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Fiber Classification and the Influence of Average Air Humidity

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The use of man-made vitreous fibers (MMVFs) as a substitute for asbestos in industrial and residential applications has raised the concerns of the potential hazards associated with inhalable aerosolized fibers. The complex movement of fiber makes it difficult to predict the pattern of fiber deposition in human airways from the behavior of spherical particles. Difficulties in producing monodisperse length fibers has been an obstacle to study fibrous particle deposition in the human respiratory system. To address this problem, a narrow length distribution of fibers was generated using dielectrophoretic classification. Dielectrophoresis is the motion of neutral matter in a nonuniform electric field due to an induced dipole moment. It is sensitive to the conductivity of the matter in the field. A fiber classifier has been used to study the influence of atmospheric humidity on the behavior of glass fibers. Glass fibers, as insulators, can not be classified by the dielectrophoretic classifier. However, our study shows that a humidity higher than 15% RH can change the conductivity of the glass fibers so as to permit their effective classification.

INTRODUCTION

The study of fibrous aerosols primarily began with health and manufacturing concerns regarding asbestos (Baron 2001). "Asbestos" refers to a group of naturally occurring fibrous metallic silicates that have been widely used in construction and industry. However, asbestos inhalation has been associated with pulmonary fibrosis and cancer. The U.S. EPA (2001) has classified asbestos as a Group A, human carcinogen, based primarily on inhalation and epidemiological studies, and has calculated an inhalation unit risk estimate of 0.23 per fiber/cm^3. Thus the use of asbestos fibers was banned in 1989. This restriction led to the wide use of man-made vitreous fibers (MMVF) as substitutes for asbestos although some of the ban was lifted in 1991 (http://www.epa.gov/asbestos/inforev.pdf).

MMVFs are inorganic fibrous materials with amorphous molecular structure obtained from several kinds of minerals. The World Health Organization classified the following as MMVFs: insulation wools (glass, rock, and slag), refractory ceramic fibers and special fibers in relation to diameter and method of production. The MMVFs are structurally similar to asbestos fibers and this similarity raised the concern over their potentials of MMVFs to induce pulmonary diseases (Brown 1994). Epidemiological studies have found high risk in groups of workers exposed to MMVFs (Marsh et al. 2001; LeMasters et al. 2003). A study using human mesothelial cell line also suggested possible cytotoxic, oxidative, and genotoxic effects for refractory ceramic fibers and rock wool (Cavallo et al. 2004).

In 2001, the U.S. EPA classified glass wool, continuous filament glass, rock wool and slag wool as "No cancer assessments on IRIS (Integrated Risk Information System)" and confirmed refractory ceramic fibers in the Group B2 as agents that are probable human carcinogen (EPA 2001). The American Conference of Governmental Industrial Hygienists (ACGIH), National Institute for Occupational Safety and Health (NIOSH) and Occupational Safety and Health Administration (OSHA) have occupational recommendations and regulations that apply to MMVF exposure. The most recently recommended acceptable exposure levels were adopted by ACGIH in 2001 (ACGIH 2001). The designation for MMVFs classifies glass wool, rock wool, and slag wool as A3 (confirmed animal carcinogen with unknown relevance to humans) with a recommended TLV (Threshold Limit Value) in the workplace of 1 fiber/cm^3 and refractory ceramic fibers as A2 (suspected human carcinogen) with a TLV of 0.2 fiber/cm^3.

Inhalation is the major route of exposure to mineral fibers that have been shown to cause lung disease in humans (Selikoff and Lee 1978). Fibers longer than 5 μm and less than 3 μm in

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diameter with aspect ratio (fiber length/fiber diameter) >3 are defined as respirable fibers because of their capability to reach the target areas of the lung and pleura.

