



Silicon Carbide Whiskers: Characterization and Aerodynamic Behaviors

Yung-Sung Cheng , Quint H. Powell , Shawna M. Smith & Neil F. Johnson

To cite this article: Yung-Sung Cheng , Quint H. Powell , Shawna M. Smith & Neil F. Johnson (1995) Silicon Carbide Whiskers: Characterization and Aerodynamic Behaviors, American Industrial Hygiene Association Journal, 56:10, 970-978, DOI: [10.1080/15428119591016395](https://doi.org/10.1080/15428119591016395)

To link to this article: <https://doi.org/10.1080/15428119591016395>



Published online: 04 Jun 2010.



Submit your article to this journal [↗](#)



Article views: 9



View related articles [↗](#)



Citing articles: 2 View citing articles [↗](#)

SILICON CARBIDE WHISKERS: CHARACTERIZATION AND AERODYNAMIC BEHAVIORS

Yung-Sung Cheng^{a*}

Quint H. Powell^b

Shawna M. Smith^a

Neil F. Johnson^a

^aInhalation Toxicology Research Institute, P.O. Box 5890, Albuquerque, NM 87185; ^bUniversity of New Mexico, Albuquerque, NM 87106

Silicon carbide (SiC) whiskers are fiberlike materials with a wide range of industrial applications. Industrial hygiene samplings of the material are taken to monitor and control possible exposures to workers. This study characterizes an SiC whisker in detail, including its width-length distribution, aspect ratio, particle density, and aerodynamic size distribution. The SiC whiskers were aerosolized, and samples from a filter, cascade impactor, and aerosol centrifuge were taken. The diameter-length distribution of SiC fibers determined by electron microscopy from filter samples was found to follow the bivariate lognormal distribution. The aerodynamic size of a fiber aerosol depends not only on the particle dimension and density but also on the orientation of its axis with respect to flow. The results show that the aerodynamic size distribution obtained from the impactor was consistent with the predicted value, assuming the long axis of the fiber was parallel to the flow toward the collection substrate. On the other hand, the aerodynamic size in the aerosol centrifuge was consistent with results for a perpendicular orientation. A larger aerodynamic size (20–25%) was obtained in the case of impactor data as compared with centrifuge data. The respirable fraction estimated from the cascade impactor data was 65%, consistent with the estimate from bivariate analysis (67%) but smaller than the estimated fraction from the aerosol centrifuge (76%). The results show that the data obtained with the bivariate analysis of fiber dimensions had good correlation with the cascade impactor data, and this approach can be used to predict the aerodynamic size distribution and the size-selective fractions for fiber aerosols from filter samples.

Silicon carbide (SiC) whiskers are manufactured fibrous materials with a single crystal structure.⁽¹⁾ This material has a wide range of industrial uses related to its high tensile strength, weight advantage over metals, and stability at high temperature. SiC particles in both fibrous and nonfibrous

forms have been detected in occupational environments.^(1,2) The dimensions of SiC whiskers are similar to some forms of asbestos, thus raising the concern of potential health effects for workers exposed in occupational environments. The cytotoxicity of SiC fibers has also been found to be comparable with that of asbestos fibers.^(3–6) Stanton et al.⁽⁷⁾ showed that SiC fibers implanted into the pleural cavity of rats consistently result in the formation of mesothelioma. Finally, Bogoroch and Luck⁽⁸⁾ and Lapin et al.⁽⁹⁾ showed that a 3-wk inhalation exposure to 500–7500 SiC fibers/mL results in pleural thickening and increased cellularity in the lungs of exposed rats.

Physical parameters of fibers, including the length, diameter, and density, are important factors in determining their respirability, deposition, and clearance in the respiratory tract. The aerodynamic behavior of a fiber aerosol is the major parameter influencing respirability and deposition in the lung. The aerodynamic diameter of a fiber aerosol is a function of its physical dimensions and orientation with respect to flow. When a fiber particle is suspended in an airflow stream with the long axis parallel to the direction of flow, the resistance is lower than if the long axis is perpendicular to the flow. For example, the aerodynamic diameter of a carbon aerosol would be 15–25% higher in a parallel orientation than in a perpendicular one.⁽¹⁰⁾ There are indications that the measured aerodynamic diameter in many sampling instruments (such as cascade impactors, aerosol centrifuges, and the aerodynamic particle sizers) may be influenced by preferred orientations. Stöber et al.⁽¹¹⁾ showed that the aerodynamic diameter of asbestos fibers collected in an aerosol centrifuge is consistent with the long axis of the fiber being perpendicular to the flow toward the collection foil. Using linear chain aggregates of iron oxide particles to simulate fiber aerosols, Kasper and Shaw⁽¹²⁾ measured the aerodynamic and mobility diameters of the aerosols with an impactor, a centrifuge, and an electrical aerosol analyzer. The ratio of the aerodynamic diameters determined by the impactor and the aerosol centrifuge ranged from 1.15 to 1.28, close to the ratio predicted for fibers collected in parallel and perpendicular orientations.

* Author to whom correspondence should be sent.

In industrial hygiene sampling, fiber aerosols are frequently collected on filters and examined under an electron or optical microscope for fiber identification, number concentration, and diameter-length distribution. The diameter-length distribution of asbestos fibers has been shown to follow the bivariate lognormal distribution.⁽¹³⁾ Based on the bivariate distribution, aerodynamic diameters in several orientations with respect to the flow can be predicted.⁽¹³⁾ However, the predicted values from the bivariate analysis should be compared with the measured aerodynamic diameters using existing sampling devices, such as an impactor, to determine the validity of this method.

Here, methods are described for generating aerosols of SiC fibers in the laboratory and for characterizing the physical and aerodynamic properties of the bulk and aerosol forms of the whisker. Diameter and length distributions of bulk and airborne materials were determined using a transmission electron microscope (TEM). An aerosol centrifuge and cascade impactor were used to determine the aerodynamic diameters and the flow orientation of SiC fibers in these instruments. The diameter-length distribution of the SiC aerosol was determined and used to predict the aerodynamic diameter. This value was compared with the aerodynamic diameter measured by a cascade impactor.

