the aerosol entering the classifier. Thus the concentration at the inlet of the first classifier must be high in order to detect a significant number of fibers at the exit of the second classifier. It was found that high concentrations caused mixing of the aerosol particles in the virtual impactor, producing a distribution with a large fraction of small particles in addition to the expected mode. Reduction of the challenge concentration resulted in poor counting statistics of particles exiting the system.

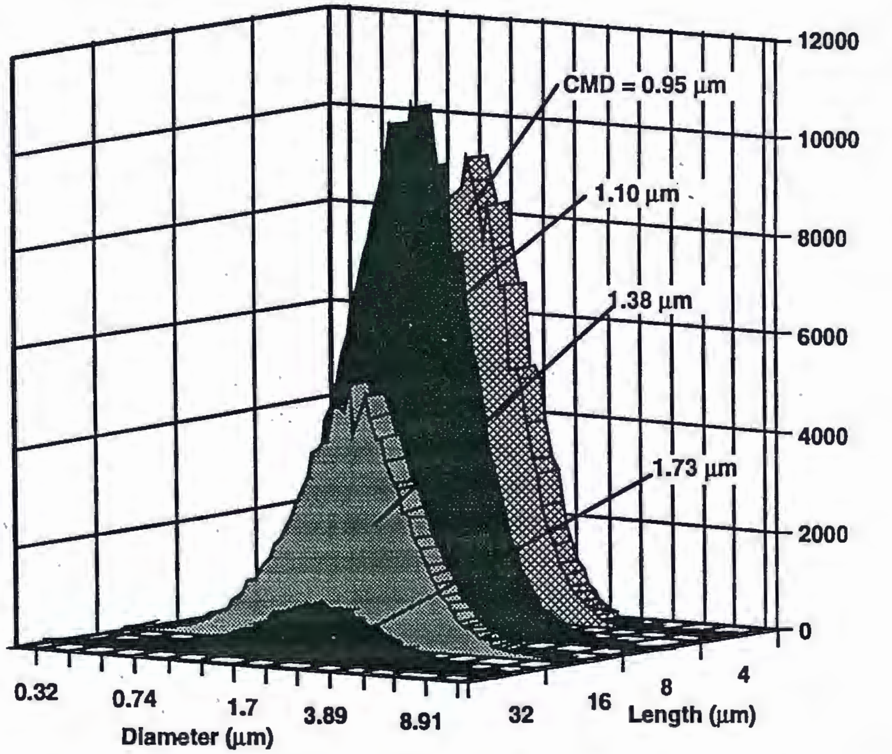

FIG. 3--*An example fiber length/diameter distribution of Owens-Corning AAA10 microfiber measured using a combination of the fiber length classifier and the Aerosizer. The diameter measured with the Aerosizer has not been calibrated and is assumed to be approximately proportional to physical diameter.*

Another way to make length/diameter distributions is to use an aerodynamic sizing instrument to measure the output of the length classifier. Two commercial instruments (Aerodynamic Particle Sizer [APS3300], TSI, Inc. St., Paul MN; Aerosizer, Amherst Process Instruments, Hadley MA) measure the velocity of particles accelerated through a nozzle. This velocity is interpreted in terms of particle aerodynamic diameter. The behavior of fibers in such high acceleration flow fields has not been examined in detail, but it appears possible that these instruments can be calibrated to indicate fiber diameter for known fiber lengths. A calibration curve of this type is being developed for the APS3300. Figure 3 shows a size distribution measured with the Aerosizer. The diameters indicated by the Aerosizer have not been calibrated in this figure, so that the physical diameters cannot be directly estimated. However, it should be noted that approximately 10^6 particles were measured over a time of several minutes, indicating good precision in the description of the distribution. Measurement of such a large number of fibers is simply not feasible by microscopy. Further efforts at calibration of the APS3300 are under way. Figure 4 shows a comparison of APS measured size distribution versus SEM-measured diameter distribution for 10 µm long fibers. The physical diameter observed using SEM had to be multiplied by 2.6 to approximately match the aerodynamic diameter observed in the real time instrument. A comparison of this type must be made for a range of lengths and diameters in order to use the APS3300 for reliable diameter distribution measurements.

