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Comprehensive Particle and Fiber Testing for Cleanroom Wipers

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ABSTRACT

A microscopy-based measurement technique has been developed for accurately quantifying the total number of particles and fibers in all size ranges released from a cleanroom wiper during conditions of simulated use. The method uses both optical and scanning electron microscopy in conjunction with computerized image analysis for counting particles and fibers. The technique uses a single sample preparation for the extraction of both particles and fibers from the wiper. Seven cleanroom wiping products were examined using this measurement technique. The results are shown for the various size groupings of particles and fibers. A composite measure of contamination is presented. This work reveals that a cleanroom wiper may appear very clean in certain size categories and appear extremely dirty in others. The results of this study show that all size ranges of particles and fibers need to be examined when determining the total contamination risk posed by any cleanroom wiper.

In the semiconductor and data storage industries, cleanroom consumables such as wipers are routinely tested to determine the extent of releasable particles in the submicron to few-micron-sized range. However, fibers and large particles may also constitute a significant contamination threat to many processes run in these critical manufacturing environments. A large-sized contaminant, such as a fiber released from a wiper, can cause serious damage over a wide area during fabrication of microelectronic circuitry. Many processes that can withstand a certain level of small-particle contamination are extremely vulnerable to large particles and fibers.

A wiper may appear very clean when tested for particles in the submicron range, yet can be a big contributor of large particles and fibers. Different parts of a wiper often contribute different sizes and types of particles. While the body of a wiper can be the major source of submicron particles, the unsealed or poorly sealed edges are usually responsible for shedding large particles and fibers. Fibers are also released from the exposed yarn loops on the cut edges of a wiper.

Laser-based liquid particle counters have been used traditionally for counting releasable particles emanating from wipers.[1,2] However, particle counters can measure only a narrow range of particle sizes. Any particles or fibers smaller or larger than the given size range either remain uncounted or are counted inaccurately.[3] The primary focus of the technical work presented in this paper is the development and implementation of a comprehensive test procedure that can address the accurate counting of all particles and fibers released from a wiper irrespective of size. This technique builds on previous work by the authors in both wiper sample preparation and direct counting of particles through microscopy.[3,4] In this new procedure, both scanning electron

Keywords: contamination control, cleanroom wipers, particles and fibers, scanning electron microscopy, optical microscopy, particle counting and sizing

microscopy and optical microscopy are used in conjunction with computerized image analysis to count and classify particles and fibers by size.

The procedure uses a single sample preparation for the enumeration of all sizes, thereby eliminating the need for separate sample preparations for particles and fibers. The preparation involves immersing and agitating a wiper in a low-surface-tension cleaning liquid and filtering the particle-laden liquid through a microporous membrane filter.[4] The filter is then mounted on a scanning electron microscope (SEM) stub and first examined for the uniformity of distribution using the optical microscope. Once uniformity is determined, large fibers are also counted during this step. The sample stub is then transferred to the SEM, and particles of different size categories are counted at different magnifications. The accuracy and the precision of the resultant counts are measured statistically.

The results are classified by size into three categories: small particles between 0.5 μm and 5 μm; large particles greater than 5 μm but smaller than 100 μm; and large particles and fibers equal to or greater than 100 μm. The use of scanning electron and optical microscopes as viewing and enumeration tools provides a direct and precise measurement of the quantity and type of contamination.

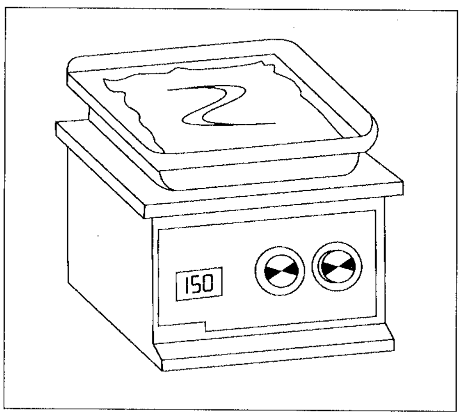


Figure 1. Test setup using an orbital shaker to agitate the wiper.