Studies on the toxicity of fibers indicate that fiber dose, fiber dimension, chemical composition and fiber durability in lung fluid are the primary factors determining fiber toxicity (Lippmann 1990; Lippmann 1993; Hill et al. 1995; Fubini 1996). The deposition density of airborne fibers on a region in the respiratory tract is clearly an important factor in determining the likelihood of disease. The results of in vitro and inhalation studies suggest that longer and thinner fibers have great toxicity (Everitt 1994; Blake et al. 1998). Fibers longer than 20 μm are thought to be more toxic because they are longer than the macrophage diameter (about 17 μm) and can damage and kill macrophages (Baron et al. 2001). In addition, the iron content of the fibers may contribute to their carcinogenic effect since Fe ions catalyze the production of reactive oxygen species with consequent induction of oxidative DNA damage and cellular transformation (Fubini 1996). For both nonfibrous and fibrous particles, solubility in the lung is also an important factor. Although particles that dissolve rapidly in vivo may have an immediate cytotoxic effect upon cells of the lung they contact, they are unlikely to induce long-term pathological changes (Morgan and Holmes, 1986). Knowledge of the fiber deposition pattern in human airways is critical for defining a thoracic fiber fraction as an index of exposure to fiber aerosols.

The respiratory deposition of spherical particles has been widely studied in human subjects and physical models and both theoretical and experimental results are in good agreement (Heyder et al. 1986; Hofmann and Koblinger, 1992; Swift et al. 1992; Cheng et al. 1993; Cheng et al. 1999; Hofmann et al. 2002; Montoya et al. 2004). Beeckmans (1972) showed that the deposition result for spherical particles could not be used to estimate the deposition of fibrous particles by means of an equivalent spherical particle diameter because this diameter is different for different mechanisms. Burke and Esmen (1978) found that if the aerodynamic diameter of glass fiber developed by sedimentation experiments was used to calculate Stokes number, the impaction would be underestimated.

A study of fibrous particle deposition in rodent lungs was performed by Yu et al. (1991). However, the qualitative and quantitative aspects of particle deposition and retention in rodents are considerably different from those in humans (IARC, V. 43, 1988). No human data are available from controlled experiments of inhaled fiber aerosols for the obvious reason that most fibrous materials are potentially hazardous when inhaled. There have been some experimental studies of fiber deposition in human physical models of TB region (Myojo 1987, 1990; Sussman et al. 1991 Myojo and Takaya, 2001) and of nasal airway (Gradon and Podgorske, 1992). Fibrous material used in these studies were different from each other. However, the aerodynamic diameters of these fibers were not less than 1.5 μm and thus the particle Brownian motion was not significant during deposition. These studies indicated that the deposition efficiencies increased as both the Stokes number for randomly oriented fibers and the fiber length increased. The deposition "hot spots" were located at the bifurcation carinal ridges of the lung models and nasal turbinate region of nasal airway model. Theoretical models of fiber deposition in human lung have also been developed (Harris and Fraser, 1976; Asgharian and Yu, 1988; Asgharian and Anjilivel, 1995). These models take account of the complex behavior of long fibrous particles that are subject to a combination of translation and rotation. Interception mechanisms besides diffusion, sedimentation, and impaction were included to derive the total deposition efficiencies. However, the interaction between long fibrous particles and airway walls should also be considered because long fibers have more surface area compared with mass equivalent spherical particles. More experimental data are necessary to improve and verify theoretical models.

To perform additional studies using monodisperse length fibers and defined realistic physical models, a dielectrophoretic fiber classifier can be used to generate classified glass fibers. Lipowiz and Yeh (1989) showed that dielectrophoresis can be used to separate conductive, neutral prolate spheroids in an electric field gradient. Based on these calculations, Baron and colleagues (Baron et al. 1994; Deye et al. 1999) developed a fiber length classifier that worked well for conductive fibrous particles. The classifier configuration consisted of two concentric tubular electrodes with a high voltage AC electric field applied at the surface of the inner electrode. Conductive fibers placed in the gradient electric field were attracted to the inner electrode with a velocity approximately proportional to the length squared. Thus, longer fibers deposited on the inner electrode more rapidly than shorter ones. Fibers that were drawn to the inner electrode but were not deposited, were removed in the classified flow at the bottom of the classifier as product.

Theory suggests that nonconductive fibrous particles cannot be length classified and will have a very low drift velocity compared to conductive fibers. These results imply that the fiber classifier should not provide a separation of non-conductive fibers. However, it was found that a humidified environment (Baron et al. 1994; Deye et al. 1999; Han et al. 1994) resulted in the classification of non-conductive fibers through the adsorption of water onto the fibers. Although fiber length classification had been achieved, the relationship between the humidity and the classification efficiency was not well defined. From the previous work (Baron et al. 1994; Deye et al. 1999), it is expected that there should be a humidity value at which nonconductive fibrous particles, such as glass fibers, exhibit conductive behavior. In this paper, we present our experimental studies of the influence of humidity on fiber length classification. We find that glass fibers take up water at increasing humidity and glass fibers become classifiable after the humidity increases above 15%.

