APPLICATION OF MONODISPERSE FIBERS AND DISCS TO EVALUATION OF THE AERODYNAMIC PARTICLE SIZER

PRINCIPAL INVESTIGATORS
M. D. Hoover
*P. J. Lipowicz**
*R. W. Hanson***
H. C. Yeh
*S. A. Casalnuovo****

Abstract — Monodisperse fibers, 1 µm in width and lengths of 5, 10, 20, and 40 µm, as well as monodisperse discs, 2, 4, 8, or 12 µm in diameter, were prepared using an integrated circuit microchip fabrication technique. Particles were silicon dioxide with a thickness of 1 µm. Examination of the particles using a scanning electron microscope showed that they were uniform in shape, with well-defined edges. The particles were suspended in distilled water and aerosolized with a Lovelace nebulizer. The monodisperse particles were used to evaluate the TSI Aerodynamic Particle Sizer (APS). Carbon fibers that were monodisperse in diameter (count median diameter = 3.42 µm, geometric standard deviation = 1.06) and polydisperse in length (count median length = 28 µm, geometric standard deviation = 2.2) were also used. The APS was found to be insensitive to fiber length and only weakly sensitive to disc diameter.

The availability of monodisperse samples of latex spheres has enabled the precise calibration of instruments to classify spherical particles by their aerodynamic, optical, or electrostatic behavior. One of the challenges of aerosol science has been to determine how instruments calibrated with spherical particles can be used to measure nonspherical particles. The lack of monodisperse particles having other shapes, such as fibers or discs, has limited progress in understanding the response of instruments to nonspherical particles. The development of a new method for producing monodisperse particles by integrated circuit fabrication techniques (1986-87 Annual Report, LMF-120, pp. 65-67) demonstrated a viable way to make nonspherical monodisperse particles for aerosol science studies. In the current work, we used the new technique to prepare monodisperse fibers and discs, generated aerosols of the new particles by nebulization, and applied the particles for evaluating the TSI Aerodynamic Particle Sizer.

MATERIALS AND METHODS

Standard silicon wafer substrates were coated with sequential layers of aluminum, silicon dioxide, and a photo-resistant polymer (photoresist). Photolithographic masks containing repetitive patterns of rectangles or circles were used to expose the photoresist layer. Approximately 10^8 particles could be defined on each wafer. The exposed photoresist was removed in a developing process and a plasma etch process was used to cut through the uncovered silicon dioxide, which created regions of defined rectangular or circular shape. The thickness of the particles was equal to that of the silicon dioxide layer (1 µm). Fibers 1 µm in width and 5, 10, 20, and 40 µm in length, as well as discs 2, 4, 8, or 12 µm in diameter were fabricated. These operations were performed in the Integrated Circuit Prototype Laboratory of the Center for Radiation Hardened Microelectronics at Sandia National Laboratories.

*AWU Post-Doctoral Participant
**AWU Summer Student
***Sandia National Laboratories

The wafers were taken to ITRI where the monodisperse fibers or discs were released into a liquid suspension by immersing the wafer for 24 h in a bath of concentrated hydrochloric acid (Ultrex grade) at room temperature to dissolve the aluminum sublayer. The suspensions were repeatedly diluted with distilled water and concentrated by centrifugation to remove the dissolved aluminum and acid. The suspensions were aerosolized with a Lovelace nebulizer operated at 20 psi (138 kPa), with an airflow of 1.5 L/min. The droplets were evaporated by dilution with 5 L/min of filtered, dry air. The particles were examined with a scanning electron microscope before and after resuspension to determine the uniformity of their shape and size, and to see if the nebulization process caused any particle breakage.

A TSI Aerodynamic Particle Sizer (APS) (TSI Incorporated, St. Paul, MN) was selected for evaluation because it provides a rapid, real-time, measurement of particle aerodynamic diameter by measuring the velocity of particles in an accelerating jet of air. We calibrated the APS using monodisperse latex particles and obtained a calibration curve identical to that obtained by Chen et al.[1] for the atmospheric pressure in our laboratory (0.84 kPa). Aerosols of the monodisperse fibers and discs were sampled by the APS and the number of particles detected per unit time was used to calculate generation efficiencies. The APS channel responses for the particles were noted and converted to aerodynamic diameter. Theoretical drag expressions for perpendicular and parallel orientations of fibers[2] and discs[3] were used to evaluate the sensitivity of the APS to the shape and size of the nonspherical particles. The density of the silicon dioxide particles was assumed to be that of amorphous silica (2.2 g/cm^3). Carbon fibers that were nearly monodisperse in diameter (count median diameter = 3.42 μm, geometric standard deviation = 1.06) and polydisperse in length (count median length = 28 μm, geometric standard deviation = 2.2) were also sampled to further evaluate the sensitivity of the APS to fiber diameter and length.

