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Evaluating sample preparation techniques for cleanroom wiper testing

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CLEANROOM TECHNOLOGIES

Using mechanical energy and a surfactant solution to remove wiper particles can approximate actual conditions of use.

The testing of cleanroom wipers has evolved over the past 20 years from simply shaking a material and visually approximating the amount of lint released to much more complex methods of separating particles from the wiper followed by quantification using sensitive analytical instrumentation. Controversies such as wet versus dry testing—removing particles via immersion in liquid as opposed to agitation in air—which predominated in the late 1980s, have today mostly been resolved in favor of wet testing.[1,2] Published by the Institute of Environmental Sciences in June 1992, IES Recommended Practice 004.2 details two methods for removing particles from a wiper for subsequent enumeration using a laser-based liquid particle counter (LPC) or optical microscopy.[2] The first technique involves gently immersing the wiper in deionized (DI) water, the second a more vigorous shaking of the wiper in a vessel containing DI water.

An earlier research project at Texwipe examined the available techniques for counting wiper particles in liquid suspensions.[3] The work demonstrated the deficiencies of devices such as laser-based LPCs, leading us to develop and recommend a measurement based on scanning electron microscopy (SEM). The use of SEM metrology for particle counting provides a method that is sufficiently sensitive to discern with great accuracy the number of particles released during sample preparation.[3–5] However, the issue of how to prepare test samples in a manner that closely approximates the actual conditions of use invited further research. Thus, we proceeded to compare existing techniques with new and modified methods for removing particles from a wiper for subsequent counting. This article describes those experiments and discusses their results.

Experimental Equipment and Conditions

Four different sample preparation techniques were evaluated using five different wipers chosen to represent a spectrum of those marketed for use in critical environments. The five types of wipers selected are made from knitted, continuous-filament polyester material with hot cut or sealed edges. As described below, these wipers were exposed to a variety of sample preparation environments during testing. The particles released from them by those preparation methods were then counted using SEM. All experimental work was performed under Class 100 or better conditions.

Minimal Stress Test

The standard minimal stress test described in IES RP-004.2 involves gently immersing the wiper to be tested in a clean photographic tray filled with approximately 500 ml of DI water. The tray is then lifted slightly to ensure that all wiper surfaces are completely wetted, and the water is allowed to move gently back and forth across the wiper before being decanted into a clean 2-L beaker. Two more rinses of the wiper are then made in the same manner, and water from both of those rinses is added to the collection beaker for enumeration of the released particles.

Because a significant amount of data generated using this sample preparation technique are available, it was used as a basis of comparison for the other techniques evaluated in this study. Tests of the five wipers were run using the same polyethylene photographic tray measuring approximately 6 × 32 × 46 cm and a 2-L beaker. Background particle counts of both the clean tray and the clean beaker were taken prior to the introduction of test sample.

Minimal Stress Test in a Low-Surface-Tension Environment

A positive relationship has been shown between the amount of mechanical energy applied and the number of particles released from a wiper.

Most cleanroom wipers are used in cleaning applications in conjunction with solutions that, typically, are formulated to solubilize certain types of contaminants and to lower the surface tension of the area to which they are applied in order to better remove nonsoluble surface contaminants.

The ability of the solutions to penetrate the interfaces between particles and surfaces affects both the surface to be cleaned and the wiper that is doing the cleaning. That is, if there are particles adhering to the wiper, they will be much more readily removed in a low-surface-tension environment than in pure DI water.

Figure 1: Applied mechanical energy test setup using an orbital shaker to agitate the wiper.

One of the goals in optimizing a technique to release particles from a wiper was to subject it to an environment similar to the conditions of use. The second test method evaluated, therefore, is identical to the standard minimal stress test except that instead of pure DI water, a common cleaning solution such as a surfactant/DI-water mixture or an isopropyl alcohol (IPA)/DI-water mixture is used as the particle-release agent. In the experiments, the surface energy of both such solutions measured <40 dyn/cm compared with DI water at 72 dyn/cm. (The results reported later for this method are those for the surfactant/DI-water mixture unless otherwise stated.)