EXPERIMENTAL DESCRIPTION

Figure 1 schematically presents the experiment setup to study the influence of humidity on glass fiber classification at Clarkson

TABLE 1
JM-475 Glass chemical composition (wt%)

SiO_2	$A1_2O_3$	Fe_2O_3	MgO	CaO	Na_2O	K_2O	B_2O_3	F	BaO	ZrO_2	ZnO
58.4	5.95	0.05	0.18	1.77	9.6	3.03	11.04	0.56	4.88	0.03	4.88

University. The setup consisted of an aerosol generation system, a classifier, a humidifier, a flow distribution system, and a measurement section.

JM-100 glass fibers were used in the study which were made from JM-475 glass (Johns Manville Corporation). The average diameter of the fiber was approximately 1 μm. The density of the glass was 2.56 g/cm^3 and its chemical composition is listed in Table 1. Bulk JM-100 glass fibers were milled and then sieved to remove fiber clumps and mixed with soda-lime glass beads (nominally 70 μm diameter, Potters Industries Inc.). The mixture of glass beads and glass fibers was first stored in a hopper and then metered through a rotary feeder into the fiber aerosol generation system whose main part was modified from a high speed orbital shaker bed (Baron et al. 2002).

The airstream, Al, with a controlled humidity passed through the fiber generator and transported the polydisperse glass fibers to the aging chamber that was 90 cm long and 9 cm in diameter. The chamber provided more contact time between the glass fibers and humidified air (approximately 5 min). The flow rate of Al was normally set higher than the aerosol feed flow to the classifier. The excess aerosol was vented through filter Fl (HEPA-CAP 75, Whatman). An EXTECH Hygro-Thermometer (EXTECH Instrument) was used to measure the humidity of the system. The accuracy of the meter is $\pm$ 3% RH (relative humidity) for a range of 10–90% RH.

For each controlled humidity experiment, the airstream without fibers was circulated for about 1 hour for the humidity to approach equilibrium before sampling. Bypass filter F2 (HEPA-CAP 75, Whatman) before the neutralizer was used to

