CLEANROOMS

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Cleanroom Wipers: State-of-the-Art Evaluation Techniques

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What makes the difference in wiper products is the amount of surface contamination they leave behind after use. Particles and fibers, non-volatile residue, metals and metal ions, anions and ESD propensity must be properly evaluated and quantified using techniques such as Soxhlet extraction, scanning electron microscopy (SEM), capillary ion electrophoresis (CIE), surface resistivity testing, charge decay testing and energy-dispersive x-ray (EDX) analysis.

A wide choice of consumable products is offered for sale to the cleanroom marketplace. Distinguishing among these products—wipers, swabs, stationery, face masks, gloves, etc.— to determine their suitability for cleanroom use is difficult without in-depth testing and measurement of critical characteristics. Testing is often an area of controversy as scientific principles are balanced against intuition and ease of testing. In this article, I will attempt to provide a framework for evaluating consumable products, indicate which tests are most important, and detail the current state-of-the-art in the testing of cleanroom wipers.

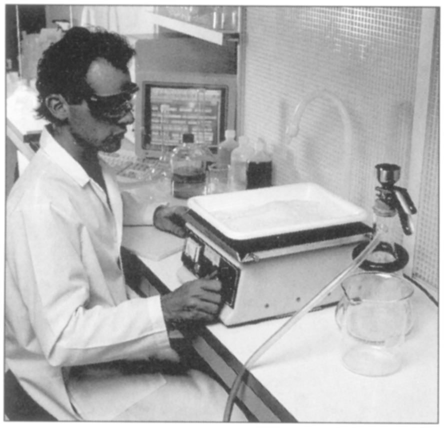
Why test? Is there really a difference?

As device geometries continue to get smaller, the ability to control contamination becomes an enabling technology for the production of integrated circuits and data storage devices. Everything that enters the cleanroom must be considered suspect, in that it can contribute to contamination and affect both yield and reliability. It is therefore incumbent on both the process engineer and the contamination control engineer to have a thorough understanding of all items that are brought into the cleanroom. Consumable articles such as wipers require particular scrutiny because of their ubiquity in the cleanroom and because they are used in almost all fabrication processes.

As it turns out, there are significant differences among the various wiping products, and these differences can be clearly shown through proper testing. The examination of wiping materials can be broken down into two categories: performance characteristics and contamination characteristics. The performance of a wiper involves qualities such as absorbency, wettability, heat resistance, and chemical resistance. These properties are easily tested for by well-accepted standard test methods[1]. A wiper is generally purchased to perform a specific task, such as cleaning a surface, absorbing a spill, or applying a chemical to a surface. Performance measurements are an indicator of how well suited the product is for that task. If performance were the only story, however, wiping products acceptable for most applications could be purchased in supermarkets at far lower prices than those products specifically designed for cleanroom use.

SEM equipment used for particle enumeration and analysis.

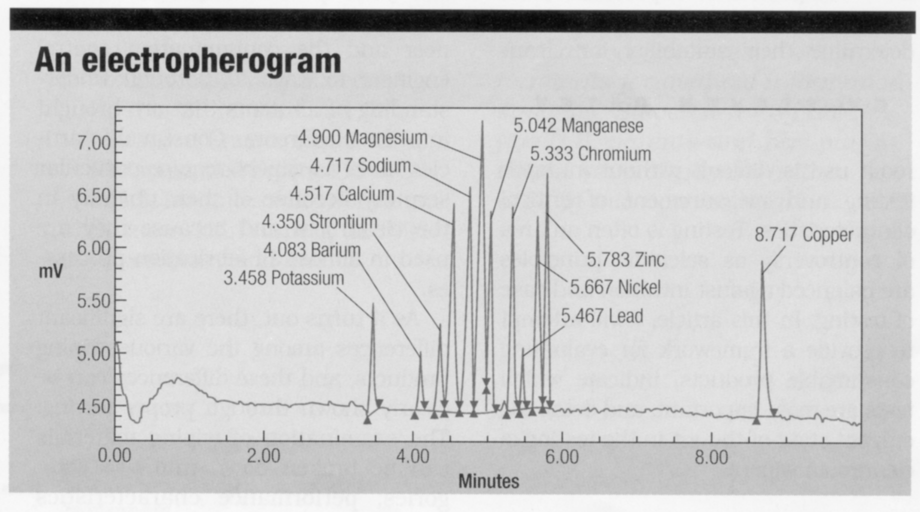
While most wipers are good at absorbing liquid or wiping wetted surfaces, the major difference among products is the amount of contamination they can leave in their wake when used. Wipers laden with microcontaminants will inevitably leave a trail of contamination on the cleaned surface. While tests for performance characteristics are relatively simple and well accepted, testing for microcontaminants released from wiping materials is far more complex. As critical levels of microcontamination become smaller, new test methods and techniques for measurement must continually be developed to accurately and realistically measure the contamination shed from wipers.