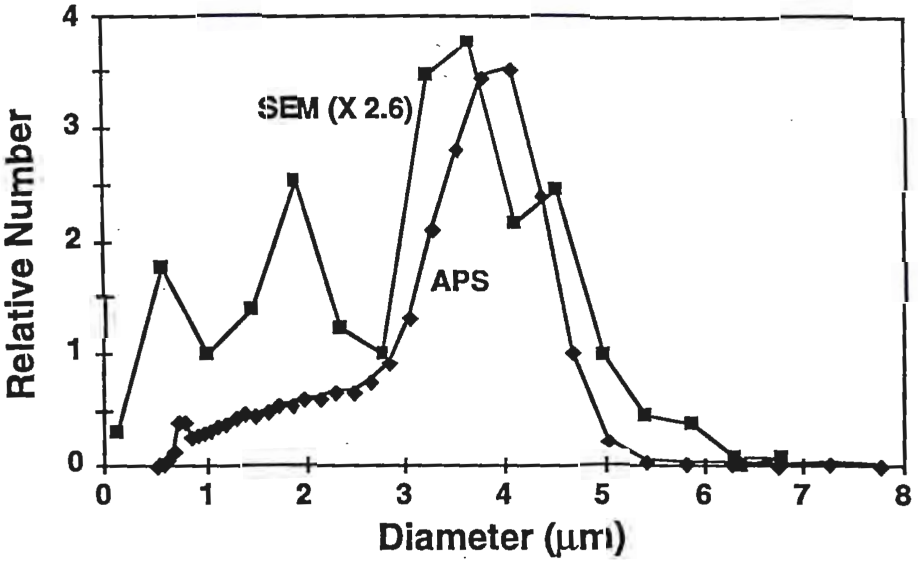

FIG. 4--*Glass fibers (length-classified 10 µm long) were classified with a virtual impactor system and collected for SEM analysis. The diameter of the SEM-measured fibers were adjusted using a factor of 2.6 to match the APS-measured diameter distribution.*

The fiber length classifier can be used for production of small quantities of selected lengths. An evaluation of the physical separation mechanism indicates that it is not feasible to scale the classifier to large dimensions that larger quantities can be generated. However, a successful effort was made to generate milligram quantities of fibers of <5, 5, 8, 22, 35, 50 µm length. These fibers were used in scaled-down *in vitro* assays to determine the effect of fiber length on macrophage response [10]. It was found that there was a clear difference in response between fibers 8 μm and shorter versus the fibers 22 μm and longer. From SEM images of fibers in this study, it was clear that the shorter fibers could be engulfed by the macrophages, while the longer ones penetrated the macrophage walls, causing measurable damage.

Other experiments are also planned. The classifier can be used to quantitatively assess penetration of fibers through other systems and devices. The development of a sampler is planned for the collection of only fibers that might reach the lung region of the respiratory system, defined as thoracic fibers by the International Standards Organization and the American Conference of Governmental Industrial Hygienists [11]. The fiber classifier will aid in the assessment of this sampler. This work is being carried out in collaboration with the Health and Safety Executive in the UK. The length- and diameter-dependent response of the FAM also will be investigated.

CONCLUSION

The fiber length classifier shows great promise in opening up areas of research and understanding of fiber behavior. The classifier has been demonstrated to separate fibers primarily according to length over the range of 5 μm to greater than 50 μm. The instrument can be used for fiber size distribution measurement as well as for production of fibers for other experiments.

DISCLAIMER

The mention of product or company name does not constitute endorsement by the Centers for Disease Control and Prevention.

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