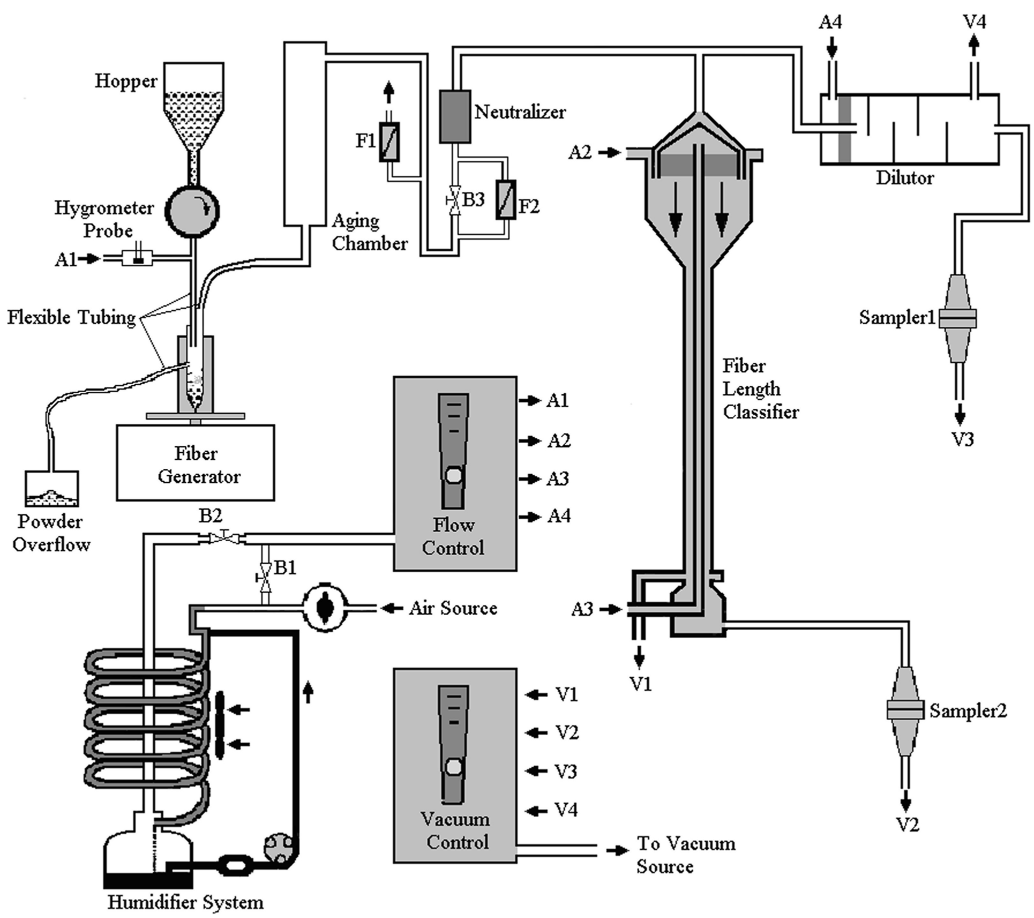

FIG. 1. Schematic of fiber classification system.

clear the aerosol flow of fibers during this circulation. Po210 ionizing units (Staticmaster) were used in the neutralizer to remove the static charges carried by the glass fibers. A copper coil air humidifier controlled the humidity of the medium air. The highest humidity achieved was around 75% RH within the system. Higher humidity than this value might cause voltage breakdown in the classifier. The humidity range for this study was chosen to be between 0% RH and 75% RH. A desiccant drier unit was used to achieve low humidity. House compressed air and vacuum were used to provide sufficient flow.

The glass fiber aerosol from the generator was split into two identical streams; one for fiber classifier and the other for an 11:1 dilutor. Both streams were conducted to a particle counter or a filter sampler. The experiments were conducted in two ways. In the first approach, LAS-X CRT counters (Particle Measuring Systems, Inc.) were used to count the number of fibers. Scattered light from particles larger than the wavelength of the laser consisted of three components: diffraction, reflection, and refraction (Gebhart, 2001). The size given by the particle counter should reflect the size, shape and orientation of the fiber. Here this size is called the "optical size." Fibers with an optical size over 0.5 μm were counted by the counter.

In the second method, Millipore AA type filter membranes were used to collect integrated fiber samples. The filter holders were made of stainless steel. An acetone vaporizer was used to make the filters transparent. The fiber number was counted using a phase contrast microscope following NIOSH method 7400 (Baron, 1994). For the aerosol from the generator, only fibers within a classified length range were counted. The classification efficiencies for different humidity values were calculated. In order to investigate the fiber length effect in the onset of classification, two sets of experiments were performed at 3 kV and 4 kV. For both methods, the efficiencies were calculated as the counts from sampling position 2 (sampler 2 in Figure 1) divided by counts from sampling position 1 (sampler 1 in Figure 1).

In order to study the adsorption of water vapor, a block of glass fibers was used to measure the weight gain upon exposure to different humidities. The fiber block was exposed to a air stream of given humidity for 6–12 h. The balance applied was a METTLER type H6T. Figure 2 schematically shows the experimental setup of this part.

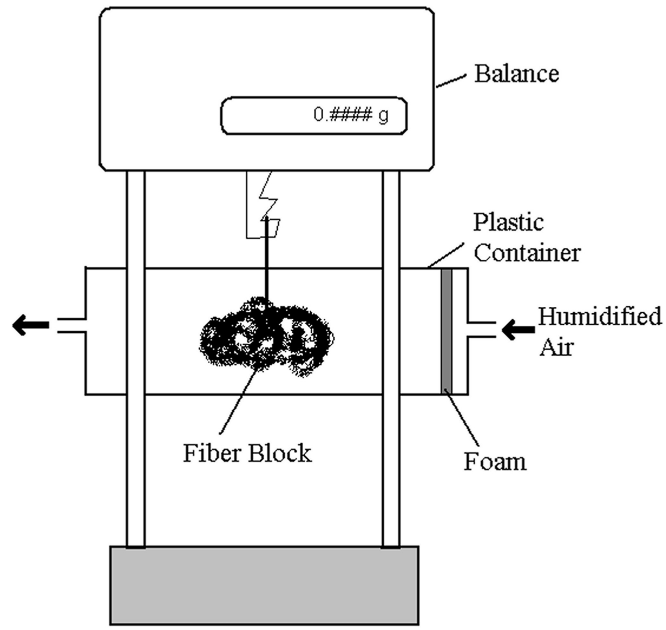

FIG. 2. Schematic of water adsorption experiment.