Laboratory orbital shaker used in sample preparation for particle testing.

Methods for testing wipers

The major contaminants released from wipers that can affect yield and reliability of semiconductor or data storage devices are physical debris, such as particles and fibers; nonvolatile residue extracted from the wiper when used with solvents; metals and metal ions; anions, which can act not only as conductors but also as corrosives; and the propensity of the wiper material to create and hold an electrostatic charge. Each of these factors needs to be evaluated and quantified for a true assessment of the contamination risk caused by bringing such products into the cleanroom.



An example of an electropherogram. The area under the peaks indicates the ion concentrations for each cation and anion.

Physical debris

First in order of significance is physical debris, or particles and fibers. The measurement of particles and fibers quantifies the physical contamination from the wiper's surface left behind after the wiper has been used. Particles are a microcosm of the surface chemistry of the wiper. They can be made of both organic and inorganic species and are therefore highly detrimental to many processes. In addition, microbial contamination or viable particles residing on wipers can be very deleterious because of their ability to multiply rapidly. Microbes such as bacteria not only have mass but also contain an array of life-sustaining chemicals that can be very damaging to semiconductor and other critical processes. Larger fibers shed from the wiper are easier to detect and eliminate, but because of their size, can cause damage over a much wider area.

The test for particles and fibers is somewhat complex but yields a great deal of information. The test method can be divided into two parts: sample preparation and metrology. The goal of sample preparation is to remove all particles and fibers which could be released from a wiper during normal to vigorous use. To accomplish this, the wiper must be placed under conditions where these particles and fibers will be released and easily captured. Water is an excellent medium for capturing particles; however, to more closely simulate conditions of use, the wiper is immersed in a clean tray containing water mixed with surfactant or isopropyl alcohol. This creates the low surface tension environment the wiper is typically exposed to during use, and allows the particles removed from the wiper to become suspended in the liquid. To maximize removal efficiency of the liquid, the tray is agitated for five minutes using a laboratory orbital shaker. The combination of immersing the wiper in a low surface tension liquid environment and then agitating the liquid allows for a significant release of particles and fibers from the wiper. The wiper is then removed from the tray and the liquid filtered through a microporous membrane filter. The filter will then be examined and the number of particles counted. Needless to say, a complete system blank must be performed before each test.

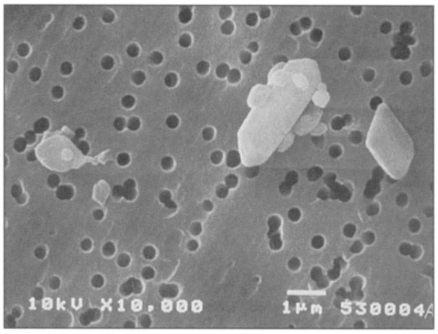
Scanning electron microscopy (SEM) is the metrology tool of choice for counting and analyzing particles, fibers and other matter released from wipers. Examination by SEM of the membrane filter prepared as described above yields a wealth of information. First, as opposed to other techniques for counting particles, SEM allows direct observation of the particles themselves. Since particles and fibers are clearly visible on the filter surface, they can easily be counted and analyzed. This technique is far more accurate than the use of laser-based liquid particle counters, which are designed to measure perfectly spherical particles under an idealized set of conditions. The SEM technique was developed because liquid particle counters lack the sensitivity and range to accurately measure particles and fibers released from wiping materials.[2]

In addition to counting, the metal content of particles can be determined under SEM using energy dispersive x-ray (EDX) analysis. This is an invaluable counterpart to ion measurement techniques which employ liquid extraction. Particles can also be identified through examination of their morphology. An additional benefit is that the SEM will detect both organic films and microbial contamination. Almost any matter removed from the wiper and collected on the filter will be detected and visually displayed on the SEM — the only exception being extremely volatile compounds that would evaporate under high vacuum. However, today's SEMs operate in a low-vacuum mode, which enables the detection and measurement of even these compounds.

In summary, SEM is a very powerful technique for detecting, enumerating and analyzing particles, fibers, and other matter captured during sample preparation. With SEM, we can paint a composite picture of the amount and type of contamination that will be released from a wiper during use.

Nonvolatile residue (NVR)

Nonvolatile residue can be extracted from a wiper when it is used with a solvent. This residue will remain on a wiped surface, leaving it coated with an organic film. Many wipers contain high levels of chemical additives to achieve specific performance characteristics. These additives, which are soluble in common solvents, are often deposited on clean surfaces that have been wiped down with the wiper. Fortunately, numerous techniques exist for detecting the presence of potential extractables.



Microporous membrane filter with collected particles viewed at 10,000x magnification using scanning electron microscopy.