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. Stock solutions were prepared by diluting 1 ml of the concentrated surfactant in 1 L of DI water and heating the mixture at 40°C with agitation. A 25-ml aliquot of the resulting solution diluted in 500 ml of DI water was utilized for testing, creating a 0.005% concentration by volume of surfactant in water. To prevent biological growth, fresh stock solution was prepared prior to each test. In the case of IPA, the test solution was prepared at a 50% concentration by mixing the alcohol and DI water in a 1:1 ratio.

Applied Mechanical Energy

When wipers are used for cleaning, a degree of mechanical energy is imparted to both the fibers in contact with the wiped surface and the fibers within the wiper body as they are stretched and twisted against one another. It has been shown that there is a positive relationship between the amount of mechanical energy applied and the number of particles released from a wiper.[6,7] It therefore seemed appropriate to develop and evaluate a sample preparation method that involves the input of mechanical energy into the test environment. This task was constrained by the necessity of ensuring that all particles released by the procedure were available for capture.

The biaxial shake test described in IES RP-004.2 succeeds in imparting mechanical energy into the test environment as well as making the released particles available for counting in a liquid suspension. However, the test wiper must be folded or contorted to fit within the diameter of the shaker jar. Different wiping materials flex in different ways, and the initial wiper position in the jar can affect the final results. In contrast, if a laboratory orbital shaker is used for agitation, the wiper can be placed flat in a clean photographic tray similar to the one used for minimal stress tests. The tray is set onto the shaker and agitated at a set number of revolutions per minute for a fixed time. Because the wiper lies flat in the tray, its entire surface is always exposed to the effects of the mechanical agitation through the liquid. (The orbital shaker has already been adopted as the preferred test device for the particle testing of gloves by IES Working Group 005.)

The basic applied mechanical energy test developed for this research involves placing a test wiper in a clean photographic tray filled with 500 ml of DI water (see Figure 1). The tray is agitated at 150 rpm for 5 minutes on an orbital shaker with an orbit radius of 3/8 in. (Model 3520, Lab-Line Instruments, Melrose Park, IL). The wiper is then removed from the tray and the liquid decanted into a clean beaker. Next, the tray is rinsed with an additional 25 ml of DI water, which is also decanted into the beaker. The contents of the beaker are filtered for particle counting by SEM. As with all the techniques evaluated, during testing background particle counts of the tray, beaker, and filtration apparatus were run before the introduction of the test specimen.

Applied Mechanical Energy in a Low-Surface-Tension Environment

The final technique evaluated combines the elements of all the tests described above: A wiper is placed in a clean photographic tray containing a cleaning solution. After the tray has been agitated using the orbital shaker for 5 minutes at 150 rpm, the wiper is removed and the liquid decanted into a clean beaker. To ensure that all particles are collected, the tray is then rinsed and the rinsewater added to the beaker before particle enumeration via SEM. (Results reported for this technique include sample preparations using both the surfactant/DI-water mixture and the IPA/DI-water mixture previously described.)

SEM Counting Procedure

Particle counting during the study was performed using a Model JSM-5800LV SEM (JEOL, Tokyo). The supporting paraphernalia included a Vacuum II cold sputter/etch unit (Denton Vacuum, Moorestown, NJ) to deposit gold or gold-palladium coatings on the SEM specimen stubs. The filtration apparatus consisted of a stainless-steel screen and funnel, a Teflon gasket, a spring clamp, and a vacuum pump capable of applying 50 torr of vacuum pressure; polycarbonate membrane filters measured 25 mm in diameter and had a pore size of 0.45 µm. SEM magnifications in the range of 2000–10,000× were used, depending on the particle density per unit area of the filter. These parameters permitted accurate visualization and counting of particles ?0.5 µm on the filter surface.