METHODS

Physicochemical Characterization of Bulk Material

Industrial-grade bulk SiC fibers (Lot Number 8291, Grade SC-9) were obtained from Advanced Composite Material Corp. (Greer, S.C.). Detailed analyses of physicochemical properties have been reported⁽⁶⁾ and are briefly summarized here. Whisker density was determined as $4.29 \pm 0.2 \text{ g cm}^{-3}$ by a helium pycnometer (Multipycnometer, Quantachrome Corp., Syosset, N.Y.). This value was higher than the theoretical density of 3.217 g cm^{-3} ,⁽¹⁴⁾ probably due to experimental errors associated with insufficient amount of bulk material used for the measurement. The authors remeasured the density using a larger quantity of the bulk material, and a value of $3.25 \pm 0.006 \text{ g cm}^{-3}$ was obtained. This value is close to the theoretical value and is used throughout the study. The specific surface area of the material was $1.4 \pm 0.1 \text{ m}^2 \text{ g}^{-1}$ determined by the Brunauer-Emmett-Teller analytic technique with the Quantasorb/Quantatecator System (Quantachrome Corp.). X-ray microanalysis of SiC whiskers showed a composition of predominantly silicon and presumably carbon, which was out of the range of the X-ray detector (Figure 1). Traces of tin, calcium, aluminum, sulfur, and manganese were identified in a small fraction of the whiskers.

Aerosol Generation

Many dry powder generation methods, including a fluid bed generator, Jet-O-Mizer/screw feeder, dry powder nebulizer, and a small-scale powder disperser (SSPD, Model 3400, TSI, St. Paul, Minn.), were attempted. Among these methods, only the SSPD succeeded in producing aerosols of SiC fibers. The SSPD consisted of a rotational disk coated with powder and a suction tube to take up the fibers from the disk and disperse them into aerosol form. The aerosol was delivered into a stainless steel

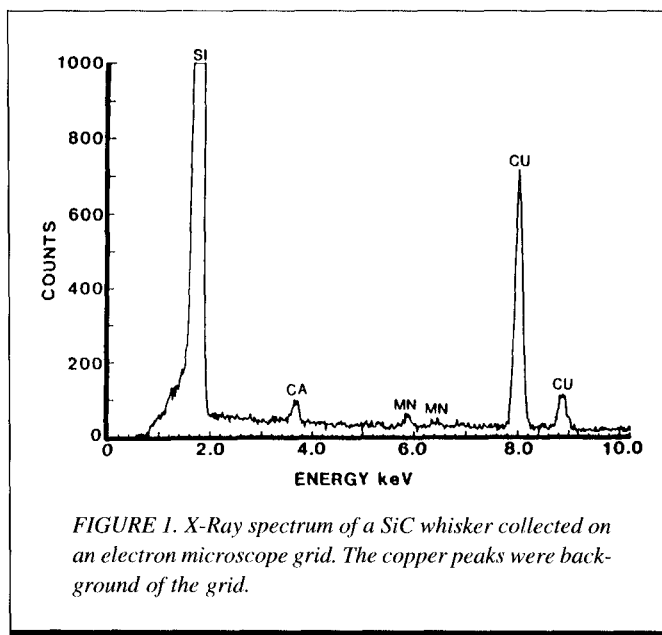


FIGURE 1. X-Ray spectrum of a SiC whisker collected on an electron microscope grid. The copper peaks were background of the grid.

chamber at a flow rate of 22.5 L min^{-1} . The generator, chamber, filter, and impactor samplers were housed in a vented glovebox enclosure.

Aerosol samples were taken from the chamber. Filter samples (25 mm 0.3- μm pore mixed cellulose acetate-cellulose nitrate filter, Millipore Inc., Bedford, MA) were taken to determine aerosol concentration and also for electron microscopy. Impactor samples (Lovelace Multijet Impactor, In Tox Prod., Albuquerque, N.M.)⁽¹⁵⁾ were taken to determine aerodynamic particle sizes. Stainless steel plates coated with Apeazon were used as collection substrates. A Lovelace aerosol particle separator (LAPS)^(16,17) was used to separate the fibers into monodisperse fractions according to aerodynamic diameter. A RAM-S unit (MIE, Bedford, MA) was used for real-time monitoring of the stability of the aerosol mass concentration. The flow rates were 2 L min^{-1} for the filter sampler and the RAM-S and 12 L min^{-1} for the cascade impactor.

Size Classification in the LAPS

A LAPS was used to separate the fibers into monodisperse fractions according to their aerodynamic diameters. Electron microscope grids were placed in several locations of the collection foil corresponding to the different aerodynamic sizes. The aerosol flow into the LAPS was 300 mL min^{-1} , and the total flow rate was 5 L min^{-1} . The LAPS rotated at 3620 rpm. Under these conditions, the calibration curve of the particle separator using monodisperse polystyrene latex particles was developed for relating the aerodynamic resistance diameter from the distance in the collection foil.⁽¹⁷⁾

Fiber Morphology and Dimensions

For a detailed morphological and fiber sizing analyses, known concentrations (0.2 mg mL^{-1}) of the bulk sample were prepared in filtered 70% ethyl alcohol. The samples were mildly

sonicated for 5 min in a ultrasonic cleaner rated at 125 W (Model 8845-4, Cole-Parmer Instrument Co., Chicago, Ill.). Membrane filters (0.3- μm pore mixed cellulose acetate-cellulose nitrate filter, Millipore Inc.) were prepared by gentle vacuum filtration to achieve a concentration of between 20 and 150 μg of material per filter. The filters were then dried at room temperature in a dust-free atmosphere, carbon-coated, mounted onto 3-mm copper grids, and dissolved in acetone vapor. The aerosol samples collected on the same filter material were prepared similarly for microscopic analysis. Size-classified samples were collected directly on electron microscope grids placed on the collection foil inside an aerosol centrifuge. The grids were treated with a drop of ethanol to make SiC whiskers lie flat on the substrates.⁽¹⁷⁾ The grids were then coated with a thin layer of carbon film.

