Determination of residual particles on surfaces. An updated method for particle extraction using ultrasonics

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Abstract—In measuring particle counts using a laser LPC (liquid particle counter), it is important to use an apparatus with a very low background count. High background counts normally reduce the degree of confidence in the final count results. A study was performed to find the right material that generated the lowest background count when subjected to ultrasonics at different frequencies. Different common materials were evaluated such as borosilicate glass, quartz and stainless steel. We found that there was a significant difference between the results obtained from containers made from different materials. It is evident that the physical and/or the chemical properties as well as condition of apparatus surfaces are important determining factors in the background count. Ultrasonic frequency and power amplitude are also significant contributing factors. The data showed that electropolished stainless steel gives the lowest background readings at high ultrasonic frequency (132 kHz) and low power amplitude.

Keywords: Ultrasonics; particles; extraction; liquid particle counter (LPC); glass; quartz; borosilicate; stainless steel; acoustics; IDEMA standard; surface roughness; surface erosion.

1. INTRODUCTION

The use of ultrasonic cleaning for the removal of small particles [1, 2] from surfaces is a common practice in microelectronics, optics, disc drive, and semiconductor industries. The primary particle removal mechanisms involve powerful bursts of energy from millions of tiny cavitation implosions, which help dislodge particles by impacting the surface with shock waves. This powerful impact is accompanied by micro-streaming currents which produces a highly accelerated liquid that rinses away the loosened particles. The relative effects of these two mechanisms vary with varying the applied ultrasonic frequency. At lower frequencies (e.g., 40 kHz), cavitation is intense, while it becomes mild with increase of the applied frequency [2]. Hence, recommendations for proper harnessing of the ultrasonic field to achieve maximum surface cleanliness with minimum ero-

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sive damage must be based on evaluation and testing. The energy released from cavitation implosions is directly proportional to the radius of the generated cavities $r$, which in turn, is inversely proportional to the ultrasonic frequency $f$ used:

$$E_{\text{implosion}} \propto r \quad E_{\text{implosion}} \propto \frac{1}{f}$$

Surface erosion of delicate surfaces by ultrasonic cavitations is well known [3, 4].

It is obvious that if inadequate cleaning process parameters are employed, the desired cleanliness may not be achieved. Residual particle contamination on surfaces can degrade the performance, production yield and life expectancy of a product. Greater implications are expressed for the integrity of critical surfaces in nano-technology applications such as wafer fabrication, integrated circuits and hard disk drive industries. In the medical field, contaminated implants or devices can have grave health consequences.

Therefore, many manufacturers have instituted quality assurance procedures for cleanliness of components using liquid particle counters (LPCs) to ensure that their parts are clean to their standards and are not likely to adversely affect the finished product.

An accurate determination of surface cleanliness is crucial. Several methods and standard procedures have been developed to measure residual particles on surfaces after cleaning, including optical microscopy, surface scanners, scanning electron microscopy (SEM), atomic force microscopy (AFM) and LPCs. The most recent instrument is a portable particle counter for flat surfaces. It utilizes surface imaging and fast data processing and storage. Similarly to other scanners, multiple scans are required to monitor large surface areas [5].

Other techniques, such as scanning electron microscopy/energy dispersive X-ray spectrometry (SEM/EDAX), secondary ion mass spectrometry (SIMS), atomic emission spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS), are also used to determine the nature and composition of residues on a surface.

2. ADVANTAGES AND DISADVANTAGES OF USING A LIQUID PARTICLE COUNTER (LPC)

A LPC has a special advantage in that the residual particles on all surfaces, including internal surfaces of a component, can be evaluated in a single test, regardless of the part geometry. Determination of particle sizes and numbers of particles is relatively fast after the initial setup time. The other methods, although more informative with respect to the nature and composition of particles, are limited to flat surfaces and small surface area.