Counting was performed by scanning a statistically representative number of fields on each filter and then averaging the number of particles per field. The data were then subjected to statistical analysis to determine the confidence level and accuracy of the sample mean. The filters from the system blanks run at the start of each experiment were examined and counted using the same technique, and net particles per field of inspection were determined by subtracting these background counts from the sample counts. To calculate the number of particles per square meter of the wipers, 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.

Sample Type	**Sample Preparation Technique**[a]			
	MS/DIW	**OS/DIW**	**MS/Surf.**	**OS/Surf.**
Wiper A	2.5	3.7	4.8	9.2
Wiper B	7.6	9.1	10.7	22.9
Wiper C	13.3	12.6	29.8	66.8
Wiper D	16.0	17.7	62.8	52.3/99.1[b]
Wiper E	16.3	25.5	3411	6345/4873[b]

Table I: Average particle counts determined for each wiper with each sample preparation technique (in millions of particles per square meter of wiper).

Results and Discussion

Each of the sample preparation techniques was evaluated using multiple samples of each wiper to ensure consistency of data. The particle count results were examined in terms of differences between wipers for each test method as well as for variation in particle counts from the four different test methods. Table I presents average particle counts for each of the wipers, A-E, from each of the four sample preparation techniques. The counts are given in millions of particles per square meter of wiping material. Table II compares the particle counts obtained by each test method for each wiper type in terms of a ratio. These data were obtained by using the particle

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[a] MS/DIW = minimal stress in DI water; OS/DIW = orbital shaking in DI water; MS/Surf. = minimal stress in surfactant/water mixture; OS/Surf. = orbital shaking in surfactant/water mixture.

[b] The first number represents the test result using a surfactant/water mixture, the second the test result using a 50% IPA/water mixture.

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counts from the control sample preparation method—the minimal stress test in DI water—as a standard. For each wiper, this number was divided into the other results shown in Table I to give the respective particle count ratios (shown rounded to the nearest whole number). For example, for wiper A, dividing 9.2 by 2.5 yielded a ratio of 4 for the technique involving orbital shaking in a surfactant.

Sample Type	**Sample Preparation Technique**[a]			
	MS/DIW	**OS/DIW**	**MS/Surf.**	**OS/Surf.**
Wiper A	1	1	2	4
Wiper B	1	1	1	3
Wiper C	1	1	2	5
Wiper D	1	1	4	3/6[b]
Wiper E	1	2	209	389/299[b]

Table II: Particle count ratios comparing data from each sample preparation to that from the standard minimal stress test (ratios are rounded to nearest whole number).

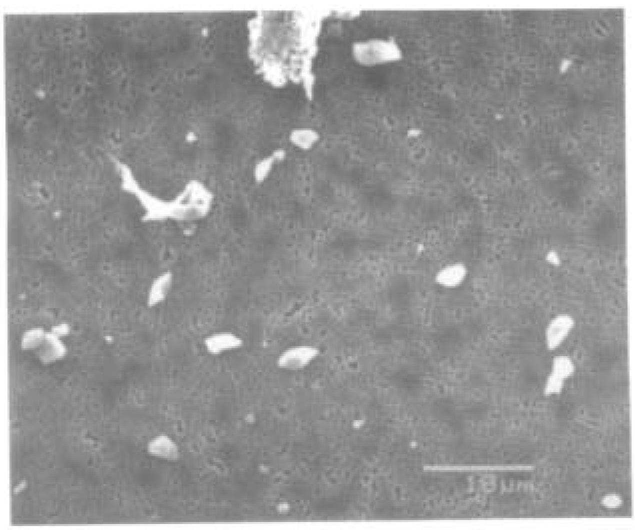

Figure 2: SEM micrograph at 2000× showing particle release from wiper D when subjected to minimal stress in a surfactant/DI-water solution.