RESULTS

Figure 1 illustrates the appearance of the monodisperse fibers and discs before they were removed from the wafer substrates. They were uniform in shape, with well-defined edges. All

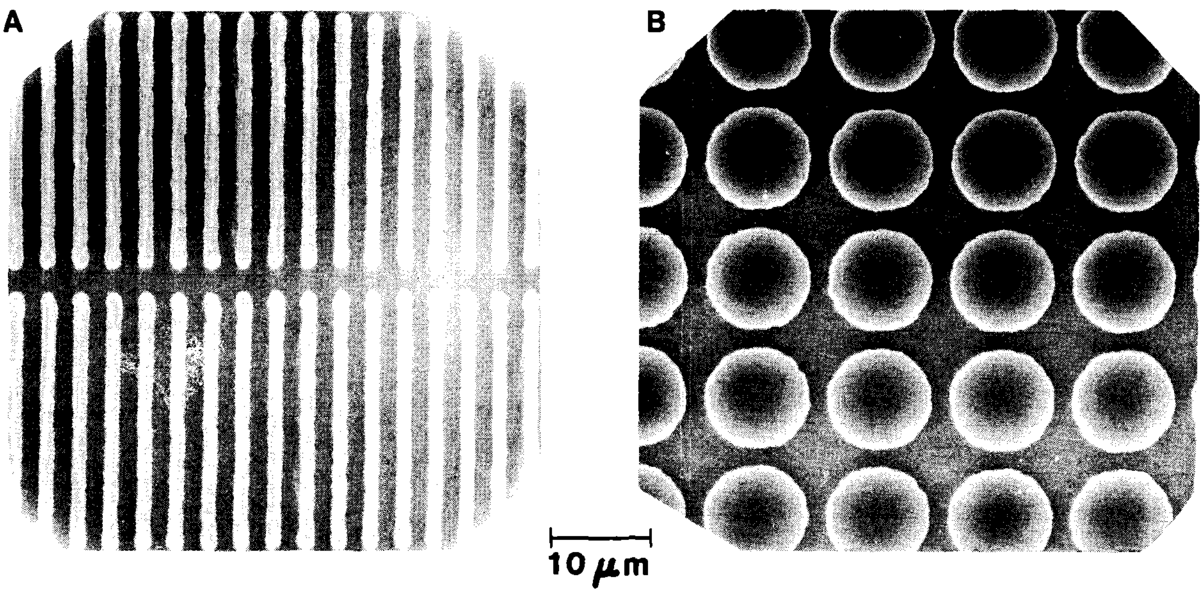

Figure 1. Representative scanning electron photomicrographs of (A) the 1 x 1 x 20 μm monodisperse fibers and (B) the 1 x 8 μm monodisperse discs, before their removal from the wafer substrates.

particles on a wafer could be suspended by the acid bath. Comparison of the volume of suspension aerosolized by the Lovelace nebulizer per unit time to the airborne particle concentration observed by the APS indicated a generation efficiency of about 3% for the 2-μm discs, dropping to 0.5% for the 12-μm discs, and a generation efficiency of about 1% for all fiber lengths. Airborne particle concentrations of 10^2 to 10^4/L were obtained. No evidence of particle breakage was noted. Figure 2 compares the APS channel location and distribution for the monodisperse latex calibration spheres, the monodisperse fibers and discs, and the carbon fibers. The shape of the response peaks for the larger discs appeared choppier because there were fewer particle counts. Figure 3 compares the aerodynamic diameters measured by the APS to the theoretically predicted aerodynamic diameters for the monodisperse fibers and discs.