Experimental Procedure

The procedure consisted of two distinct operations: first, the preparation of the sample; and second, the counting of the fibers and particles collected on the filter.

Fibers were counted manually using an optical microscope at 20X magnification. Large and small particles were counted using a SEM at 200X and 3000X magnifications, respectively. At 200X, computer-aided image analysis and counting were used. At 3000X, counting was performed either manually or automatically, depending on the number of particles per field of view.

To make the visualization of particles and fibers very clear and unambiguous in the manual counting process, the optical microscope was equipped with a video camera and a computer with frame-grabbing software. The same computer hardware and software were also used for automatically counting particles using a SEM at 200X and 3000X magnifications.

Sample Preparation

All experimental work was performed under a Class 100 or cleaner laminar flow workstation. The procedure was designed to use a single sample preparation for analysis of all particle sizes. Therefore, one sample stub was prepared per test specimen. Since one sample stub was used to quantify the entire releasable contamination from a wiper, the sample preparation technique needed to aggressively capture the total contamination burden both in terms of particles and fibers.

The procedure involved immersing a wiper in a standard low-surface-tension cleaning liquid, such as a surfactant/water solution, and then subjecting the wiper to mechanical energy by agitation of the liquid. The combination of stress and exposure to cleaning liquids substantially enhanced the release of particles and fibers from a wiper into the test liquid. This served as a realistic indicator of the contamination load that could be released from a wiper during actual use.[4]

The test wiper was placed into a clean 6 cm × 32 cm × 46 cm (2.4 in. × 12.6 in. × 18.1 in.) polyethylene photographic tray filled with 500 mL of deionized water and a 25-mL aliquot of a 0.1 percent surfactant-based cleaning solution. The tray was then agitated using an orbital shaker (Model 3520 from Lab-Line Instruments) at 150 rpm for 5 min (see Figure 1). Because the wiper stayed flat in the tray, its entire surface was always exposed to the effects of the mechanical agitation through the liquid.

In formulating the cleaning solutions, polyoxyethylenated alkyl phenol type surfactants, such as Triton X-100, were found to work very well in terms of low particulate content and the ability to release particles from wipers. An isopropyl alcohol/deionized water solution works equally well.

After agitation, the wiper was removed, the size was measured, and the liquid from the tray was poured into a clean 2-L beaker. To ensure that all the released particles and fibers were collected, the tray was rinsed with an additional 25 mL of deionized water, and the rinse water was added to the beaker. The contents of the beaker were filtered under vacuum using a 0.40-µm polycarbonate membrane filter. The filtration setup consisted of a stainless steel screen and a steel funnel, a Teflon gasket, a spring clamp, and a vacuum pump capable of delivering minimum 50 torr of vacuum pressure.

The filter was allowed to air dry and was then transferred to an aluminum specimen stub. The perimeter of the filter was affixed by applying several spots of conductive carbon paint. The last step of the sample preparation involved applying a thin layer of gold coating by using vacuum sputtering under an argon atmosphere.

For this research, seven polyester cleanroom wipers were evaluated. All the selected wipers were made from knitted, continuous-filament polyester material. Six of them had treated edges, either hot-cut or border-sealed, and one was an unsealed wiper. The latter was tested to compare the contamination characteristics in relation to the sealed-edge products. All tests were run using the same photographic tray, beaker, and filtration assembly. As with all other evaluation techniques, background particle and fiber counts of the system blanks, consisting of the clean tray, the clean beaker, and other paraphernalia, were taken by preparing background stubs prior to the preparation of each test sample stub.

Manual Counting of Large Fibers (≥ 100 µm) Using Optical Microscopy at 20X Magnification

The sample stub for each test was first viewed using a stereobinocular microscope at 20X magnification. The surface of the membrane filter was uniformly and sufficiently illuminated with an incandescent fiber-optic light source. The fibers/particles on the filter surface were then brought into focus for examination and counting.

