

A hand holding a wooden mallet with the word "CLEANLINESS" written on the head. The background is a blurred document.

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For Cleaning Process Improvement

# VERIFICATION

## *An Introduction to Parts Cleaning Contamination Measurement*

*Dwight Beal*

Each year the need for stricter cleanliness levels for cleaned parts is discovered. One professional may work in the semiconductor industry, where critical dimensions are shrinking every hour, or so it seems. Another professional may work in the automotive industry, where it is being learned that the initial operation of an engine will determine its life expectancy.

In all cases, the tolerable level of contamination is constantly decreasing. The problem is, how to measure this contamination? With the exception of the semiconductor wafer, most contamination sensitive parts are complex in shape and therefore do not lend themselves to traditional scanning techniques; therefore, different methods need to be used when testing these items. Let's take a look at the three more common techniques currently used and discuss the advantages, disadvantages and benefits of each.

## Methods of Extraction

### High-pressure spray

In many cases, removing the contamination is accomplished by a simple spray wash. A solvent/aqueous solution is sprayed from a high-pressure nozzle onto the surfaces of the part. The effluent is captured as it runs off the part into a clean container.

This method is traditionally used when the contamination of interest is of relatively large size (>50 microns), or the size of the part is exceptionally large. There are two elements that must be carefully investigated in this extraction technique. The first is the cleanliness of the solution after it exits the spray nozzle. Even if exceptionally clean fluids are used, there are potential instances of receiving a contaminated batch from the supplier, or the delivery mechanics contaminating the fluid.

The second element of concern is the cleanliness of the effluent collection vessel. Checks of the cleanliness of test fluid must be made frequently enough to insure its adequacy for the test. The effluent collection vessel must be cleaned and checked before each test. The good news in this test is that the particle size is typically large and therefore it is relatively easy to clean the test apparatus to produce exceptionally low background values. The problem becomes that without some way of knowing about cleanliness excursions in these elements, parts could fail that are actually within specification.

Technique is another element of variation in the high-pressure spray method. As an example, suppose that one operator believes that the purpose of his/her job is to try to get as many particles off the part as possible. This individual painstakingly covers every millimeter of the part with slow deliberate strokes. Another operator believes that number of parts tested during the day is an important indicator of job performance. This individual attempts to cover every millimeter with rapid strokes, believing that as long as the entire part is wet, a representative sample of the particles will be collected. Both of these operators have valid beliefs.

The first operator will remove more particles from the part. The second operator will process more parts. As long as each operator maintains consistent technique, both will generate data that is representative of the cleanliness of the parts, in other words, when parts are dirtier than normal, both will show data with higher particle counts. The problem comes when trying to compare the data produced by the two operators.

In general, the data will be randomly mixed together making it less obvious that the differences can be attributed to operator technique. This, combined with statistical variation in the cleanliness of the parts themselves will create the appearance that the parts have a relatively wide variation in cleanliness. The fact that most of the parts are acceptable means that the upper control limit will necessarily be quite high. This may allow for parts that are actually outside of acceptable levels to be passed on to manufacturing as good parts.

One final limitation to this technique is measurement of cleanliness inside small openings and pathways. For instance, in automotive manufacturing, many paths for the lubricating oil are machined in the engine casting. It is not always possible to get a spray test method to extract the residual machine tailings from these pathways. Yet this is often the source of significant contamination that leads to catastrophic failure.

### Sloshing or Swirling

This method is typically used for smaller parts or to test containers themselves for cleanliness. To test small parts, a clean container is filled with a carefully measured volume of clean solvent/aqueous cleaning fluid. The parts are added, and the operator then swirls the container of fluid for a designated interval of time. The number of particles added to the testing fluid determines the cleanliness of the part.

To test the cleanliness of the container itself, the clean fluid is added to the container, the container lid is added, and it is rocked back and forth for a specified interval of time. To effectively quantify the cleanliness of the container, this testing method requires the operator to slosh the liquid onto all surfaces of the container without spilling any of the fluid in the process. Failure to do so could leave potentially damaging particles on the part surface resulting in a failed detection.

The sloshing/swirling method of extraction suffers from the same caveats as the high-pressure spray method. All of the test materials must be verified clean before each test. Because this method is often used for smaller particles, the cleaning technique must be more rigorous, and carefully followed if reasonable backgrounds are to be achieved (<1% of expected contamination by part). Allowances must be made for the

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increased level of operator involvement during the cleaning process in this case. Expect to double the time required for every increase in measurement sensitivity required. In other words, if it requires 10 minutes to clean prior to a test at 2-microns, expect 20 minutes for cleaning prior to a test at 0.5 microns and about an hour for cleaning prior to testing at 0.2 microns.

Once again, operator technique will influence the results. An operator with more vigorous sloshing/swirling technique will produce greater numbers of particles. In one application the slosh test has shown that no cleaning during manufacturing passed just as many products as ones that had been through three cleaning steps. In this particular case, one operator performed the test using consistent technique.

Other users of this method also have been known to standardize on one operator to perform the test. Both of these situations produce significant questions on the value of this method. If the test method cannot tell the difference between a part that has been cleaned and one that has not been cleaned, how can any useful decisions be made about the cleanliness of the part? If only one operator can perform the test, what happens to the data when that operator is no longer available? Answers to these questions must be addressed if this method is to become the primary qualification tool for the company products.

### Ultrasonic Extraction

This method can be used for almost all types, materials and sizes of parts, and cleanliness levels. The principle involved is to immerse the part into a bath containing the solvent/aqueous cleaning fluid. The bath has transducers bonded to the surface, which produce ultrasonic energy in the cleaning fluid.