Carbon fibers were also used to show the influence of humidity on conductive fiber classification. The experiments were performed at Lovelace Respiratory Research Institute in New Mexico where an identical fiber classifier was used. The carbon fibers were provided by Hercules Inc. in Wilmington, DE with a uniform diameter of 3.66 μm and density of 1.83 g/cm^3.

Water extraction experiments were performed to evaluate the solubilities of glass fibers and glass beads used in the present study. Glass fiber blocks or glass beads were leached in pure water in closed sample bottles at 20°C for 5 min. An OMEGA PHH-20 pH meter was used to measure the pH of the resulting solution and a YSI Model 32 conductance meter (Scientific Division Yellow Springs Instrument Co., Inc.) was applied for conductance measurements. The pH and conductance of pure water used were also measured for reference.

TABLE 2
Glass fiber classification under different conditions

Fiber length (μm)	Measured length range (μm)	Applied voltage (kV)	Inner/Outer sheath flow (LPM)	Fiber aerosol flow rate (LPM)	Sheath flow relative humidity (%)
5	4–6[a]	6.56	4.0/4.0	0.5	75
10	9–12[b]	4.00	4.0/4.0	0.5	75
30	25–33[c]	1.52	4.0/4.0	0.5	75

[a]Count average length: 5.1 μm, standard deviation: 0.7 μm, coefficient of variation: 13.7%.
[b]Count average length: 10.1 μm, standard deviation: 1.2 μm, coefficient of variation: 11.9%.
[c]Count average length: 28.8 μm, standard deviation: 2.9 μm, coefficient of variation: 10.1%.

TABLE 3
Carbon fiber classification under different conditions

Fiber length (μm)	Measured length range (μm)	Applied voltage (kV)	Inner/Outer sheath flow (LPM)	Fiber aerosol flow (LPM)	Sheath flow relative humidity (%)
155	150–160[a]	0.25	2.0/2.0	1.25	<5
70	65–75[b]	0.50	2.0/2.0	1.25	<5

[a]Count average length: 153.5 μm, standard deviation: 20.8 μm, coefficient of variation: 13.6%.

[b]Count average length: 71.6 μm, standard deviation: 13.5 μm, coefficient of variation: 18.9%.

RESULTS AND DISCUSSION

The fiber classification system was first examined with glass fibers by introducing a humidified air flow (around 75% RH) into the system as the carrier gas and sheath flows as described by Baron et al. (1994). Using humidified air proved to be an effective way to improve the conductivity of nonconductive fibers, such as asbestos and glass fibers. Table 2 lists the conditions for glass fiber classification. For conductive fibers, such as carbon fibers, the medium air does not need to be humidified. Table 3 shows the conditions for carbon fiber classification at Lovelace Respiratory Research Institute in New Mexico. These data show that the classification system can generate fiber aerosol with narrow length distribution. However, the size distributions in Table 3 are slightly wider than those in Table 2. This difference is in agreement with the previous results that a higher ratio of aerosol flow to sheath flow causes wider size distributions (Deye et al. 1999).

For the experiments investigating the influence of humidity, glass fibers were used. Voltages of 3 kV and 4 kV AC at 50 Hz were applied to the inner electrode of the classifier. Figure 3 shows the experimental results. The trend of particle counter data agreed well with that from the filter samples. When the relative humidity reached around 15%, the classification efficiency substantially increased. The results indicate that these fibers show sufficient conductivity for dielectric classification above 15% RH. The classification length has no significant influence in the onset of classification. These results generally agreed with the results of Lilienfeld (1985). He argued that highly resistive glass fibers showed conductive behavior whenever the ambient relative humidity to which these fibers were exposed exceeded 30%. For carbon fibers, several humidities ranging from 0% RH to 50% RH were applied during fiber classification. As expected, no significant change of classification percentage was observed since they are conducting regardless of any added water.