Before beginning the counting of fibers, the entire surface of the filter was initially scanned by moving it randomly in vertical and horizontal directions while noting the uniformity of the distribution of particles and fibers throughout the filter. If the inspection revealed any nonuniformity of particle and fiber distribution, the particular stub was discarded and replaced by preparing a new sample stub using wipers from the same batch.

For the actual counting of all the 100-µm and larger-sized particles and fibers on the filter, the entire filter area was slowly scanned vertically and horizontally by moving the X and Y arms of the sample stage. During each increment, fibers and large particles were counted. By performing a size calibration study beforehand, size criteria could be established for accurate counting of particles and fibers that were 100 µm or larger in any dimension. The vertical and horizontal movements were continued until the filter was completely scanned and all large-size fibers and particles were counted.

The system blank filter prepared prior to each sample run was also examined and counted by following the same procedure. The net amount of fibers and particles 100 µm and larger was determined by subtracting background numbers from the sample counts. Finally, the number of particles and fibers per square meter of wiper material was determined by dividing the net count by the area of the wiper. The results were compared for all seven wipers. The average counts for the large particles and fibers (□ 100 µm) for all seven wipers are presented in Table 1 under 20X.

Table 1. Summary of Results

Wiper	20X	200X (in thousands)	3000X (in millions)
A	3057	747	10.3
B	8524	429	16.8
C	5699	5656	932
D	3009	948	34
E	1680	1042	199
F	5733	5933	109
G	1044	92	5.2

Note: Average particle and fiber concentration (number per square meter) for each wiper is at the indicated magnifications.

The data in Table 1 are graphically presented in Figures 4, 5, and 6. Note that the rank ordering in terms of cleanliness of the seven wipers varies according to the particle size range examined with the exception of Wiper G, which is the cleanest in all size categories.

Computer-Assisted Counting of Large Particles (5 µm–100 µm) Using Scanning Electron Microscopy at 200X Magnification

Next, particles between 5 µm and 100 µm were counted by using scanning electron microscopy, where the same stub was examined at 200X magnification. Because the particle density at this magnification is so great for a typical wiper, computerized image analysis and counting were used during this step.

The sample stub used in the optical microscope was first transferred to a SEM sample holder, which was then slid inside the sample chamber of the SEM. The chamber was evacuated, the filament was turned on, and the SEM was prepared for viewing the particles. The SEM parameters, such as focus, contrast, brightness, and tilt angles at specific magnifications, were adjusted properly to permit accurate visualization of particles on the filter surface.

For the computerized image analysis and automated counting, size restrictions on the particles and fibers to be included were set by programming the appropriate parameters in the computer software. The area parameters were initially determined by conducting a size reference study in the SEM using a sample stub containing standard 5-µm and 100-µm polystyrene microspheres.

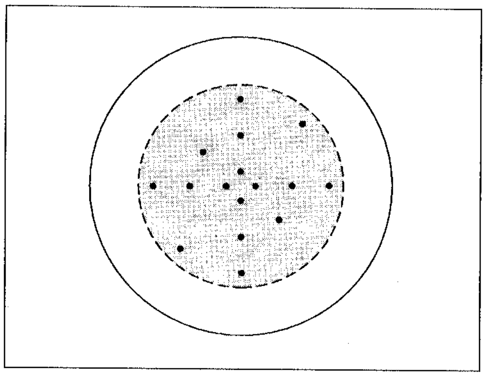


Figure 2. Layout of 16 preselected points for the counting at 200X.

Counting was performed by scanning a statistically representative number of fields[5] on each filter and then averaging the number of particles per field. Sixteen such locations were preselected for the counting at 200X and stored in the computer memory in the SEM. The locations were retrieved one at a time and particles were counted in each field. The locations were selected to cover the central area of the filter, the area at approximately half the active radius of the filter, and the area proximal to the edge of the filtration surface as shown in Figure 2.