The ultrasonic energy imparts significant cleaning power to the surface of the part, which is quite effective at removing even strongly bonded particles. Because the extraction force is constant, more reliable and repeatable particle removal can be obtained. This method is typically used in applications that require removal of smaller sized particles, or where the presence of contamination is more critical to the product's yield.

As with the previous two methods, the cleanliness of the test apparatus is critical. There must be a method for testing the cleanliness of the test container complete with its test fluid. An important addition is the need to make this background measurement after exposing the test container and fluid to the ultrasonic energy.

Simply rinsing or washing the container easily removes loosely attached particles, but ultrasonic energy will release the stronger bonds causing added particle loading to the test fluid. It is important to account for, or better yet, eliminate these additional particles to prevent falsely rejecting clean parts.

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By achieving a lower background the data will track the part's true level of contamination more accurately. While a subtle statement, this is actually very critical to the performance of this method. Inadequate background preparation will produce difficult to analyze results. Once again, you can expect a doubling of cleaning time as sensitivity to smaller sized particles is required.

A somewhat less obvious error in this test (and the slosh/swirl test above) is the time for extraction. Most ultrasonic generators come with timers to control the operating interval of the ultrasonic energy. The typical generator timer has a “turn past 5 minutes, then set time” operating style. The trouble is, most parts cleanliness tests use extraction intervals of <2 minutes that require the operator to manually time the generator.

Anything longer than 30 seconds seems to be beyond the attention span of a typical operator, and a conversation between coworkers, or other thoughts about the day will usually intervene - occasionally producing longer than expected extraction times. This variation will provide added variability in the data, increasing the standard deviation.

### Methods of Sample Analysis

After the particles have been extracted from the test part, they must be counted and sized, and the results recorded for analysis. Once again, there are several methods for classifying the particles.

#### Gravimetric Analysis

Gravimetric analysis requires the use of a previously weighed membrane filter. The test fluid is then pulled through the filter. The filter is dried, and re-weighed. The additional mass of the filter is presumed to be from the particles extracted from the part. Furthermore, it is assumed that the particles are of the same density and size distribution as the original tests that were used to set acceptable mass levels.

Changes in these variables will produce data that ranges from wider standard deviation values to completely useless. Predominantly, the variation should remain in the increased standard deviation category, but how does one determine the differences? Additionally, with each sample transfer, care must be taken not to contaminate the test fluid, or the membrane filter. Tests must be performed periodically to ensure that the drying process doesn't contaminate the test membrane. One careless action can ruin the entire test.

#### Optical Inspection

Optical inspection requires the use of a clean membrane filter. The test fluid is pulled through the filter. The filter is then placed on a microscope stage and inspected for particles larger than a specific dimension. Due to the magnification provided by the microscope, it is typical that only a small region of the filter is inspected.

In situations where parts are truly clean, there may only be a few killer particles anywhere on the filter. How can one be assured that the location of the killer particle is included in the inspection region? Problems also occur in this method when particles are not uniform in shape. Particles may be measured over the longest dimension, average size, length of x-axis, etc. Different inspectors may use different methods, or simply see the size differently resulting in additional data variability. Again, with each sample transfer, care must be taken not to contaminate the test fluid, or the membrane filter. One careless action can ruin the entire test.

#### Optical Particle Counter

An optical particle counter can also be used to sample the test fluid. Advantages of this method include rapid sampling capability, excellent sample-to-sample repeatability that results in confidence in each test, accurate sizing information and elimination of operator subjectivity and errors; however, optical particle counters are not an absolute measure of particle size.

They measure the equivalent optical size of the particle, reporting a value equivalent to a calibration particle. If the particle is similar to a fiber, in other words, long and thin, then orientation in the sample cell can produce different results. These limitations result in little to no variation in the test results, when compared to the possible variations produced during the extraction process.

## Efficiency and Accuracy

One of the common themes in each of the extraction and evaluation methods described is time and labor. An operator must be intimately involved in each step of the test. They must clean the sample test chambers, perform background checks, extract the particles, and measure the contribution by the test part.

As the cleanliness requirements increase, measurements at smaller particle sizes will be necessary, which increases the time required to clean the apparatus. Because of the labor and time requirement, these tests are limited to laboratory checks.

Parts are removed from the cleaning system and sent to the lab for testing. The results of these tests may not be available for a day to a week later, depending on workload in the lab. Indeed, many industries only use these methods when experiencing a major problem in yield for this reason. Some opt to skip the testing completely in favor of shotgun servicing of the cleaning equipment. The rationale is that by the time the results point to the actual problem, enough productivity will be lost to merit the more rapid, but expensive, equipment maintenance.

These limitations produce extremely expensive repercussions in the manufacturing process when parts must be recalled or re-examined to prevent reliability problems in the customer's hands. Also, at best, these methods offer a very small sample of the overall control in the cleaning process.

New technologies that help maintain high throughput make it possible to move testing out of the laboratory onto

the manufacturing floor. Many advantages are realized by performing the testing right in the manufacturing area, when the parts exit the cleaning system.

Foremost, contaminated parts can be eliminated from reaching final stages of manufacturing. This will reduce or eliminate product recall/rework due to contamination failures. Additionally, increased information is available about the performance of the cleaning system. Filter changes, bath changes, and major maintenance can be performed only when necessary, all of which increase the tool's up-time and utilization rate. Changes to the cleaning chemistry, or routine, can be immediately verified for benefit/detriment to the overall cleaning process. Permanently implementing only changes that benefit the cleaning process lead to better results in manufacturing.

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