The grids were examined and photographed in a JEOL 1000CX electron microscope at a magnification of 2000. The photographic negatives were enlarged to a final magnification of 6000, and montages were prepared from the prints. The lengths and diameters of the fibers were measured directly from a photographic montage. Fiber diameters were measured by using a 6 \times magnifying eye piece and a reticle displaying 100-mm divisions. The reticle was also used to measure fibers < 2.5 μm in length; longer fibers were measured to the nearest 0.5 μm (actual length on the photograph) with a metric draftman's ruler. Approximately 300-500 individual fibers from each sample were measured to derive a diameter-length distribution for the sample. For particles with nonuniform diameters, the shortest diameter was recorded following the World Health Organization procedure.⁽¹⁸⁾

AERODYNAMIC DIAMETER OF ELONGATED PARTICLES

The relationship between the aerodynamic resistance diameter (d_{ar}), aerodynamic equivalent diameter (d_{ac}), and the dimensions of the fiber can be expressed as:⁽¹³⁾

$$d_{ar} = d_{ac} \sqrt{C(d_{ac})} = \sqrt{\frac{\rho C(d_a)}{\rho_0 \chi}} d_{vc}, \quad (1)$$

where d_{vc} is the volume equivalent diameter ($= (1.5 W^2 L)^{1/3}$), ρ and ρ_0 are the unit and particle densities of SiC, d_a is the adjusted sphere diameter for calculation of the slip correction factor, and χ is the dynamic shape factor. The dynamic shape factor of a cylindrical fiber is a function of the orientation of the fiber axis with respect to the flow direction. Figure 2 shows data of dynamic shape factors for cylinders with their long axis perpendicular to the flow χ_{\perp} and parallel to the flow (χ_{\parallel}) as a function of the aspect ratio ($q = L/W$).⁽¹⁹⁻²¹⁾ The dynamic shape factors predicted from the theory for prolates⁽²²⁾ agree quite well, as shown in Figure 2:

$$\chi_{\perp} = \frac{\frac{8}{3} (q^2 - 1) q^{1/3}}{(2q^2 - 3) \sqrt{(q^2 - 1)} \ln (q + \sqrt{(q^2 - 1)}) + q}, \quad (2)$$

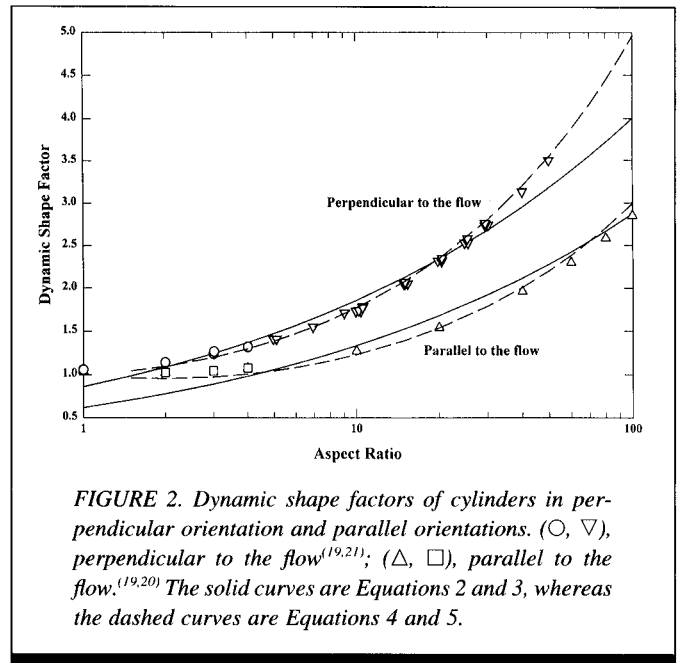


FIGURE 2. Dynamic shape factors of cylinders in perpendicular orientation and parallel orientations. (\circ , ∇), perpendicular to the flow^(19,21); (Δ , \square), parallel to the flow.^(19,20) The solid curves are Equations 2 and 3, whereas the dashed curves are Equations 4 and 5.

$$\chi_{\parallel} = \frac{\frac{4}{3} (q^{-2} - 1) q^{-1/3}}{(2q^2 - 1) \sqrt{(q^2 - 1)} \ln (q + \sqrt{(q^2 - 1)}) - q}. \quad (3)$$

Simplified equations to fit the same data are obtained as follows:

$$\chi_{\perp} = 0.862q^{1/3}, \quad (4)$$

$$\chi_{\parallel} = 0.619q^{1/3}. \quad (5)$$

Equation 1 can be rearranged to

$$\frac{d_{ar}}{WC(d_a)} = \sqrt{\frac{\rho}{\rho_0 \chi}} (1.5q)^{1/3}. \quad (6)$$

Substituting Equations 2-5 into 6, the right side of the equation is now only a function of the aspect ratio, q . The adjusted sphere diameter, d_a , can be calculated as follows:^(23,24)

$$d_a = \frac{d_{vc} G}{\chi}, \quad (7)$$

$$G_{\perp} = 0.216q^{-2/3} (0.763 + 4.436q), \quad (8)$$

$$G_{\parallel} = 0.216q^{-2/3} (3.693 + 1.526q). \quad (9)$$

G , χ , and d_a are orientation dependent. The adjusted sphere diameter can then be used to calculate the slip correction factor:^(25,26)

$$C(d_a) = 1 + \frac{2\lambda}{d_a} \left(1.142 + 0.558 \exp \left(-0.999 \left(\frac{d_a}{2\lambda} \right) \right) \right). \quad (10)$$



FIGURE 3. TEM of bulk SiC fibers

The aerodynamic equivalent diameter, d_{ac} , can be used in Equation 6 to arrive at:

$$\frac{d_{ac}}{W} = \sqrt{\frac{\rho C(d_a)}{\rho_0 \chi C(d_{ac})}} (1.5q)^{1/3}. \quad (11)$$

This expression can be further simplified by assuming

$$\sqrt{C(d_a)/C(d_{ac})} = 1, \quad (12)$$

or a constant value of k closer to 1, and by substituting the simplified expression for χ (Equations 4 and 5):

$$d_{ac} = \sqrt{\frac{\rho}{\rho_0 S}} (1.5)^{1/3} W^{5/6} L^{1/6}, \quad (13)$$

TABLE I. Length-Diameter Distribution of SiC Aerosol

	Diameter (μm)								
	0-0.05	0.05-0.1	0.1-0.2	0.2-0.3	0.3-0.4	0.4-0.6	0.6-0.8	0.8-1.0	>1.0
0-2	8	51	59	31	15	9	1	1	
2-4	2	18	48	26	28	15	1		
4-6	1	5	15	19	19	13	2		
6-8		3	8	12	12	6	3		
8-10			5	8	14	7	3		
10-15			5	14	14	12	4		1
>15			7	5	12	17	2	1	