The first two columns of ratio results in Table II indicate that adding mechanical agitation to the standard minimal stress sample preparation technique had very little effect on particle release for any of the wipers. Ratios in the third column, which represents minimal stress in a low-surface-tension environment, do differ from those representing the two tests in pure DI water.

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[a] MS/DIW = minimal stress in DI water; OS/DIW = orbital shaking in DI water; MS/Surf. = minimal stress in surfactant/water mixture; OS/Surf. = orbital shaking in surfactant/water mixture.

[b] The first number represents the test result using a surfactant/water mixture, the second the test result using a 50% IPA/water mixture.

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However, these results are inconsistent among the group of wipers, ranging from no significant change for wiper B to a dramatic change of over 200 times the original minimal stress test results for wiper E. A more modest increase of 2–4 times was seen for wipers A, C, and D. Finally, the fourth column, which represents the combination of mechanical agitation and a low-surface-tension environment, shows gains in particle release for all the wipers with the exception of wiper D. For wipers A, B, C, and E, there was roughly a twofold gain in particle release over that achieved by preparing samples using minimal stress in a low-surface-tension environment. The application of mechanical energy clearly had an effect in this environment, in contrast to the negligible effect noted in a pure DI-water environment. Based on an analysis of the ratio data, it can be concluded that the most effective mechanism for releasing particles from wipers involves both mechanical agitation and the immersion of a wiper in a low-surface-tension environment; this also most closely approximates conditions during use.

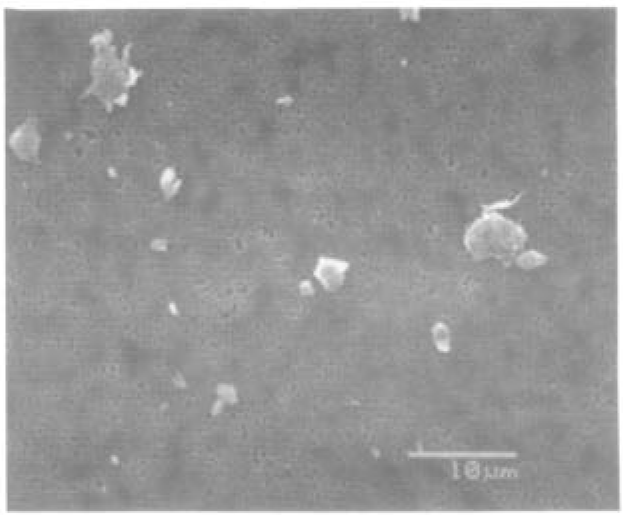

Figure 3: SEM micrograph at 2000× showing particle release from wiper D when subjected to orbital shaking in a surfactant/DI-water solution.

It is also noteworthy that the various wipers reacted differently to the four sample preparation techniques. Particle release from wipers A, B, and C was fairly consistent for all techniques, showing a modest rise when a low-surface-tension environment was introduced. However, wiper D demonstrated a sharper rise in particle counts when subjected to a low-surface-tension environment under conditions of minimal stress (see Figure 2), but its counts declined when mechanical agitation was added (see Figure 3). This variation was certainly not what one would predict; therefore, we suspected that some other phenomenon was taking place that was influencing the results. For example, if some substance added to the wiper fabric during manufacture was reacting with the test surfactant, that reaction could lead to particle agglomeration, reducing the overall particle count, or to the redeposition of particles onto the wiper during agitation. Additional tests using an IPA/DI-water mixture as the surface tension-reducing agent led to the predicted results of increased particle release (see Figure 4). There is no clear-cut explanation for this anomaly, except that wipers treated with chemicals can react in unexpected ways to typical cleaning solutions.