A main disadvantage is that it is an indirect method. This method involves ultrasonic extraction of particles from the component surfaces into a liquid medium [6], and counting of particles collected in the liquid medium using a laser sensor.
Also, a small number of new particles may generate from the original tested surfaces because of potential cavitation erosion. However, the latter can be greatly minimized by using high-frequency ultrasonics (>125 kHz) and satisfying the conditions of frictionless and uniform exposure of component surfaces to the particles dislodging forces.

3. THE IDEMA STANDARD METHOD [7]

To determine residual particles with nano- and sub-micrometer sizes the IDEMA standard method [7] calls for applying ultrasonic energy to a suspended part in a glass beaker in a specific volume of filtered deionized water for a specified time and then draw a sample and count the particles using an LPC at preset channel sizes.

The following standards are referenced as important guidelines in developing new procedures for the determination of residual particles by an LPC.

- ASTM-D 1193 “Specification for Reagent Water”
- ASTM-F 24 “Method for Measuring and Counting Particulate Contamination on Surfaces”
- ASTM-F 312 “Microscopically Sizing and Counting Particles”
- ASTM-F 575 “Particle Concentrations in Liquids”

The big challenge in applying the IDEMA standard is to find the ideal glass beaker that produces a low background count. Practically, it is a matter of trial and error. Even though when one is found, reproducibility of results can be of concern.

4. MATERIALS AND METHODS

4.1. Method for particle extraction with ultrasonics

The following procedure, that we developed, is an adaptation following the general standards. For the purpose of this study, no components were tested. Only different beakers were evaluated for their background particle count. The procedure entailed the following steps.

1. The deionized water was degassed and the DO (dissolved oxygen) was measured at 3.5–4.0 ppm and filtered prior to use in Step 2.

2. The test beaker was filled with 500 ml of filtered (0.2 µm) deionized water with very low particle background (< 5 particles/10 ml) and then subjected to high frequency ultrasonics for 30 s. The frequency used in this evaluation was 152 kHz (we found that a lower frequency of 40 kHz, as recommended in Ref. [7], gave consistently higher background particle counts).
3. Multiple samples (minimum of 3) were drawn. Particles were measured with an LPC and total particle count per sample between 0.5 and 2 µm was recorded.

4.2. Test apparatus

The test apparatus is shown in Fig. 1. The component to be measured must be suspended in solution to avoid friction with the container surfaces. The test container (1 l) is held suspended in the center of a large ultrasonic tank, normally 20 l in size.

4.3. Beaker preparation

Several beakers made from different materials were evaluated in this study. It was very important to prepare all the test beakers under the same conditions. A procedure was devised and applied to all four different kinds of beakers used in the evaluation. To ensure maximum cleanliness, the beakers were exposed to low and high frequencies as described below for an extended time (20 min).

1. Ultrasonic wash at 40 kHz, in 5% Chem-Crest 275 (highly alkaline detergent), at 65°C for 5 min.
2. Ultrasonic rinse at 40 kHz, in deionized filtered water, at 50°C for 5 min.
3. Ultrasonic rinse at 132 kHz in deionized filtered water, at 50°C for 5 min.
4. Ultrasonic rinse at 192 kHz in deionized filtered water, at 50°C, 5 min.
5. Dry in HEPA-filtered (0.2 µm) re-circulated, heated air at 85°C for 15 min.

It must be noted that all the preparations and evaluations were performed in a Class-100 cleanroom.

5. RESULTS AND DISCUSSION

Because of the inconsistencies and high numbers of background particles borosilicate glass (Pyrex) beakers were deemed not good enough for further consideration (Table 1).
Table 1. Results of beaker materials evaluation

<table>
<thead>
<tr>
<th>Beaker material</th>
<th>Average of total particle counts 0.5–20 µm (per 10-ml sample)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pyrex</td>
<td>28 883</td>
</tr>
<tr>
<td>18-8 Non-polished stainless steel</td>
<td>2092</td>
</tr>
<tr>
<td>Quartz</td>
<td>287</td>
</tr>
<tr>
<td>18-8 Electropolished stainless steel</td>
<td>29</td>
</tr>
</tbody>
</table>

All tests were conducted in a Crest Ultrasonics 132 kHz four-sided overflow tank which holds 20-l of deionized water. Each beaker (800 ml) was filled with 500 ml of 0.2-µm filtered deionized water and was ultrasonicated at 40% total power amplitude (0.6 W/cm²) for 30 s. An average of three tests was recorded. Reported numbers in this table do not include the background water count.