N = 552

where $S = 0.862$ and 0.619 for perpendicular and parallel orientations, respectively. If the joint width-length distribution can be shown to follow the bivariate lognormal distribution, then the aerodynamic equivalent diameter, d_{ac} , can be shown to follow the lognormal distribution with a count median diameter of⁽¹³⁾

$$CMd_{ac} = \sqrt{\frac{\rho}{\rho_0 S}} (1.5)^{1/3} (CMW)^{5/6} (CML)^{1/6}, \quad (14)$$

where CMW and CML are the count median width and length of the distribution. The geometric standard deviation of the aerodynamic equivalent diameter is

$$\sigma_g = \exp[(0.694\beta_L^2 + 0.0278\beta_W^2 + 0.278\tau\beta_W\beta_L)^{1/2}], \quad (15)$$

where β_L and β_W are $\ln(\sigma_L)$ and $\ln(\sigma_W)$, respectively, and τ is the correlation between fiber length and width.⁽¹³⁾ The mass medium aerodynamic diameter (MMd_{ac}) can then be calculated using the Hatch-Choate equation.⁽²⁷⁾

Size Selective Fractions of SiC Aerosols

The inspirable, thoracic, and respirable fractions of the SiC fiber were estimated from the aerodynamic size (d_{ac}) distribution based on the 1992 American Conference of Governmental Hygienists (ACGIH) definition.^(28,29) For polydisperse aerosols, the size-selective fractions were calculated by integrating the individual definition over the aerodynamic size range of the SiC fibers:

$$RF = \int_0^\infty \text{Definition} \frac{1}{\sqrt{2\pi}d_{ac} \ln \sigma_g} \exp\left(-\frac{x^2}{2}\right) dd_{ac}. \quad (16)$$

Calculations were performed using the Mathcad[®] 4.0 software (MathSoft Inc., Cambridge, Mass.) on a desktop computer.

RESULTS

The SiC fiber aerosol was delivered into a chamber. Filter samples (Millipore 0.3 μm PHWP membrane filter) were taken to determine the aerosol concentration and for electron microscopy. The aerosol concentration was maintained at about 0.2-0.6

TABLE II. Length–Diameter Distribution of SiC Bulk Material

	Diameter (μm)								
	0–0.05	0.05–0.1	0.1–0.2	0.2–0.3	0.3–0.4	0.4–0.6	0.6–0.8	0.8–1.0	>1.0
0–2	7	14	39	6	12	7			
2–4	1	5	27	12	25	17	2		
4–6	1	3	6	5	12	9	3	1	1
6–8			4	7	7	6	5		
8–10			3	6	9	6	4		
10–15			5	9	8	8	2		1
>15			4	2	10	9	4		

N = 334

mg m^{-3} according to filter samples taken during runs ranging from 2 to 6 hr.

Fiber Morphology and Dimensions

The diameter–length distributions from both bulk and aerosol samples were determined from filter samples. Figure 3 shows the montage picture of a filter sample of bulk SiC. The majority of SiC whiskers was straight rods, but some were hooked with one or two straight bends. The percentages of whiskers with a single bend and double bends were 12 and 2.7%, respectively.

Tables I and II list the length–width matrix of bulk material and aerosolized SiC whiskers obtained by electron microscopy. The diameter and length distributions of the bulk and aerosol samples were similar, indicating that all SiC fibers can be aerosolized. Both the length and diameter distributions can be described as lognormal. As discussed previously,⁽¹³⁾ the joint diameter–length distribution can be represented by a bivariate-lognormal distribution. Table III lists the parameters of the diameter and length distributions. The count median length (CML = 3.43 μm) of the aerosol sample was smaller than that of the bulk sample (CML = 3.97 μm) at $p < 0.05$ using t-test between the means. Similarly, the count median width (CMW = 0.198 μm) of the aerosol sample was smaller than that of the bulk sample (CMW = 0.224 μm) at $p < 0.005$ using t-test between the means. The median aspect ratios of the bulk material and aerosol sample were 17.7 and 17.3, respectively; they were not statistically different. This is consistent with the fact that more losses may be expected for larger fibers because of deposition in the chamber and delivery line. Therefore, the aerosolized SiC fibers are smaller than the bulk fibers.

Using Equations 13–15, the aerodynamic equivalent diameters of the SiC aerosols were calculated from the bivariate analysis for both perpendicular and parallel orientations. The MMd_{ac} were 2.24 and 2.78 μm for perpendicular and parallel orientation,

respectively, and the geometric standard deviation was 1.85. The ratio of the two values of the aerodynamic diameter was 1.24. The calculated aerodynamic diameter was compared with that measured by the impactor.

Data from LAPS

Size-classified SiC samplers from electron microscope grids placed at different locations in the collection foil of the LAPS were analyzed under TEM for fiber dimensions. The aerodynamic particle sizes of these grids ranged from 0.65 to 1.9 μm . Transmission electron photomicrographs from seven electron microscope grids were taken, and dimensions of individual fibers were measured using an Edscop eyepiece. Between 53 and 194 fibers were examined in each grid. The length and diameter distributions of the size-classified particles are listed in Table IV. Our results showed a good separation of particles by diameter as indicated by the small standard deviation (Figure 4).

The relationship between the fiber dimension and the aerodynamic diameter was expressed in Equation 6. The dynamic shape and slip correction factors for a prolate particle with its long axis perpendicular and parallel to the flow toward the collection foil were calculated using Equations 2, 3, and 7–10. Figure 5, A and B, shows the relationship of the aerodynamic resistance diameter and the aspect ratio of the fiber and compares experimental data with the prolate theory (Equations 2 and 3). The data were scattered, but the theoretical curve for perpendicular orientation fits the experimental data much better than in the case of parallel orientation. The square roots of the sum of the squares were 29.4 and 34.1 (N = 675) for the perpendicular and parallel orientations, respectively. The equation for the perpendicular orientation was simplified by substituting Equation 4 for 17:

$$\frac{d_{\text{ar}}}{W\sqrt{C(d_{\text{a}})}} = 2.22 \left(\frac{L}{W} \right)^{1/6} \quad (17)$$

TABLE III. Dimensions of SiC Whiskers

	Number	Diameter (CMW) (μm)	GSD	Length (CML) (μm)	GSD	Aspect Ratio (CMAR)	Correlation (τ)
Bulk	334	0.224	1.91	3.97	2.78	17.7	0.501
Aerosol	552	0.198	1.83	3.43	2.99	17.3	0.511

TABLE IV. Size Distribution of the Classified SiC Straight Fibers

Aerodynamic Diameter		
d_{ae} (μm)	Diameter (μm)	Length (μm)
1.90	0.59 ± 0.18	12.0 ± 8.9
1.28	0.40 ± 0.095	7.7 ± 6.6
1.20	0.38 ± 0.115	5.0 ± 3.8
1.07	0.36 ± 0.091	5.0 ± 4.4
0.87	0.28 ± 0.078	3.6 ± 3.5
0.73	0.22 ± 0.039	3.0 ± 2.4
0.65	0.19 ± 0.058	2.8 ± 2.6

Values are means \pm standard deviation.