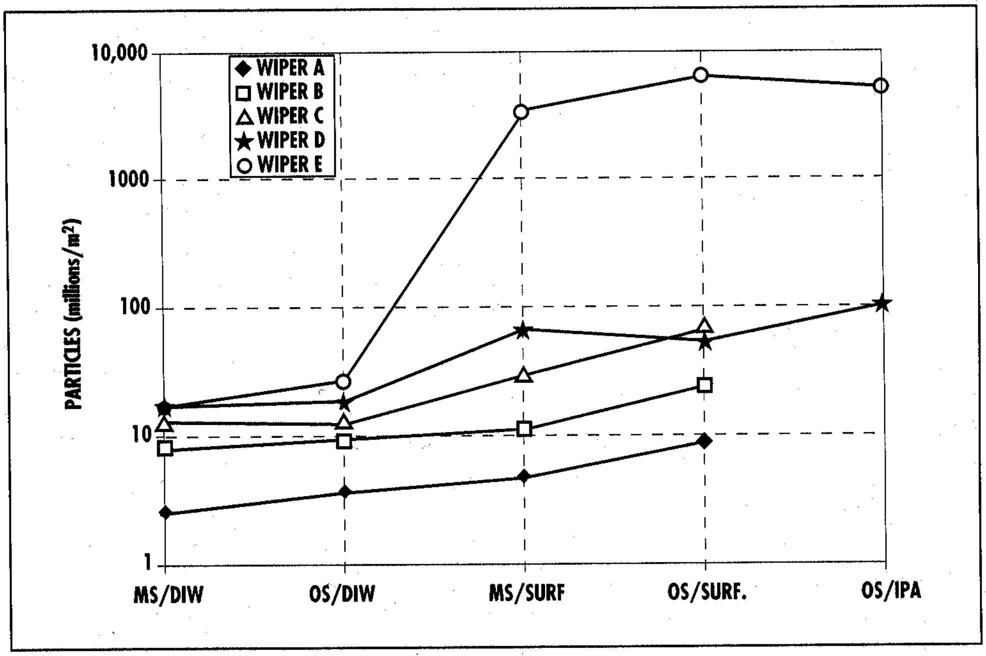

Figure 7: Semilog plot of wiper particle counts for the various sample preparation techniques.

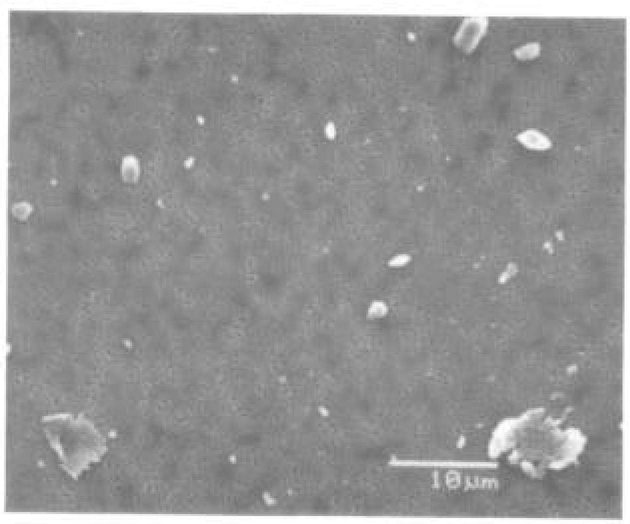

Figure 4: SEM micrograph at 2000× showing particle release from wiper D when subjected to orbital shaking in an IPA/DI-water solution.

Perhaps the most striking results are those from wiper E. This wiper tested relatively clean using sample preparation techniques that involved pure DI water, but once surfactant solutions were introduced to reduce surface tension, the particle counts increased by more than two orders of magnitude. The SEM micrographs presented as Figures 5 and 6 clearly show the significant difference in the number of particles released following orbital shaking in DI water and following agitation in a low-surface-tension environment, respectively. Such increases occurred with both the surfactant test solution and the IPA solution. As with wiper D, it appears that the wiper E fabric was chemically treated to enhance its wiping properties. Although impervious to water, the chemical was easily released in the cleaning agents, accounting for the dramatic increases in particle counts when such agents were introduced into the testing.