The data were then subjected to statistical analysis with an objective to achieve ±10-percent accuracy at a confidence level of 95 percent. The filters from the system blanks prepared at the start of each experiment were examined and counted using the same technique. Net particles per field of inspection were determined by subtracting the background counts from the sample counts. The area of the active filter, the area of the field in the SEM at the selected magnification, and the area of the wiper sample were all determined to calculate the number of particles per square meter of the wiper. The average counts for the large particles (5 µm to 100 µm) for all seven wipers are presented in Table 1 under 200X.

Manual/Computer-Assisted Counting of Small Particles (0.5 µm–5 µm) Using Scanning Electron Microscopy at 3000X Magnification

The procedure for counting particles between 0.5 µm and 5 µm is similar to what has been described for the 200X study. The magnification used for this particle size range is 3000X. Both manual and computer-assisted counting techniques were used depending on particle density per viewing area of the filter. The samples showing less than 25 particles per field of inspection were counted manually. However, computer-based image analysis and counting, similar to that used in the 200X study, were used for fields where the particle density was greater than 25 particles per field and deemed too high to be accurately determined by manual counting.

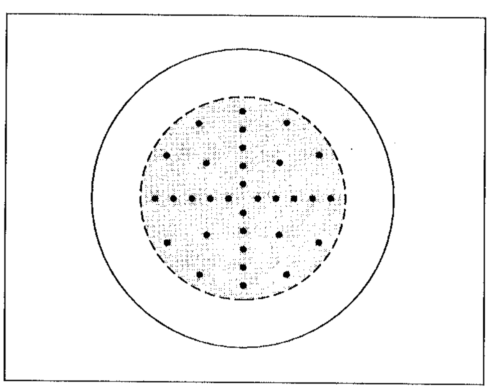


Figure 3: Layout of 32 preselected points for the counting at 3000X.

Once again, counting was performed by scanning a statistically representative number of fields on each filter and then averaging the number of particles per field. At 3000X, 32 such locations were preselected for counting and stored in the computer memory in the SEM (see Figure 3). The locations were retrieved one at a time and particles were counted in each field.

In order to restrict the sizes of the particles counted using the image analysis software, area parameters were initially determined by conducting a size reference study in the SEM using standard 0.5-µm and 5-µm polystyrene microspheres. The software was then programmed accordingly. The correction for the background level of particles and the final calculation for the total particles per square meter of the wiping material were all performed using the steps described in the 200X study. The average counts for the small-sized particles (0.5 µm to 5 µm) for all seven wipers are presented in Table 1 under 3000X.

RESULTS AND DISCUSSION

The idea of size-differentiated counting initially surfaced as supplementary information during the development of an accurate method for particle enumeration using scanning electron microscopy. Initially, the primary objective was to develop a SEM counting method for releasable submicron-size particles in cleanroom wipers using appropriate magnifications, such as 2000X to 5000X. However, lower magnifications, such as 100X and 200X, were also routinely used to inspect the uniformity of particle distributions in the filter samples. Gradually, it became evident that the types and levels of contamination in wipers show a wide spectrum of variation depending on the magnifications used for viewing the sample filter. The current test procedure incorporates all those findings, making it a method appropriate for complete quantification of particles and fibers in cleanroom wipers.

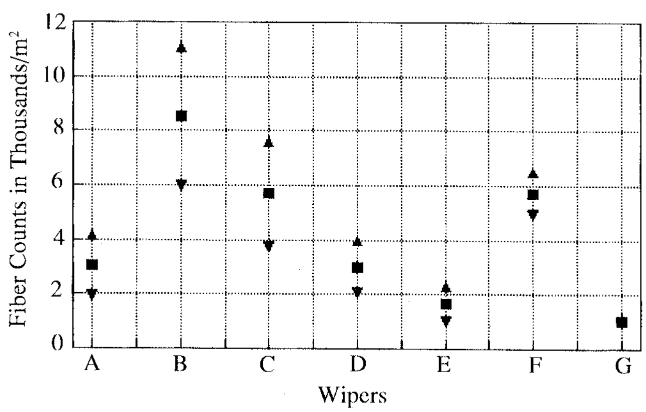


Figure 4. Fiber release comparison (showing high-low ranges) from wipers A—G, as determined by optical microscopy at 20X magnification.