Figure 6 shows that this equation also agreed well with the experimental data. If we use the aerodynamic equivalent diameter (d_{ac}), then Equation 17 becomes

$$\frac{d_{ac}}{W} = 2.22 \sqrt{\frac{C(d_n)}{C(d_{ac})}} \left(\frac{L}{W}\right)^{1/6} \quad (18)$$

The mean values of $[C(d_n)/C(d_{ac})]^{0.5}$ were calculated to be 1.06 and 1.01 ($N = 675$) for SiC fibers in perpendicular and parallel orientations, respectively.

Bent Fibers

Small portions of the SiC fibers were not straight rods but rather hooked rods as shown in Figure 7. From the classified fiber samples obtained in the LAPS, we measured the diameter-length angle between the two portions of the fiber and the percentage of the longest portion of the bent fibers. The results are

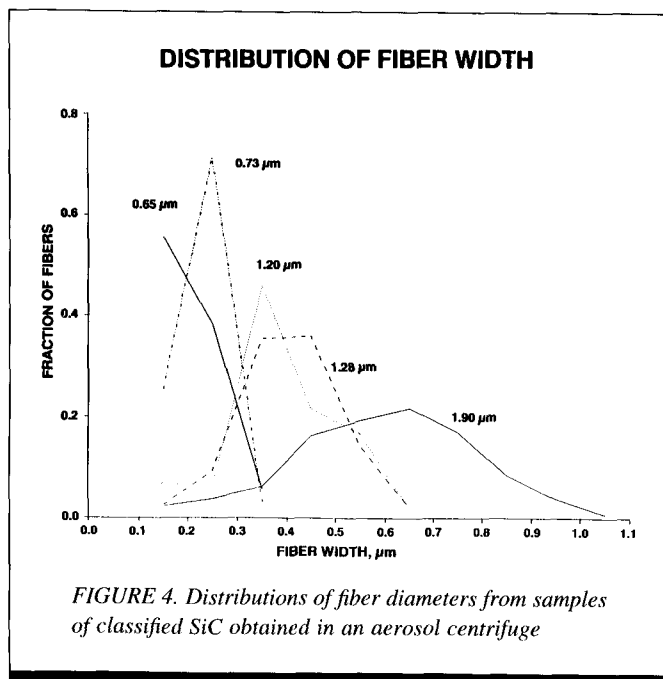


FIGURE 4. Distributions of fiber diameters from samples of classified SiC obtained in an aerosol centrifuge

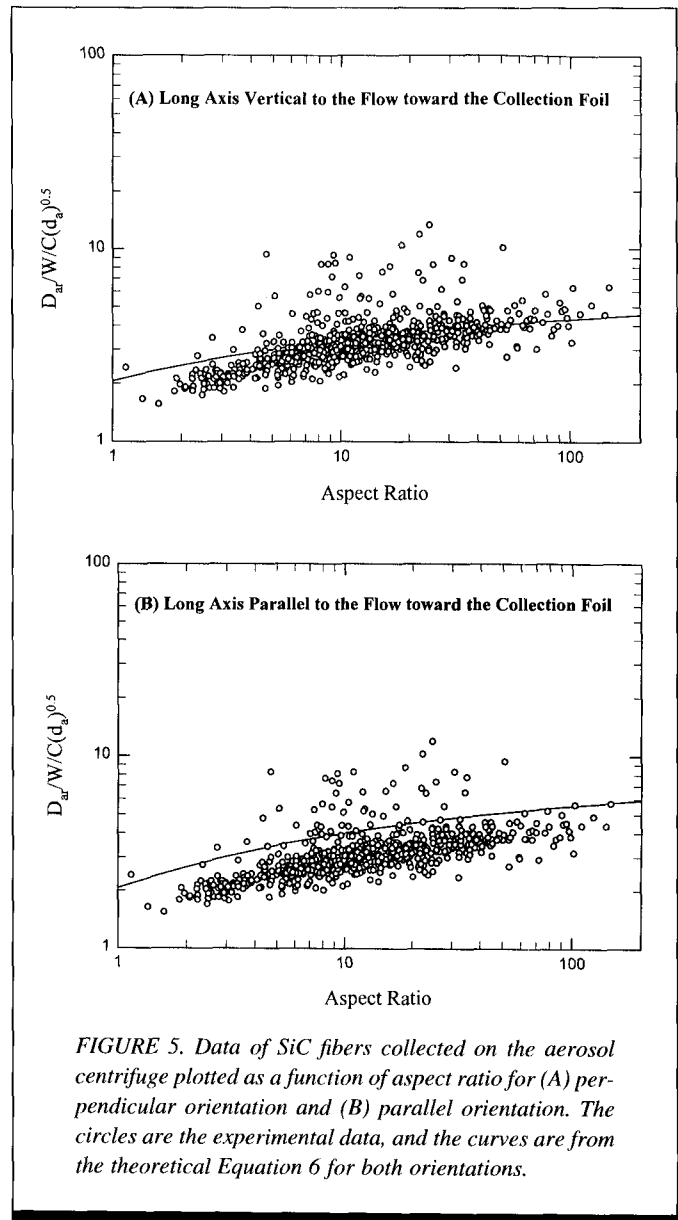
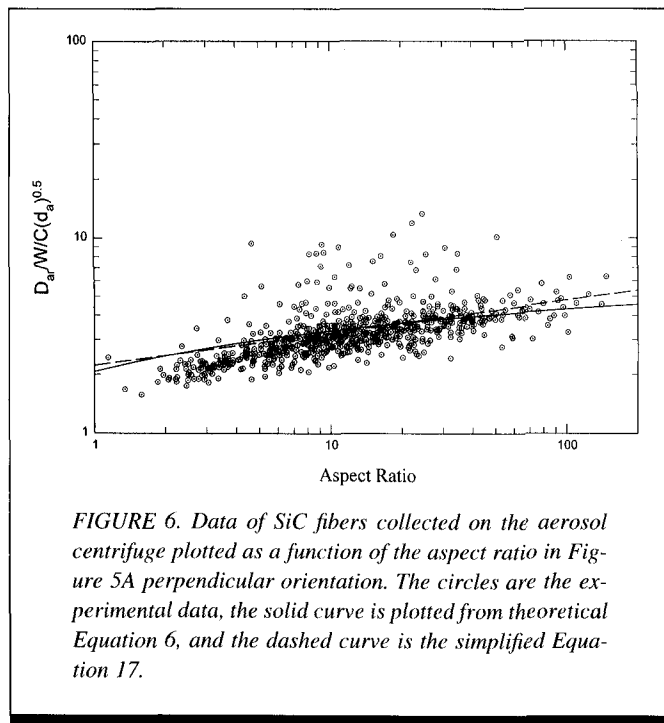


