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Electron Microscopy Study of Refractory Ceramic Fibers

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In epidemiological studies designed to identify potential health risks of exposures to synthetic vitreous fibers, the characterization of airborne fiber dimensions may be essential for assessing mechanisms of fiber toxicity. Toward this end, air sampling was conducted as part of an industry-wide study of workers potentially exposed to airborne fibrous dusts during the manufacture of refractory ceramic fibers (RCF) and RCF products. Analyses of a subset of samples obtained on the sample filter as well as on the conductive sampling cowl were performed using both scanning electron microscopy (SEM) and transmission electron microscopy (TEM) to characterize dimensions of airborne fibers. Comparison was made of bivariate fiber size distributions (length and diameter) from air samples analyzed by SEM and by TEM techniques. Results of the analyses indicate that RCF size distributions include fibers small enough in diameter ($< 0.25\ \mu$m) to be unresolved by SEM. However, longer fibers ($> 60\ \mu$m) may go undetected by TEM, as evidenced by the proportion of fibers in this category for TEM and SEM analyses (1% and 5%, respectively). Limitations of the microscopic techniques and differences in fiber-sizing rules for each method are believed to have contributed to the variation among fiber-sizing results. It was concluded from these data that further attempts to characterize RCF exposure in manufacturing and related operations should include analysis by TEM and SEM, since the smallest diameter fibers are not resolved with SEM and the fibers of longer length are not sized by TEM.

Keywords Occupational Exposure, Refractory Ceramic Fibers, Scanning Electron Microscopy, Synthetic Vitreous Fibers, Transmission Electron Microscopy

Synthetic vitreous fibers (SVFs), which encompass mineral wool, glass fiber, and refractory ceramic fibers (RCFs), are heat-resistant and lightweight, making them effective insulation materials. SVFs differ from naturally occurring fibers in morphology and chemical composition; the former are amorphous, glassy, and produced by melting inorganic substances such as rock, clay, slag, or sand. Generally, SVFs are larger in diameter, cleave in the transverse plane, and do not produce fibrils.

RCFs are produced from kaolin clay, alumina/silica, or alumina/silica/zirconia and can resist temperatures up to 2,600°F. This high-heat-resistant property makes RCFs useful for industrial applications such as lining insulation for furnaces and kilns. Since the 1970s, SVF production has increased significantly in the United States, from 247 million kg in 1974 to 632 million kg in 1984.(1) U.S. production of RCFs alone has grown from an estimated 36 million kg in 1990 to approximately 49 million kg (108 million pounds) in 1997.(2,3) Because RCFs are durable in physiological fluids,(4) and their size distribution falls within the respirable range (less than 3.5 μm diameter and less than 200 μm length),(5,6) there has been increased interest in recognizing any potential health effects from occupational exposures. In addition, animal studies have indicated that fiber dimensions and durability may be critical in determining potential health risks.(4,7,8) Recent inhalation studies show RCFs have carcinogenic effects in rats at high doses;(9,10) yet in the same studies the development of mesothelioma in the absence of a clear dose-response relationship suggests other factors may be involved. In this context, fiber dimensions in occupational exposures, though not well-characterized, have been identified as essential risk criteria for assessing the toxicity of man-made fibers.(11)

In an ongoing, industry-wide, prospective morbidity study of workers exposed to RCFs during the manufacture of RCF products, extensive air monitoring has been performed to determine fiber exposure levels using phase contrast microscopy (PCM); additional samples have been collected for analysis by scanning electron microscopy (SEM) and transmission electron microscopy (TEM) to determine fiber size distribution. Fibers collected on the conductive cowl, where it has been shown that a considerable percentage is accumulated during sampling,[12] were also measured and included in the data analysis.