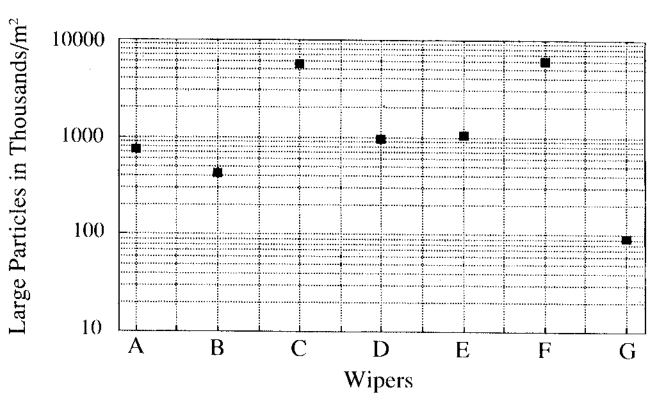


Figure 5. Semilog plot of average values of large particle counts for wipers A—G, as determined by scanning electron microscopy at a magnification of 200X.

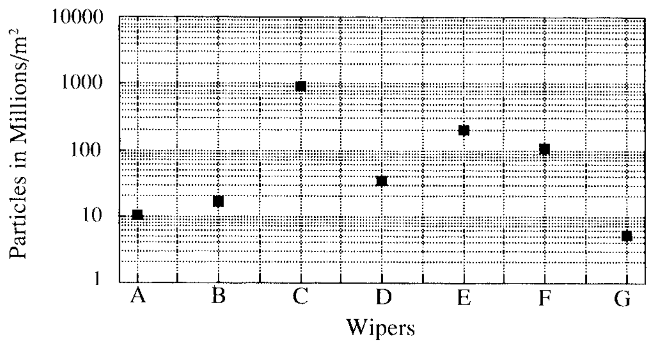


Figure 6. Semilog plot of average values of small particle counts for wipers A—G, as determined by scanning electron microscopy at a magnification of 3000X.

The viewing area at a high magnification such as 3000X is only 1,533 μm^2, which makes the complete capture of widely scattered large particles and fibers unlikely, considering the number of fields scanned. A more macroscopic picture is painted at the lower magnifications, where the field areas are much greater and the capture of large particles and fibers is more ensured. However, at the lower magnifications, small particles are unlikely to be seen. To ensure the complete capture of particles and fibers in all size ranges, both optical and scanning electron microscopy techniques are used for this test procedure.

It is apparent from the photomicrographs that wipers tend to show different types and levels of contamination when inspected by microscopic techniques at different magnifications. Wiper A, for instance, was very clean when tested for small particles (0.5 µm to 5 µm) at 3000X. However, an examination at lower magnifications revealed a substantially higher amount of particles and fibers in the larger size ranges. Note that the high burden of large particles and fibers did not interfere with the accurate counting of small particles at 3000X. Wiper B exhibited a similar profile to Wiper A with the exception of large fibers (□ 100 µm), which were detected at 20X magnification. This profile is consistent with the fact that Wiper B was the only wiper with untreated edges.

Wiper C is an example of a wiper that showed a high burden of contamination in all size categories and was found to be the dirtiest among all the wipers used for this study. Wiper F also exhibited large amounts of contaminants in all size categories. (Due to space limitations, photomicrographs of Wiper F are not shown; see data in Table 1.)

Wiper E showed an interesting profile in that it was relatively clean at the lower magnifications (20X and 200X), but extremely dirty in small particles at the higher magnification (3000X). This suggests an applied surface treatment or defects in the base polymer itself. The number of particles and fibers found in Wiper D were similar to Wiper A, except in submicron-sized particles which were three times higher in Wiper D. (Photomicrographs of Wiper D are not shown; see data in Table 1.) The last set of photomicrographs from Wiper G showed a contamination profile that was uniformly clean over all size ranges.