FIGURE 5. Data of SiC fibers collected on the aerosol centrifuge plotted as a function of aspect ratio for (A) perpendicular orientation and (B) parallel orientation. The circles are the experimental data, and the curves are from the theoretical Equation 6 for both orientations.

listed in Table V. The number of fibers in each size fraction ranged from 5 to 21. The overall mean angle was $78.9 \pm 24.1^\circ$ (mean \pm standard deviation), and the mean percentage of the longest portion was $67.6 \pm 11.5\%$ (mean \pm standard deviation) with $N = 86$. There were no clear trends of angle and longest portion dependence as a function of aerodynamic diameter.

Impactor Data

The aerodynamic equivalent diameter of the SiC fibers was in the respirable size range as determined by a cascade impactor. The MMd_{ac} was $2.80 \pm 0.17 \mu\text{m}$, and the geometric standard deviation was 2.00 ± 0.05 ($N = 3$). On the other hand, the MMd_{ac} were also calculated from the bivariate lognormal distribution of the SiC aerosols (Equations 11, 14, and 15). The measured MMd_{ac} was larger than the predicted values from the bivariate analysis (2.24 and $2.78 \mu\text{m}$ for perpendicular and parallel direction, respectively) but was closer to the value of $2.78 \mu\text{m}$.



The geometrical standard deviation from the bivariate analysis (Equation 15) was 1.85. The calculated MMd_{ac} used the mean values of 1.01 and 1.06 for $[C(d_n)/C(d_{ac})]^{0.5}$ in Equation 11 for

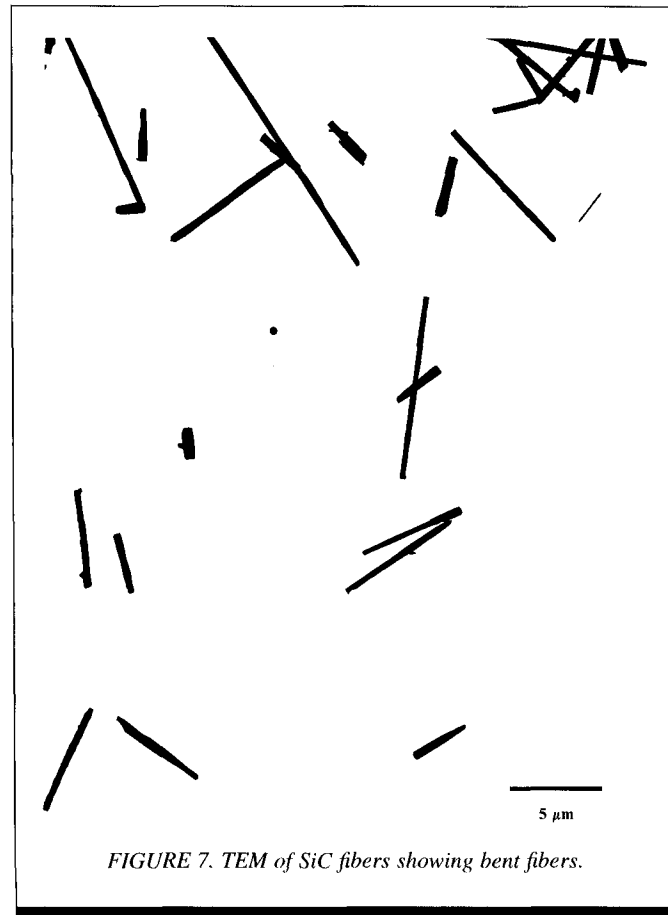


TABLE V. Size Distribution of the Classified SiC Bent Fibers

Aerodynamic Diameter d_{ae} (μm)	Diameter (μm)	Length (μm)
1.90	0.63 ± 0.11	14.6 ± 11.0
1.28	0.39 ± 0.102	5.6 ± 2.6
1.20	0.43 ± 0.092	5.6 ± 2.9
1.07	0.35 ± 0.040	4.5 ± 1.8
0.87	0.28 ± 0.065	5.7 ± 5.1
0.73	0.22 ± 0.027	3.4 ± 2.5
0.65	0.19 ± 0.057	2.2 ± 0.83

Values are means \pm standard deviation.

the perpendicular and parallel orientations. Figure 8 shows a histogram of size distributions obtained from the impactor and the bivariate analysis (parallel orientation).

In summary, the aerodynamic diameter (d_{ac}) of the SiC whiskers obtained from the impactor and aerosol centrifuge can be expressed as

$$d_{ac}(\text{centrifuge}) = 2.24W^{5/6}L^{1/6}, \quad (19)$$

$$d_{ac}(\text{impactor}) = 2.78W^{5/6}L^{1/6}. \quad (20)$$

These expressions are for fibers with their long axis perpendicular and parallel to the flow toward the collection plate, respectively.