The focus of this report is the results of SEM and TEM analyses of a subset of the samples collected for characterization of fiber dimensions. Analytical cost considerations influenced the development of a strategy for analyzing samples from representative operations, as resources would not allow for analysis of all samples collected. The objectives of this investigation were threefold: to assess any difference between fiber size distributions on sample filters as determined using SEM and TEM; to assess any difference between fiber size distributions on conductive sample cowls as determined using SEM and TEM; and to compare the fiber size distributions found on sample filters and those found on conductive sample cowls. These comparisons were performed for the purpose of determining which samples and analytical techniques should be utilized in further study of RCF size distributions.

Setting an appropriate occupational exposure limit for RCFs continues to be a subject of some debate. The National Institute for Occupational Safety and Health (NIOSH) has recommended that occupational exposure to fibrous glass, mineral wool, and other man-made vitreous fibers be limited to three fibers per cubic centimeter (3 f/cm^3) for up to a 10-hour time-weighted average (TWA).[13] The American Conference of Governmental Industrial Hygienists (ACGIH®) Committee on Chemical Substances Threshold Limit Values (TLVs®) recently published a notice of intended changes which lists a proposed TLV for RCFs at 0.2 f/cm^3 as an 8-hour TWA.[14] The Refractory Ceramic Fibers Coalition (RCFC) has adopted a voluntary industry-recommended exposure guideline of 0.5 f/cm^3 as an 8-hour TWA.[15] The International Agency for Research on Cancer (IARC) classified glass wool, rock wool, slag wool, and ceramic fiber as group 2B, possibly carcinogenic to humans.[1] The U.S. Environmental Protection Agency (EPA) has also classified RCFs as a probable human carcinogen (B2) based on animal data, and has recognized the need for additional human exposure monitoring.[16] Similarly, the ACGIH describes RCF as a suspected human carcinogen (A2).[14]

METHODS

Production Processes

Although the individual production processes vary slightly by plant, all RCF products begin with the manufacture of ceramic fiber. This involves blending of the raw materials in a batch house. The batch mix is then transferred either manually or mechanically to a furnace where the batch is melted. The melted batch material flows from the furnace and is fiberized by either a spinning or blowing process.[17] RCF is either bagged; baled; or manufactured into blankets, felts, boards, textiles, or other specialty products. Many of the manufacturing processes are performed mechanically and monitored by machine operators. Post-production processes such as cutting, sanding, packaging, handling, and shipping are more labor-intensive, although there is potential for exposure to airborne fibers throughout production.

Air Sampling and Analysis

Personal air samples were collected at six RCF manufacturing facilities throughout the United States. Air sampling for fiber counts as determined by PCM was conducted quarterly for 10 percent of the entire workforce at each facility. Of those persons sampled, the protocol called for 1 percent of the workforce to carry simultaneously another pump for collection of the samples to be analyzed for fiber size distributions using SEM and TEM. All workers were randomly selected each quarter from all job categories with potential for RCF exposure. Job categories were defined at the onset of the study and are described elsewhere.[18]

Sampling was conducted according to NIOSH Analytical Method 7400, using a personal sampling pump attached to 25-mm cassettes positioned within the breathing zone of the worker being sampled.[19] Air samples were drawn through the cassettes, which consisted of reusable, nickel-plated, conductive cowls and 0.45-μm pore-size mixed cellulose ester (MCE) membrane filters. Samples were collected at a flow rate in the range of 1.5–2.0 L/min. Following sampling, cassettes were sealed, signed, and returned to the researchers for analysis accompanied by chain-of-custody forms throughout the process.

When sample cassettes were received, the sample filter was removed and placed into a sample tin, which was sealed and labeled. The conductive cowl was rinsed onto a 0.45-μm MCE membrane filter in accordance with a standardized cowl rinsing procedure.[20] The filter containing the cowl rinse was placed into a sample tin which was then sealed and labeled. Over 500 air samples were collected according to this protocol, from which a subset of 124 was selected. This subset was selected using weighted criteria which ensured that the number of samples from specific job areas and tasks was representative of both wet (e.g., wet tanks, vacuum cast) and dry (e.g., finish, chopper) manufacturing processes, and proportional to the number of workers in these areas.