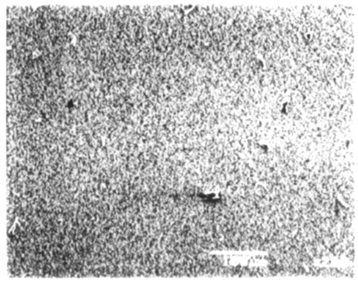
As the photomicrographs show, each wiper has its own contamination profile in the various particle and fiber size ranges. The fact that particles or fibers are prevalent in one size range and not in another can be a function of the manufacturing, fabrication, or cleaning processes.

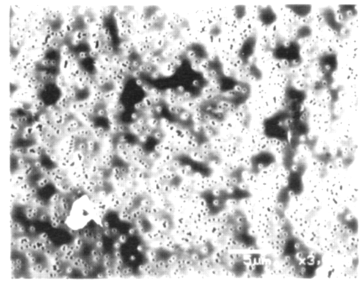
Quantitative Comparison

For a quantitative comparison, multiple samples from each wiper batch were evaluated using the new test procedure to ensure consistency and also to generate statistically reliable average numbers for the particle and fiber counts. In reporting the final results, numbers were rounded up to two significant digits if the first digit of the number was three or greater, and three significant digits if the first digit was less than three. The numbers within each size category were then used for drawing the comparison in cleanliness between wipers. Table 1 presents average particle count for each wiper in all three size categories.