Size-Selective Mass Fractions

Based on the aerodynamic size distribution obtained from the impactor, the inspirable, thoracic, and respirable mass fractions of the SiC aerosol were calculated (Table VI) following

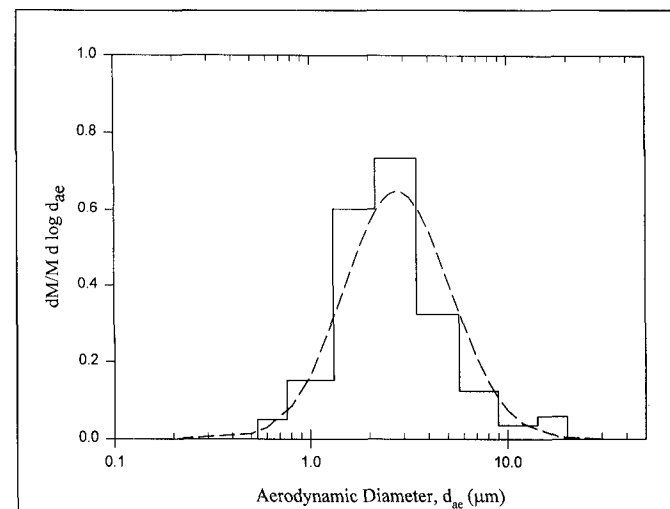


TABLE VI. Size-Selective Mass Fraction of SiC Aerosol

	<i>Inspirable Fraction (%)</i>	<i>Thoracic Fraction (%)</i>	<i>Respirable Fraction (%)</i>
Impactor data (2.80 μm , $\sigma_g = 2.0$)	90.9	88.0	65.3
Bivariate analysis (2.78 μm , $\sigma_g = 1.85$)	91.2	89.2	66.9
Bivariate analysis (2.24 μm , $\sigma_g = 1.85$)	92.7	91.7	76.0

the ACGIH⁽²⁹⁾ definition. These fractions were 91, 88, and 65%, respectively, using the impactor data. Over 65% of the SiC aerosol particles were respirable. The estimated size-selective mass fractions using aerodynamic diameters (parallel orientation) estimated from the bivariate analysis were within 3% of those from the impactor data. However, if the aerodynamic diameter in a perpendicular orientation was used to estimate the size-selective mass fraction, an overestimate of 16% for the respirable fraction would result. All measured physical properties are listed in Table VII.

DISCUSSION

SiC whiskers are fiberlike materials containing a small portion of bent fibers. SiC whiskers in all size classifications can be dispersed as aerosols; therefore, analysis of the bulk material should yield accurate information in terms of the physical, chemical, and biological characterization. However, SiC aerosols have smaller median widths and lengths, consistent with the notion of preferential losses of larger and longer fibers in the aerosol generation and transport processes. Both the bulk material and the aerosol form had a median aspect ratio of about 17.

A major issue addressed in this article is how to correlate the measured physical properties of SiC fibers to the aerodynamic behavior as determined from the aerosol sampling instruments. In industrial hygiene practices, filter samples are taken for number concentration and morphometry measurements of fiber aerosols. Cascade impactor and other sampling techniques have been used to determine the aerodynamic size distributions, which can then be used to estimate the respirability of the aerosol. Data from these two sampling techniques have rarely been analyzed simultaneously for consistency. The approach described here allowed us to predict the aerodynamic size distribution and size-selective fraction, including the respirable fraction from the bivariate analysis of SiC fiber dimensions obtained in filter samples. The predicted aerodynamic size distribution of the SiC aerosol agreed with the measured aerodynamic size distribution obtained in a cascade impactor. In addition, comparison of the impactor and predicted data showed that most SiC fibers in impactor substrates were collected with their long axis parallel to the flow toward the substrate. This result is consistent with the observations of Kasper and Shaw⁽¹²⁾ for elongated chain aggregates collected in a cascade impactor.

Because the SiC fibers were elongated, the flow resistance and therefore the aerodynamic diameter depend on the fiber width, aspect ratio, and particle density, as well as the orientation with respect to flow. A larger aerodynamic diameter (15–25%) and lower resistance would be expected for a fiber with its long

axis oriented parallel to the flow rather than in a perpendicular orientation. The flow resistance as expressed in the dynamic shape factor of regular cylindrical fibers was shown to agree with analytical equations, and a simplified form was obtained for prolate par-

ticles. Our data obtained from the aerosol centrifuge and impactor showed that these equations were also applicable for the SiC fibers.

The aerosol centrifuge provided additional information on the aerodynamic behavior of the SiC. The centrifuge was used to separate SiC fibers according to aerodynamic size, and samples for each aerodynamic size fraction were examined for physical dimensions. Our results showed that the estimated aerodynamic size from the aerosol centrifuge data was in agreement with the prolate theory with the long axis perpendicular to the flow toward the collection foil, consistent with data obtained for asbestos fibers⁽¹¹⁾ in a Stöber centrifuge.

Therefore, the aerodynamic size distribution of SiC fibers depends strongly on the instrument, especially the fiber orientation in the detection section of the instrument. For example, the SiC fibers have a parallel orientation in the impactor nozzle, and they have a perpendicular orientation toward the collection foil of the LAPS. Cascade impactor data result in a larger aerodynamic size distribution than aerosol centrifuge data. The differences in the aerodynamic size distribution, in turn, influence the estimate of respirable fractions for SiC fibers. In the absence of impactor or other aerodynamic size data, one can use the detailed width-length information from filter samples to estimate the aerodynamic size distribution following the bivariate analysis outlined in this article. The SiC data prove the validity of this approach. This approach provides calculated aerodynamic sizes for parallel, perpendicular, and random orientations. The values for parallel and perpendicular orientations can be used as a substitute for the impactor and aerosol centrifuge data, respectively.

One requirement of this approach is that the value of the particle density must be accurate to calculate the aerodynamic diameter in the bivariate analysis. Particle density can be measured from the bulk material. When material for measurements is insufficient, then the theoretical value of the particle density can be used if the chemical form of the fiber sample is positively identified. For samples of unknown material, the bivariate approach might yield large errors because of the uncertainty in particle density. In this

TABLE VII. Physical Characteristics of SiC Aerosol

<i>Property</i>	<i>Values</i>
Density (g/cm ³)	3.25
Surface area (m ² /g)	1.4 \pm 0.1
Mass Median Aerodynamic Diameter (μm) (GSD)	2.80 (2.00)
Inspirable fraction (%)	91
Thoracic fraction (%)	88
Respirable fraction (%)	65

case, we recommend using the aerosol sizing instruments, including the cascade impactor for aerodynamic size distribution.