Sample filters and the companion cowl rinse filters were shipped to an independent laboratory for microscopic analysis. The remaining samples have been archived for future analyses. The laboratory was chosen based on its American Industrial Hygiene Association (AIHA) accreditation, certification under the National Voluntary Laboratory Accreditation Program, and its participation in several round-robin exchanges, including a quality-control program developed by researchers at The Johns Hopkins University (JHU) for electron microscopy (EM) analyses of RCFs. In addition, the laboratory had analyzed air samples for a study of RCF exposures among end-users conducted by researchers at JHU.[21] The protocol established for that study regarding sample preparation and choice of EM fiber sizing criteria was followed for this investigation, and is described below.

Samples analyzed by SEM were prepared and measured using the World Health Organization (WHO) method for measuring airborne man-made mineral fibers,[22] with slight modifications as described here. Although the WHO method cautions against the use of MCE filters, which may be unstable in the electron beam, for this investigation MCE filters were used to allow analyses of the same samples by both SEM and TEM techniques.

A quarter section from each of the filters was cut and mounted directly onto an SEM stub using conducting colloidal graphite. The sample was allowed to dry thoroughly, and then was placed inside the bell jar of a vacuum evaporator. A 1 mm × 5 mm section of graphite rod was then evaporated onto the sample. Samples were analyzed by SEM at a magnification of 2,000× to determine fiber dimensions, with energy dispersive x-ray spectroscopy (EDS) providing elemental information. SEM operating conditions were as follows: accelerating voltage—20 kV; working distance—25 mm; spot size—2:00 position; raster rate-fast scan—3 scans/sec; detector type—secondary electron; filament type—tungsten; and magnification calibration—400 mesh standard grid. Fibers meeting the criteria established in NIOSH 7400 B rules (i.e., length $> 5\,\mu$m, diameter $\leq 3\,\mu$m and aspect ratio $\geq$ 5:1) were sized from the SEM screen rather than from photomicrographs.

The TEM samples were prepared according to NIOSH Analytical Method 7402.[19] Samples were analyzed by TEM at a magnification of 20,000× to determine fiber dimensions. The elemental chemistry of each fiber was checked by EDS to confirm that it was "silica rich," and the diffraction pattern of the fiber was checked using selected area electron diffraction (SAED). RCFs are noncrystalline and therefore are identified as having no diffraction pattern. For the TEM analysis, fibers were selected for sizing according to the EPA Asbestos Hazard Emergency Response Act (AHERA) criteria (i.e., length $> 0.5\,\mu$m and aspect ratio $\geq$ 5:1) described in 40CFR763.83. The two EM analyses were conducted on wedges from the same filters; hence, sizing data obtained by both EM analysis methods represent the same air samples but not the same fibers.

Fiber sizing data received from the laboratory were analyzed statistically using SAS software, version 6.04, for the personal computer. Only fibers identified through elemental analysis as silica rich were included in the statistical analysis. Sizing data obtained from the laboratory were log-transformed since fiber dimensions have been demonstrated to be log-normally distributed.[23] As such, the fiber dimensions can be characterized by the geometric mean, geometric standard deviation, and the correlation of the natural logs of the diameter and length. These values were calculated and are presented in Table I. Because it is also equivalent to say that the natural logs of the diameter and length are normally distributed, estimates of the arithmetic median (1), arithmetic mean (2), and standard deviation (3) were derived from the transformed distribution using the following equations[24]:

$$\text{Median} = e^{\mu} \quad [1]$$

$$\text{Mean} = \alpha = e^{\mu + {}^1/_2\sigma^2} \quad [2]$$

$$\text{SD} = \beta = \left\{e^{2\mu+\sigma^2}\left(e^{\sigma^2} - 1\right)\right\}^{1/2} \quad [3]$$

TABLE I

Geometric mean (GM), geometric standard deviation (GSD), and correlation of the natural logs of the diameters and lengths ($R_{d,l}$) of fibers by electron microscopy technique (SEM vs. TEM) and collection location (filter vs. cowl)

Category	GM_{length}	GSD_{length}	$GM_{diameter}$	$GSD_{diameter}$	$R_{d,l}$
A[A]	22.2	1.8	1.2	1.8	0.24
B[B]	6.9	2.9	0.5	2.7	0.70
C[C]	26.0	1.9	1.7	1.7	0.38
D[D]	4.3	4.6	0.3	3.7	0.83

[A]Found on sample filter, analyzed by SEM.
[B]Found on sample filter, analyzed by TEM.
[C]Found on conductive cowl, analyzed by SEM.
[D]Found on conductive cowl, analyzed by TEM.