The second material evaluated was a 18-8 grained stainless beaker. It is known that the average surface roughness for deep-drawn stainless steel can be as low as 0.8 µm. The results were much better, but these did not meet the desired low level of background particles.

Fused quartz was evaluated next and the results were very promising. Fused quartz is mainly silicon dioxide while Pyrex glass contains about 80% silicon dioxide and other oxides like B₂O₃ (13%), Na₂O (4%) and Al₂O₃ (2.3%). The surface composition is obviously different and may contribute to the observed phenomenon.

The relatively high number of particles produced in the stainless steel beaker was attributed to its surface roughness. It is known that electrolytic polishing of stainless steel provides smooth, particle-free and corrosion resistant surface. The average surface roughness $R_a$ of deep-drawn stainless steel [8] is 3.2–0.8 µm and for electropolished stainless steel the $R_a$ is 0.8–0.1 µm.

Evaluation of an electropolished stainless steel beaker produced the best results among the materials tested as shown in Table 1. Electropolishing was done at a specialized industrial facility. The two stainless beakers used in the evaluation were made of 18-8 stainless steel.

The results shown in Table 1 clearly reveal large differences in the numbers of particles generated by different materials. The electropolished stainless steel was the best among all the materials tested. It gave the lowest number of particles that was deemed acceptable in this evaluation.

For long-term reuse of the same beaker, an extended test was designed to simulate a beaker that was in use for 50 consecutive tests (Figs 2 and 3).

It must be noted that the ultrasonic power amplitude utilized in this test (Figs 2 and 3) was about 4-times the power amplitude used in all other evaluation tests (2.4 W/cm² versus 0.6 W/cm²). For comparison, two materials were selected, namely the quartz and electropolished stainless steel beakers.
Figure 2. Results of extended test on quartz surface (particles generated in quartz beaker with 132 kHz, 100% power).

Figure 3. Results of extended test on electropolished stainless steel (particles generated in electropolished stainless steel beaker with 132 kHz, 100% power).
The results clearly demonstrate that the electropolished stainless steel produced much less number of particles and after 9 min it averaged about 400 particles. The quartz beaker produced >20 000 particles after 9 min and kept increasing steadily.

We also found that the number of particles generated was directly proportional to the applied power. In Fig. 4 the background particle count increased with the rise in power. Therefore, an excessive power in this extraction test may give false or misleading picture of surface cleanliness.

In conclusion, it is safe to state that electropolished stainless steel should be the material of choice for apparatus to be used in the extraction of residual particles after cleaning.

Since the ultrasonic power amplitude and frequency are important factors in the evaluation of surface cleanliness by the extraction method, it is critical to first explore the appropriate lowest level of ultrasonic amplitude to apply when the method is used to extract the cleaned surfaces of particular types of parts.

In general, it is recommended to use the lowest ultrasonic power amplitude that generates the lowest number of fresh particles from an absolutely clean surface. This same low energy should be sufficient to release residual contaminant particles.

6. CONCLUSIONS

The LPC extraction method is a practical and viable method for determination of residual particles on surfaces after cleaning. With respect to test container material with low acceptable background particle count, electropolished stainless steel is superior to other materials such as fused quartz and Pyrex glass.
The ultrasonic frequency, power and test duration must be evaluated and optimized for every material type for manufactured parts to be tested for surface cleanliness. For example, the test parameters determined for parts or components made of steel may not be appropriate for testing similar parts made of aluminum or plastic.

Acknowledgements

The author would like to thank Mr. Walter Pasicznyk and Mr. Maurice O’Donahue for their valuable contributions to this work.

REFERENCES