There are several tests available for measuring and analyzing nonvolatile residue released from wipers in a given solvent. The simplest involves boiling the wiper in the solvent for five minutes, then evaporating the solvent and weighing the residue. Weighing must take place on a very sensitive analytical balance, as residue is often in the order of tenths of a milligram. A more extensive technique involves Soxhlet extraction, where the wiper is slowly extracted using condensed solvent vapor over a period of many hours. The residue can then be collected and weighed. With both techniques, residue can be analyzed and identified by means of infrared spectrophotometry. A final technique for establishing the presence of minute amounts of NVR is scanning electron microscopy (SEM). Once the wiper is extracted, the extract solution is filtered through a microporous membrane filter. Upon coming in contact with the filter surface, much of the nonvolatile residue precipitates and forms a film. The filter can then be viewed and analyzed under the SEM.

Metals and ions

Metals and ions can cause significant harm to processes in many ways. Metals in the form of particles can become inadvertent dopants or form conductive bridges across circuit lines. In addition, as with any particle, metal particles can interfere with photolithography processes, making their detection and analysis doubly important. Anions are not only conductive, but can cause corrosion in metalized layers. This is particularly detrimental because it is a reliability issue, and the device can fail long after final test. Both cations and anions can be released through particles or in moisture exposed to the wiper surface. Extraction of the wiper in liquid and subsequent measurement with analytical instrumentation will quantify the burden of ions on the wiper surface.

As mentioned previously, metals released from wipers in the form of particles can be detected and measured using SEM with energy-dispersive x-ray (EDX) analysis. But what about soluble ions on the surface of a wiper? Again, the technique for the measurement of soluble cations and anions has to be broken down into sample preparation and metrology. The sample preparation in this case is fairly straightforward. Ions are released from the wiper into water by immersing a known mass of wiper material into warm deionized water for 15 minutes. Once the wiper is extracted and the ions removed into solution, the concentration of each ion needs to be determined.

The sensitivity criterion for any technique used today should be in the low parts-per-billion (ppb) range, as this level of ionic concentration can be detrimental to many of today's semiconductor fabrication processes. Detection limits in the parts-per-million (ppm) range are no longer sufficient. Several techniques have the required detection limits. One stands out, however, as extremely versatile, capable of analyzing both cations and anions, and also providing detection limits down to parts per trillion (ppt).

This technique is capillary ion electrophoresis (CIE). CIE provides extreme sensitivity with low limits of detection, as well as the versatility to analyze an entire group of cations or anions in each analysis. CIE works by inducing ions to migrate through a narrow capillary under a strong electric field. The migration time of each ion is detected by indirect UV absorption, and the ionic concentration is displayed by peak area in a spectrogram called an *electropherogram*. Capillary ion electrophoresis is used in many laboratories and critical manufacturing environments today for ion analysis and is currently being proposed as an ASTM standard for ion testing.

Electrostatic discharge

Finally, the potential for wipers to generate an electrostatic charge is becoming a greater cause for concern. This is particularly true in the data storage industry, but it is also an important consideration for the semiconductor industry. A simple way to avoid this problem is to use wipers in the wetted state. The problem still exists for dry wiping, and therefore, measurements of electrostatic discharge potential for wipers have been developed.

With ESD becoming a more important contaminant, any material that has the potential to hold a static charge needs to be evaluated for that propensity. Evaluation tools are relatively simple and commercially available. The tests include surface resistivity, which measures the ability of an object to conduct electrical charge along its surface, and charge decay, which measures the time it takes for a known electrostatic charge applied to an object to diminish by 90 percent. Both tests should be conducted under conditions of controlled humidity and temperature. Through these measurements, materials can be characterized as insulative, conductive, or static dissipative. The ideal for ESD protection is for materials to be kept in a static dissipative range, where they will bleed off an electrostatic charge at a moderate and controlled rate.

Summary

This summarizes the current state of the art in testing of cleanroom wipers. As device geometries continue to shrink and contamination control becomes more critical, we can look forward to the challenge of developing more sensitive techniques to measure the potential contamination released from wipers and other consumable products. The test methods described here have been developed and tested on thousands of wipers over the past several years. More sensitive and relevant test methods are in constant development as the nature and quantity of contamination that can cripple a process becomes more elusive. Test method development is an ongoing enterprise and must strive to keep up with the ever more critical demands for contamination control in high technology industries.

References

1. Listed below are several sources of information on the performance characteristics of wiping materials:

i. "Evaluating Wiping Materials Used in Cleanrooms and Other Controlled Environments," IES Recommended Practice RP-CC-004.2, Institute of Environmental Sciences, Mt. Prospect, IL, 1992.

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2. H. Bhatacharjee, S. Paley, T. Pavlik, O. Atterbury, "The Use of Scanning Electron Microscopy to Quantify the Burden of Particles Released from Cleanroom Wiping Materials," Scanning, 15, 301–308 (1993).

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