Fiber size distributions were determined for each of the six facilities. Fiber size distributions also were determined for each of the following job categories: end of line (fiber packaging); shipping; wet processes; front line (furnace); and finishing. Pearson length-diameter correlations (R) of the log-transformed data were determined.[25] Aspect ratios were determined by dividing each fiber length by its diameter. Nonparametric comparison of the log-transformed fiber size distributions for the sampling cowl and filter was performed. The median, two-sample test (normal approximation), which does not require normality of data or homogeneity of variances, was used to compare the medians of the distributions. Comparison of the log-transformed SEM and TEM fiber size distributions also was performed using the median, two-sample test. This test provides a Z-statistic, which was considered for this study to indicate a statistically significant difference if the comparisons produced a Z-statistic with an associated p value < 0.05.

Results of the air sampling analyses were categorized according to where the fibers were collected (air sample filter or conductive cowl) and the analytical technique used. The four categories derived include: a) sample filter/SEM; b) sample filter/TEM; c) conductive cowl/SEM; and d) conductive cowl/TEM.

RESULTS AND DISCUSSION

Fiber sizing was performed using SEM analysis on all 124 samples, 39 of which were also analyzed using TEM. The distribution of samples among the four categories previously described was as follows: 64 samples in category A (sample filter/SEM); 29 samples in category B (sample filter/TEM); 60 samples in category C (conductive cowl/SEM); 10 samples in category D (conductive cowl/TEM). The smaller number of samples in categories C and D (cowls for both SEM and TEM) resulted from initial difficulties with perfecting the cowl washing and analysis techniques.

Size Distributions

The number of fibers identified from both filter and cowl samples in various size categories is displayed in Table II. The

TABLE II
Number of fibers by length and diameter as determined by SEM (S) and TEM (T) from cowl and filter locations combined

	Diameter (μm)							
Length (μm)	0–0.1	>0.1–0.15	>0.15–0.25	>0.25–0.5	>0.5–1.0	>1.0–2.5	>2.5	Row totals
0–2.0	0 S	2 S	0 S	1 S	0 S	0 S	0 S	3 S
	57 T	29 T	42 T	10 T	0 T	0 T	0 T	138 T
>2.0–5.0	0 S	3 S	4 S	9 S	23 S	8 S	2 S	49 S
	17 T	13 T	53 T	59 T	43 T	0 T	0 T	185 T
>5.0–10.0	2 S	2 S	6 S	60 S	178 S	261 S	3 S	512 S
	3 T	7 T	18 T	32 T	81 T	37 T	0 T	178 T
>10.0–20.0	0 S	0 S	11 S	166 S	427 S	1286 S	135 S	2025 S
	1 T	4 T	12 T	32 T	45 T	68 T	6 T	168 T
>20.0–60.0	1 S	0 S	4 S	242 S	666 S	2045 S	461 S	3419 S
	1 T	0 T	5 T	17 T	31 T	47 T	13 T	114 T
>60.0–80.0	0 S	0 S	0 S	11 S	27 S	133 S	67 S	238 S
	0 T	0 T	0 T	0 T	1 T	4 T	2 T	7 T
>80.0	0 S	0 S	0 S	4 S	2 S	20 S	26 S	52 S
	0 T	0 T	0 T	0 T	1 T	1 T	0 T	2 T
Column	3 S	7 S	25 S	493 S	1323 S	3753 S	694 S	6298 S
Totals	79 T	53 T	130 T	150 T	202 T	157 T	21 T	792 T