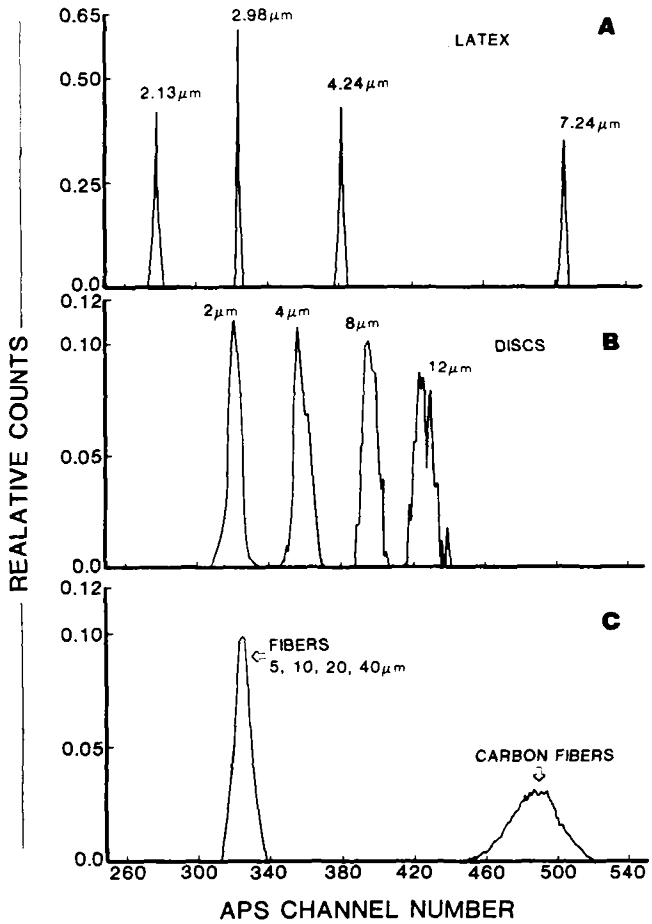

Figure 2. Comparison of the APS channel location and distribution for the monodisperse latex calibration spheres, the monodisperse fibers and discs, and the carbon fibers.

DISCUSSION

The sensitivity of the APS to disc diameter is weak, with the APS diameter lower than the theoretical diameter when the aspect ratio of thickness to diameter falls below 0.25. This implies that the APS might be used to determine the aerodynamic diameter of discs, if appropriate correction factors are developed.

The APS is insensitive to fiber length, with all particles of the same diameter falling in the same channels, regardless of length. The broader distribution of APS response to the carbon fibers (Fig. 2) can be fully attributed to the variation of fiber diameter within the sample.

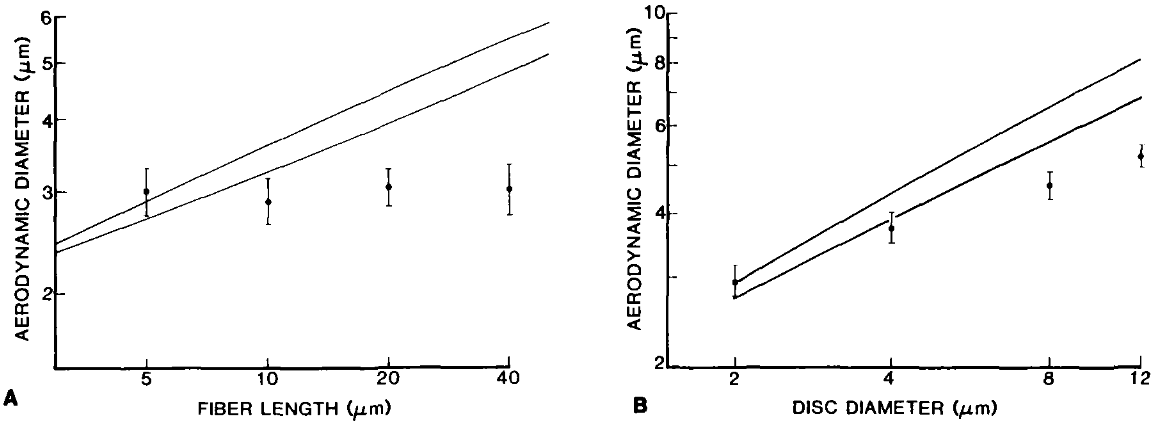

Figure 3. Comparison of the aerodynamic diameters measured by the APS to theoretically predicted aerodynamic diameters for (A) the monodisperse fibers and (B) the monodisperse discs. The upper theoretical curves are for orientation of the particle axis of symmetry parallel to the flow and the lower theoretical curves are for orientation of the particle axis of symmetry perpendicular to the flow for fibers of 1 μm x 1 μm cross section or discs of 1 μm thickness.

This implies that the APS might be used to determine the geometric diameter distribution of fibers, because such measurements will be independent of fiber length.

For both fibers and discs, the aerodynamic diameters measured by the APS fell outside the envelope of theoretical predictions for parallel and perpendicular orientations, and the width of the APS aerodynamic response for each particle size was less than the theoretical difference between parallel and perpendicular orientations. Thus, the question of whether nonspherical particles assume a preferred orientation in the APS remains unanswered.

The integrated circuit fabrication method is a viable way to make nonspherical monodisperse particles for aerosol science studies. Particle thickness of 1 μm has been used and it may be possible to adapt the process to preparing particles of other thicknesses. The method for removing the particles from the wafer substrate was efficient, but a more efficient aerosolization method than nebulization would improve the application of the monodisperse particles.

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