The weight gain experiments showed that the block did not gain additional weight after 6 hours exposure to an air stream with fixed humidity. The results (Figure 4) show a linear relationship between weight gain and the air humidity. If we assume that the adsorbed water molecules distribute evenly on the surface of the fiber whose diameter is a uniform 1 μm, the weight gain for 15% RH corresponds approximately to a thickness of 5.9 Å. The mean Van Der Waals diameter of water molecule is 2.82 Å (Franks, 2000) and a film consisting of two layers of molecules can be expected at 15% RH. Considering the compact pattern of the fiber block, the actual weight gain of equivalent amount of fibrous particles should be higher.

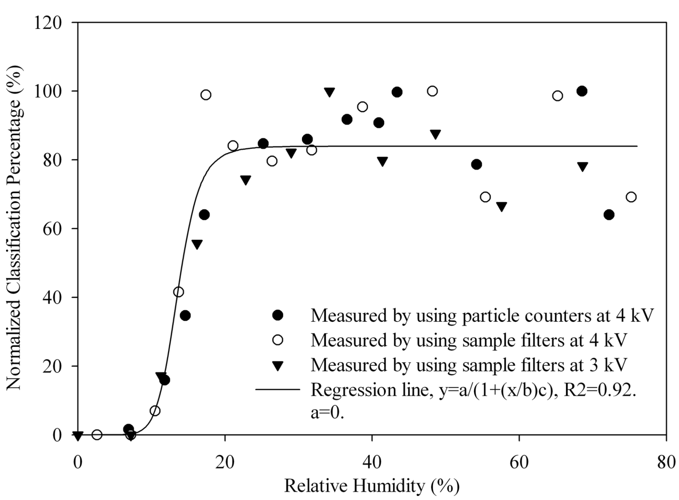

FIG. 3. Humidity of medium air vs classification percentage.

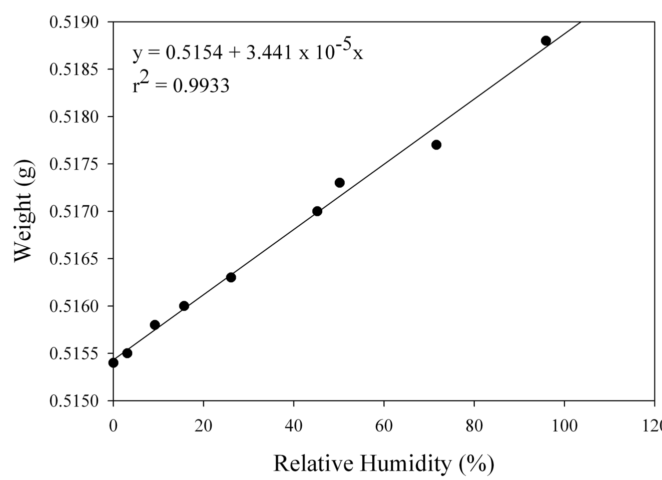

FIG. 4. The weight of a glass fiber block under different humidity of medium air.

TABLE 4
Water extraction analysis

	Water extraction of glass fiber block			Water extraction of glass beads beads			Water	
	pH	Conductance (μS/cm)	S/V, $\times 10^4$ (m^{-1})	pH	Conductance (μS/cm)	S/V, $\times 10^4 (m^{-1})$	pH	Conductance (μS/cm)
1	8.93	35.9	1.40	9.42	85.6	1.27	6.71	0.581
2	8.77	22.2	0.42	9.16	31.4	0.50	6.97	0.500

Our experimental results clearly indicate that the resulting conductivity of the glass fibers is not only caused by dielectric polarization of fibers. Fuchs (1964) used an inhomogeneous electric field to determine the apparent density of uncharged particles and found that mineral oil droplets showed conductive properties equivalent to mercury droplets. He argued that ionizing contaminants contained in the oil droplets made them conductive and proposed that the particles could be considered as conductors in an electric field. Lilienfeld (1985) postulated that surface adsorption of water caused the increase in conductivity. His experiments showed that glass fibers (diameter 2 μm, length 10–20 μm) behaved like conductors whenever the ambient relative humidity to which these fibers were exposed exceeded 30% in a AC field. This conductivity change of fibers that have low bulk conductivity is partially due to the microscopic size of the particle (Baron et al. 1994). We think the adsorption of water molecules accompanied by the dissolution of ions present on the surface or leached from the inside of the particles contributed to the change in conductivity.