length and diameter characteristics of the fiber size distributions are presented by plant and by job in Tables III and IV, respectively. With regard to sizing, fiber length and diameter values for category C (conductive cowl/SEM) proved to be the largest. There are two probable explanations for this. The first is the higher limit of resolution for SEM, which allows very small fibers (diameter < 0.25 μm) to go largely undetected, thereby skewing the fiber distribution toward the larger-diameter fibers. Another possible explanation is the size-selective collection of longer fibers on the cowl, possibly by impaction or interception, although the exact mechanism of fiber deposition on the cowl has not been well-characterized. Similarly, the size distributions for category B (sample filter/TEM) contained the smallest length distributions; one exception is noted in Table I where category D (conductive cowl/TEM) appears to have fibers with the smallest geometric mean length. The smallest mean diameter distribution was found in category D, which is not what is expected based on the inertial effects. This phenomenon may be explained by the small number of fibers evaluated in this category ($n = 62$) on the 10 samples. A general trend observed across the four categories (by analytical method/collection site) in Tables I, III, and IV indicates that fibers in category C (conductive cowl/SEM) had lengths and diameters greater than those of category A (sample filter/SEM). Overall, these results are in general agreement with a study by Robbins et al.[26] of end-user RCF size distributions determined using SEM, in which fibers found on conductive cowls were larger in diameter than fibers found on sample filters. This trend was not apparent for fibers analyzed by TEM, as evidenced by the comparability among values listed in categories B and D. It is also noted that fiber lengths and diameters as measured by TEM (categories B and D) are less than those measured by SEM (categories A and C).

SEM versus TEM

Significant differences were found between the SEM and TEM fiber size distributions: for sample filter length (SEM vs. TEM), $Z = -19.9$ ($p < 0.0001$); for sample filter diameter (SEM vs. TEM), $Z = -16.9$ ($p < 0.0001$); for cowl length (SEM vs. TEM), $Z = -5.1$ ($p < 0.0001$); and for cowl diameter (SEM vs. TEM), $Z = -5.3$ ($p < 0.0001$). This is likely due to the inability to resolve the smaller fibers with SEM, evidenced by the small proportion of fibers detected by SEM with diameters less than 0.25 μm, shown in Table II. The inability to resolve or include longer fibers using TEM may account for the small proportion of fibers with lengths greater than 60 μm identified by TEM (Table II), since longer fibers may extend beyond the viewing grid, which has dimensions of 84 μm $\times$ 84 μm. Only 1 percent (9/792) of all fibers identified by TEM measured greater than 60 μm in length, as compared to 5 percent (260/6298) of all fibers measured by SEM. Differences between criteria for sizing fibers, specifically the NIOSH 7400 B rules for SEM and the EPA AHERA rules for TEM, are likely to have affected the sizing distributions determined by the two microscopy methods. For example, fibers less than 5 μm in length would generally not be sized by SEM, but would be sized by TEM if the criteria of having an aspect ratio greater than or equal to 5:1 was met. Note that fibers less than or equal to 5 μm in length comprise 41 percent (323/792) of all fibers evaluated by TEM; of the fibers analyzed by SEM, less than 1 percent (52/6298) were sized and reported as less than or equal to 5 μm in length.

TABLE III

Descriptive statistics for fibers sized by analytical method and collection site (Categories A–D) and by plant