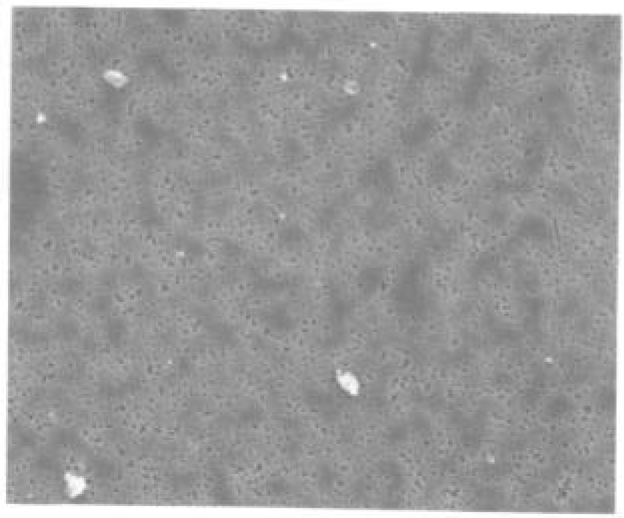

Figure 5: SEM micrograph at 2000× showing particle release from wiper E when subjected to orbital shaking in DI water.

While the particle count ratios were roughly comparable for wipers A, B, and C for all the sample preparation techniques, the actual number of particles released increased proportionally for each of these wipers as more environmental factors were incorporated in the sample preparation technique. For example, as Figure 7 indicates, these wipers generated twice as many particles in the test involving applied mechanical energy in a low-surface-tension environment as in the test using minimal stress in a similar environment. Any claims of proportionality between the wipers break down, however, when results for wipers D and E are examined. As discussed previously, the behavior of these wipers in a low-surface-tension environment was complex and unpredictable because their surfaces reacted in some way with the test solutions to generate particles.

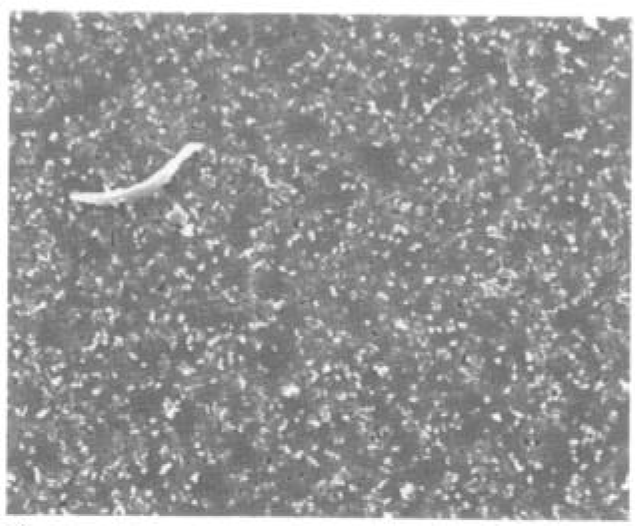

Figure 6: SEM micrograph at 2000× showing particle release from wiper E when subjected to orbital shaking in a surfactant/DI-water solution.

Conclusion

To provide useful risk-assessment data, testing of consumable materials that are brought into cleanroom environments needs to closely simulate the conditions of use. The goal of the research described here was to explore the design of such a test for cleanroom wipers. The resulting data clearly indicate that there are significant differences in the particle counts obtainable using different sample preparation techniques. While counts from the five tested wipers differed when the well-accepted minimal stress test in DI water was used, that variation was insignificant in comparison with the differences achieved using the more rigorous test method involving applied mechanical energy in a low-surface-tension environment. Although the number of particles released in all wipers is greatest using applied mechanical energy combined with a low surface tension environment, this test most clearly and realistically shows how the wipers will perform under actual conditions. Rather than understate the contamination risk, this test method provides a true assessment of risk during typical use conditions.

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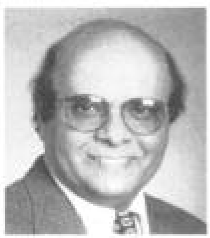

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