ACKNOWLEDGMENTS

This research was conducted at the Inhalation Toxicology Research Institute and jointly funded by the Office of Health and Environmental Research, U.S. Department of Energy, under Contract No. DE-AC04-76EV01013 and NIOSH grant R01-OH 02922.

REFERENCES

1. **Chan, T.L., W. Kibbel, and J.B. D'Arcy:** Detection of fibers and aerosols in the manufacture of silicon carbide whisker-reinforced metal matrix composites at the R&D stage. *Appl. Occup. Environ. Hyg.* 8:889-895 (1993).
2. **Beaumont G.P.:** Reduction in airborne silicon carbide whiskers by process improvement. *Appl. Occup. Environ. Hyg.* 6:598-603 (1991).
3. **Begin, R., A. Dufresne, A. Cantin, S. Masse, P. Sebastien, and G. Perrault:** Carborundum pneumoconiosis. *Chest* 95:842-849 (1989).
4. **Vaughan, G.L., J. Jordan, and S. Kerron:** The toxicity, *in vitro*, of silicon carbide whiskers. *Environ. Res.* 56:57-67 (1991).
5. **Vaughan, G.L., S.A. Trently, and R.B. Wilson:** Pulmonary response, *in vivo*, of silicon carbide whiskers. *Environ. Res.* 63:191-201 (1993).
6. **Johnson, N.F., M.D. Hoover, D.G. Thomassen, Y.S. Cheng, A. Dalley, and A.L. Brooks:** *In vitro* activity of silicon carbide whiskers in comparison to other industrial fibers using four cell culture systems. *Am. J. Ind. Med.* 21:807-823 (1992).
7. **Stanton, M.F., M. Layard, A. Tegeris, M. Miller, M. May, and E. Kent:** Carcinogenicity of fiber glass: pleural response in the rat in relation to fiber dimension. *J. Natl. Cancer Inst.* 58:587-603 (1977).
8. **Bogoroch, R. and S.R. Luck:** Workplace handling requirements and procedures for acmc silicon carbide whiskers based on a subchronic inhalation study in rats. In: *Proceedings of the International Conference on Whisker and Fiber-toughened Ceramics*. Philadelphia, PA: ASTM International, 1988. pp. 81-89.
9. **Lapin, C.A., D.K. Craig, M.G. Valerio, J.B. McCandless, and R. Bogoroch:** A subchronic inhalation study in rats exposed to silicon carbide whiskers. *Fundam. Appl. Toxicol.* 16:128-146 (1991).
10. **Liu, B.Y.H., D.Y.H. Pui, X.Q. Wang, and C.W. Lewis:** Sampling of carbon fiber aerosol. *Aerosol Sci. Technol.* 2:499-511 (1983).
11. **Stöber, W., H. Flachsbarth, and D. Hochrainer:** The aerodynamic diameter of latex aggregates and asbestos fibres. *Staub-Reinhalt Luft* 30:1-12 (1970).
12. **Kasper, G. and D.T. Shaw:** Comparative size distribution measurements on chain aggregates. *Aerosol Sci. Technol.* 2:369-381 (1983).
13. **Cheng, Y.S.:** Bivariate lognormal distribution for characterizing asbestos fiber aerosols. *Aerosol Sci. Technol.* 5:359-368 (1986).
14. **Weast, R.C.:** *Handbook of Chemistry and Physics*, 69th ed. Boca Baton, FL: CRC Press, 1988.
15. **Newton, G.J., O.G. Raabe, and B.V. Mokler:** Cascade impactor design and performance. *J. Aerosol Sci.* 8:339-347 (1977).
16. **Kotrappa, P. and M.E. Light:** Design and performance of the Lovelace aerosol particle separator. *Rev. Sci. Instrum.* 43:1106-1112 (1972).
17. **Cheng, Y.S., H.C. Yeh, and M.D. Allen:** Dynamic shape factor of a plate-like particle. *Aerosol Sci. Technol.* 8:109-123 (1988).
18. **World Health Organization:** Reference methods for measuring man-made mineral fibres (MMMF). Copenhagen, Denmark: WHO, 1985.
19. **Heiss, J.F. and J. Coull:** The effect of orientation and shape on the settling velocity of non-isometric particles in a viscous medium. *Chem. Eng. Progress* 48:133-140 (1952).
20. **Youngren, G.K. and A. Acrivos:** Stokes flow past a particle of arbitrary shape: a numerical solution. *J. Fluid Mech.* 69:377-403 (1975).
21. **Kasper, G., T. Niida, and M. Yang:** Measurements of viscous drag on cylinders and chains of spheres with aspect ratio between 2 and 50. *J. Aerosol Sci.* 16:535-556 (1985).
22. **Gans, R.:** Wie fallen Stäbe und Scheiben in einer reibenden Flüssigkeit? Sitzungsber. *Math-physik Klasse Akad. Wissen Munchen* 41:198-203 (1911).
23. **Dahneke, B.E.:** Slip correction factors for nonspherical bodies. Iii. The form of the general law. *J. Aerosol Sci.* 4:163-170 (1973).
24. **Chen, B.T., R. Irwin, Y.S. Cheng, M.D. Hoover, and H.C. Yeh:** Aerodynamic behavior of fiber- and disc-like particles in a millikan cell apparatus. *J. Aerosol Sci.* 24:181-195 (1993).
25. **Allen, M.D. and O.G. Raabe:** Slip correction measurements of spherical solid aerosol particles in an improved millikan apparatus. *Aerosol Sci. Technol.* 4:269-286 (1985).
26. **Cheng, Y.S., M.D. Allen, D.P. Gallegos, H.C. Yeh, and K. Peterson:** Drag force and slip correction of aggregate aerosols. *Aerosol Sci. Technol.* 8:199-214 (1988).
27. **Hatch, T. and S.P. Choate:** Statistical description of the size properties of non-uniform particulate substances. *J. Franklin Inst.* 207:369 (1929).
28. **Soderholm, S.C.:** Proposed international conventions for particle size-selective sampling. *Ann. Occup. Hyg.* 33:301-320 (1989).
29. **American Conference of Governmental Industrial Hygienists:** *1992-1993 Threshold Limit Values for Chemical Substances and Physical Agents and Biological Exposure Indices*. Cincinnati, OH: ACGIH, 1992.