	Category A[A]				Category B[B]				Category C[C]				Category D[D]			
	L (μm)		D (μm)		L (μm)		D (μm)		L (μm)		D (μm)		L (μm)		D (μm)	
Plant	Median	Mean (SD)	Median	Mean (SD)	Median	Mean (SD)	Median	Mean (SD)	Median	Mean (SD)	Median	Mean (SD)	Median	Mean (SD)	Median	Mean (SD)
1	18.3	22.3 (15.7)	1.4	1.7 (1.0)	6.7	10.6 (13.2)	0.6	0.9 (1.0)	24.7	28.8 (17.3)	1.8	2.0 (0.9)	2.2	4.2 (6.8)	0.2	0.3 (0.3)
2	25.6	29.7 (17.7)	1.2	1.5 (1.0)	8.7	14.6 (19.7)	1.0	1.0 (1.5)	27.2	31.7 (19.0)	2.0	2.1 (0.7)	N/A	N/A (N/A)	N/A	N/A (N/A)
3	23.0	24.5 (8.9)	0.9	1.0 (1.0)	6.5	11.2 (15.9)	0.4	0.6 (0.8)	24.9	27.2 (11.9)	1.3	1.4 (0.5)	6.6	15.3 (32.1)	0.4	0.4 (0.3)
4	21.4	24.7 (14.3)	1.2	1.5 (1.0)	6.7	12.1 (18.3)	0.4	0.7 (0.9)	29.9	34.7 (20.2)	1.8	2.0 (1.2)	5.3	17.6 (55.4)	0.4	1.1 (3.2)
5	20.6	25.5 (18.5)	1.2	1.5 (1.1)	5.1	11.4 (23.1)	0.4	0.7 (1.1)	36.7	43.6 (27.9)	1.4	1.7 (1.2)	5.1	25.2 (121)	0.4	0.8 (2.1)
6	25.5	30.3 (19.6)	1.5	1.7 (0.9)	7.7	15.1 (29.8)	0.5	0.8 (1.2)	34.0	40.8 (25.9)	1.8	1.8 (0.7)	12.5	74.3 (429)	1.0	3.7 (11.3)
	N = 4461				N = 730				N = 1837				N = 62			

L = fiber length; D = fiber diameter; SD = estimated standard deviation; N/A = none available; N = number of fibers sized per category.
[A]Found on sample filter, analyzed by SEM.
[B]Found on sample filter, analyzed by TEM.
[C]Found on conductive cowl, analyzed by SEM.
[D]Found on conductive cowl, analyzed by TEM.

TABLE IV
Geometric size distributions for fibers sized by analytical method and collection site (Categories A–D) and by job

	Category A[A]				Category B[B]				Category C[C]				Category D[D]			
	L (μm)		D (μm)		L (μm)		D (μm)		L (μm)		D (μm)		L (μm)		D (μm)	
Job	Median	Mean (SD)	Median	Mean (SD)	Median	Mean (SD)	Median	Mean (SD)	Median	Mean (SD)	Median	Mean (SD)	Median	Mean (SD)	Median	Mean (SD)
End of line	24.8	27.2 (12.4)	1.1	1.3 (0.8)	7.4	13.5 (20.8)	0.4	0.7 (1.0)	26.0	28.7 (13.3)	1.4	1.5 (0.6)	9.2	30.6 (97.0)	0.6	1.9 (6.0)
Shipping	19.5	23.9 (16.7)	1.2	1.4 (1.0)	7.4	11.9 (15.0)	0.6	1.0 (1.1)	24.1	28.7 (18.4)	1.8	2.0 (1.0)	3.9	11.6 (33.3)	0.2	0.2 (0.2)
Wet process	19.6	23.4 (15.2)	1.5	1.7 (1.0)	6.5	11.6 (16.9)	0.5	3.6 (2.6)	26.6	30.4 (16.8)	1.9	2.1 (0.9)	3.4	9.4 (24.5)	0.3	1.8 (11.6)
Front line	24.4	27.7 (15.0)	1.3	1.5 (0.9)	7.5	15.0 (26.3)	0.4	0.7 (0.9)	38.4	43.0 (21.9)	1.8	2.0 (1.0)	N/A	N/A (N/A)	N/A	N/A (N/A)
Finishing	22.1	25.8 (15.6)	1.3	1.5 (1.0)	6.2	10.8 (15.4)	0.4	0.7 (0.8)	27.3	33.1 (22.7)	1.6	1.8 (1.0)	5.1	25.2 (122)	0.3	0.8 (2.1)
	N = 4461				N = 730				N = 1837				N = 62			

L = fiber length; D = fiber diameter; SD = estimated standard deviation; N/A = none available; N = number of fibers sized per category.
[A]Found on sample filter, analyzed by SEM.
[B]Found on sample filter, analyzed by TEM.
[C]Found on conductive cowl, analyzed by SEM.
[D]Found on conductive cowl, analyzed by TEM.