Grant and Salthouse (1977) measured the surface resistance during dew formation on a piece of glass (SiO2: 71.5%; Na2O: 12.5%; CaO: 8.1%) that was cleaned before the experiments. They found that the surface resistance decreased steadily with decreasing temperature until it was possible to detect a dew deposit on the surface. Their work confirmed the existence of an adsorbed water film, the thickness of which remained constant once nucleation started. The volume conductivity of the film was about 0.01 S/m according to their work. For silica glass, the volume conductivity is $3 \sim 25 \times 10^{-9}$ S/m at 350°C (Lide, 1994). The higher conductivity required at least 10^{-15} gm/mm^2 of ions on the glass surface. Based on the elevation of the observed dew point above the actual dew point (Raoult effect), the estimate of the total amount of soluble material present on the surface should be around 10^{-10} gm/mm^2, much greater than the number contributing to conduction. Grant and Salthouse (1977) changed humidities by cooling while the present study added water to the system. Assuming a similar volume conductivity of the film on the surface of glass fibrous particle, the effect of this thin film on the conductivity change will be estimated.

In order to demonstrate the influence of the adsorbed film, the following discussion is based on a spherical glass particle covered with a thin film in a spatially uniform AC electric field. Glass is normally classified as an insulator with a dielectric constant ranged from 3.8 to 9.5 at 100 MHz and 20°C (Lide, 1994). The dielectric constant of glass does not vary strongly with frequency with less than a 5% difference as the frequency varied from 500 Hz to 1 MHz (Morey 1938). For an insulating particle with a very thin film of adsorbed water which has sufficiently high conductivity compared with the core, the principal mechanism of ion transport involves motion tangential to the particle surface. According to Jones (1995, 2003), if the thickness of the film $\delta \ll d$, the diameter of the particle, the effective complex permittivity of an equivalent, homogeneous spherical particle will be:

$$\varepsilon_p = \varepsilon_p + \frac{4\varepsilon_f}{d}$$
$$\varepsilon_f = \varepsilon_f + \frac{\sigma_f}{j\omega}$$

where ε_p is the complex permittivity of the insulating particle; ε_f and σ_f are surface permittivity and surface ohmic conductivity of the film respectively; $j = \sqrt{-1}$ and ω is radian frequency of the electric field. Calculation shows that a 10 Å adsorbed film will increase the relative permittivity of a 1 μm diameter glass bead to 15,000 for a 50 Hz AC field. The conductive shell does substantially change the electrical properties of the insulating particle. Masuda and Itoh's (1989) experimental research on the electro statical alignment of $A1_2O_3$ short fibers in freon showed that the orientation of these fibers was strongly influenced by surface conductivity.

The discussion so far has focused on the basis of the existence of the surface electrolyte. However, the sources and quantity of the ions are not clear. Table 4 lists the results of water extraction experiments. S/V is the ratio of glass surface to water volume. It is shown that the conductance of the solution increased significantly while the pH values also increased after extraction from the glass surface. The increasing pH value implies that leaching from the glass surface might be the dominant process for conductance change. Glass beads (soda-lime glass) have a higher leaching rate at a similar value of S/V compared with glass fiber blocks. It is most likely that soluble materials at the solid-water -adsorbed interface, such as sodium and boron, can be readily dissolved and thus, a conductive surface layer is formed.

CONCLUSION

The dielectrophoretic classifier was effective in separating conductive fibers into narrow length distributions. For non-conductive fibers, such as glass fibers, humidification can be used to increase the overall conductivity of the fibers and make classification possible. Humidification of greater than 15% RH provides sufficient conductivity for glass fiber classification. Leached soluble materials from the glass surface might be the main source of ions. Multilayer adsorption of water molecules appears to form a conductive surface layer to mobilize the ions. This thin conductive surface film improves the overall conductivities of micrometer sized fibers.

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