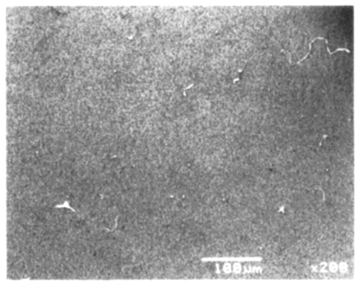
Photomicrographs

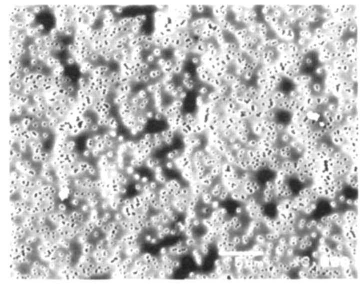

Wiper A 20X

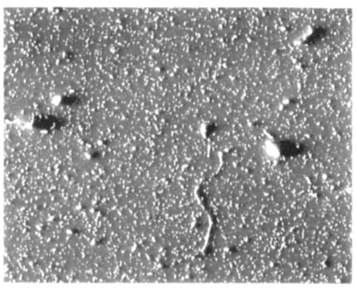

Wiper A 200X

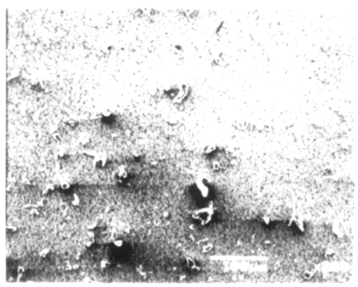

Wiper A 3000X

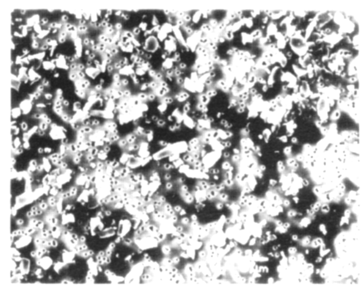

Wiper B 20X

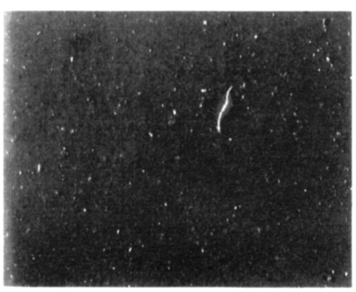

Wiper B 200X

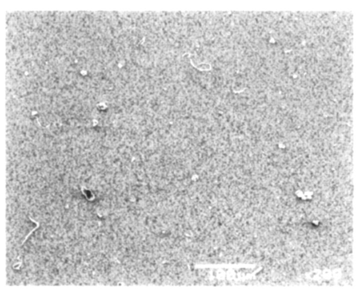

Wiper B 3000X

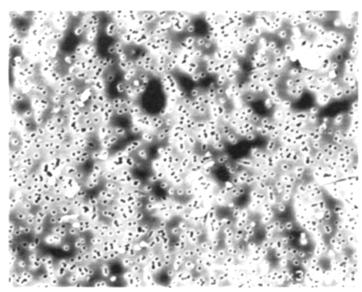

Wiper C 20X

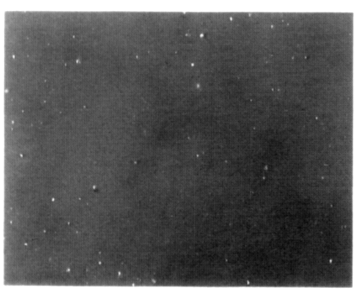

Wiper C 200X

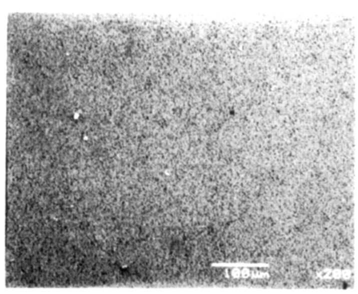

Wiper C 3000X

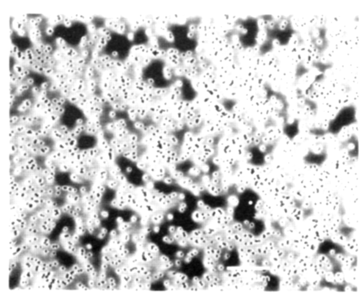

Wiper E 20X


Wiper E 200X


Wiper E 3000X


Wiper G 20X


Wiper G 200X


Wiper G 3000X

Total Contamination Index

Table 2 compares the particle counts obtained at each magnification for each wiper type in terms of a ratio. The data were obtained by using the particle counts from the cleanest wiper (Wiper G) as a standard. The ratios were obtained by dividing the number of particles in each wiper by the corresponding particle count for Wiper G and then rounding them to the nearest whole number. For example, for wiper A at 20X, dividing 3,057 by 1,044 yielded a ratio of three. Similarly, for the same wiper at 200X, a ratio of seven was obtained by dividing 747 by 92, and so on. The ratio results depict comparatively the levels of particles and fibers in the various size ranges each wiper could release during use. The ratios were then summed up across all magnifications to provide a total contamination index as a means of direct comparison among wipers.

The current study revealed that in order to obtain an accurate picture of the cleanliness of a cleanroom wiper, the full range of the particles and fibers released from that wiper must be determined. Looking at a single magnification and drawing conclusions by measuring particles limited to only one particular size range is inappropriate and can lead to erroneous results. A composite consisting of particle counts at the three magnifications described provides a comprehensive analysis of the amount of particles and fibers a wiper can release during use, and thereby provides an effective measure of the contamination risk that a wiper poses to a critical manufacturing process.

Table 2. Particle Count Ratios for the Seven Wipers Compared with Wiper G

Wiper	20X	200X	3000X	Total Contamination
G	1	1	1	3
A	3	8	2	13
B	8	5	3	16
C	6	62	180	248
D	3	10	7	20
E	2	11	38	51
F	5	65	21	91

Note: Table 2 data are graphically presented in Figures 7 and 8.