By Plant and Job Analyses

Generally, appreciable differences in fiber dimensions by plant did not exist (Table III). However, the estimated mean diameters for one plant were consistently among the smallest, while the lengths were not. No additional patterns in terms of length or diameter distributions existed among the six plants.

Results of the by job analyses of fiber size distributions are presented in Table IV. Again, no appreciable differences existed among the five job categories. However, the Front Line job consistently had the longest fibers in all four categories, while the End of Line job had the thinnest fibers in three of the four categories.

Length-Diameter Correlation

Pearson length-diameter correlations of the log-transformed data on fiber dimensions in the four categories are stronger in TEM analyses for both sample filters (category B, $R = 0.83$) and conductive cowls (category D, $R = 0.70$) than are the SEM-analyzed sample filters (category A, $R = 0.24$) and conductive cowls (category C, $R = 0.38$). This may result from the inability to resolve the very small diameter fibers with SEM. Consequently, as fiber length decreases, fiber diameter decreases but stops artificially near the SEM limit of resolution (~0.25 μm), resulting in a very weak correlation. Also, fiber exclusion criteria for SEM sizing analysis is more strict than that of the TEM method.

Aspect Ratio

The estimated mean aspect ratios proved to be relatively large (19.2 to 22.9), indicating that the fibers characterized by both methods are predominantly long and thin. Specifically, the mean and median (and standard deviation) for the aspect ratios were the following: sample filter/SEM—20.1, 13.9 (21.1); sample filter/TEM—19.2, 15.6 (13.5); conductive cowl/SEM—19.3, 14.0 (18.3); and conductive cowl/TEM—22.9, 17.6 (22.9).

Cowl versus Filter

Significant differences were found between the conductive cowl and sample filter fiber distributions for both SEM length ($Z = 11.4$, $p < 0.0001$) and diameter ($Z = 16.5$, $p < 0.0001$) with the cowl fibers being larger in both dimensions. Significant differences were also found between cowl and sample filter fiber distributions analyzed by TEM [length ($Z = -2.1$, $p < 0.036$) and diameter ($Z = -2.05$, $p < 0.04$)], as the cowl fibers were larger in length only. This may result from the inertial forces affecting the larger fibers as they enter the cassette.

CONCLUSIONS

From this initial analysis of a subset of samples, it was concluded that RCF size distributions include fibers too small in diameter (< 0.25 μm) to be resolved by SEM. By contrast, analysis of samples by TEM enables detection of all fiber sizes when the microscope is operated in the size-selective mode.(27) When no size selection is specified, TEM fiber measurements will be confined to shorter fibers which are more numerous. Of the fibers sized by TEM, only 1 percent measured longer than 60 μm, which suggests that longer fibers may not be detected by TEM or may be excluded under TEM fiber sizing criteria; 5 percent of the fibers sized by SEM exceeded 60 μm in length. It also was noted that significant differences exist between fiber length and diameter distributions found on the sample filter and the conductive cowl Small-diameter fibers are found on both the sample filter and conductive cowl; however, fibers found on conductive cowls, appear to be longer.

On the basis of this investigation, analysis of the additional archived air samples using TEM is planned. Study results which have become available since the start of this investigation have also indicated that TEM is preferable for use with MCE filters.(28) However, fiber loss associated with the preparation of samples for TEM analysis and a potential bias toward shorter fibers with TEM may explain why TEM is less sensitive for resolving the longest fibers. Therefore, SEM may be more useful for measuring distributions of longer fibers collected on appropriate filters. Future studies will continue to include fibers rinsed from the inner walls of the sampling cowl.

The implications of these findings are of practical importance. Depending upon the analytic technique by which sizing information on airborne fibers is obtained, the application of these data to health studies or for implementing controls to limit workplace exposures could have considerable effects on their outcomes.

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