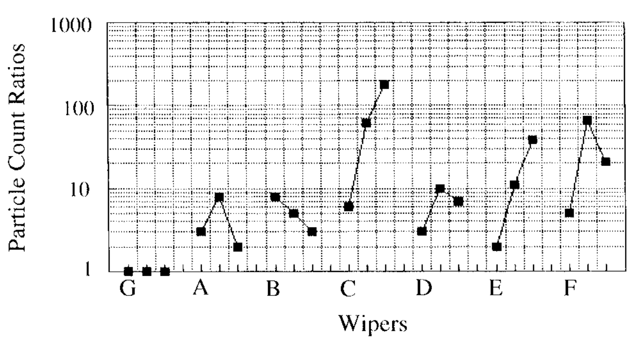


Figure 7. Semilog plot of the ratio of particle counts for all wipers, as determined by microscopy at various magnifications. For each wiper, the first number (from left) is at 20X, the second is at 200X, and the third is at 3000X.

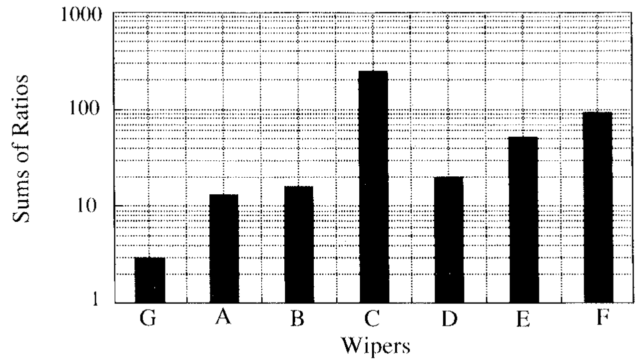


Figure 8. Semilog plot of the total contamination index for all wipers.

CONCLUSION

A new microscopy-based comprehensive test method for measuring the various size ranges of particle and fiber contaminants was used to compare samples of seven different knitted polyester cleanroom wipers. The technique used a single sample preparation, but examined the sample at three different magnifications, using both optical microscopy and scanning electron microscopy. The goal was to explore the effectiveness of the method in quantifying various size groupings of particles and fibers. The experimental test results revealed substantial differences between the cleanliness of the test samples. The most intriguing part of the test data was that while some of the wipers appear very clean in certain size categories, they can be extremely dirty in other size categories. This inconsistency can be masked if only a single magnification is used.

A method of using ratios of particle counts compared with the cleanest wiper in the different size categories was used. Wipers were compared by individual size category as well as by the sum of their relative ratios to provide a total contamination index. The results of this study show conclusively that a comprehensive analysis of all size ranges of fibers and particles needs to be performed to determine the contamination risk imposed in the selection of a cleanroom wiper.

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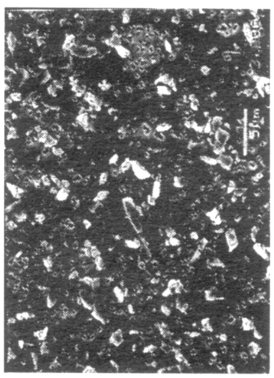
Steven J. Paley is president of The Texwipe Company LLC, Upper Saddle River, New Jersey. In addition to overall corporate responsibilities, Mr. Paley oversees all research and development, engineering, and manufacturing process development. His previous employment includes IBM Corporation where he worked on the System 38 computer development, and AT&T Bell Laboratories where he was involved in the development of specialized flat screen computer terminals for medical information systems. He is a member of the Institute of Electrical and Electronic Engineers and a Senior Member of the Institute of Environmental Sciences and Technology where he serves on the working group for wiper testing. Mr. Paley has co-authored numerous papers on contamination metrology for consumable cleanroom products. Mr. Paley holds an M.S. degree in Mechanical Engineering/Product Design from Stanford University as well as a B.S. degree in Electrical Engineering, and a B.A. degree in English literature from Tufts University.



COVER PHOTO: SEM (Scanning Electron Microscopy) utilized as a metrology tool of choice for the counting and analyzing of particles and fibers. This 3000X photomicrograph, taken at Texwipe's Research and Development Laboratory, shows particle distribution on a polycarbonate membrane filter. Photo used courtesy of Texwipe.