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The Particle Cleanliness Validation System

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THE PARTICLE CLEANLINESS VALIDATION SYSTEM

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Biography

Irving F. Stowers is a senior scientist at LLNL in the Laser Fusion Program where he has worked since 1975. His expertise includes laser and flashlamp damage to optics; design and construction of the Shiva, Nova, and NIF Laser Fusion Systems; fabrication of laser fusion targets; and the design and construction of ultra precision machine tools. He is the author of over 90 papers with more than 20 in the field of precision cleaning and contamination control. He received his BS in Mechanical Engineering from Northeastern University and his MS and ScD from MIT.

Douglas L. Ravizza is a senior technologist and the designer and builder of the PCVS system and has worked at LLNL since 1983. He specializes in the design and construction of complex electro-mechanical systems, especially those involving digital imaging processing.

Abstract

The Particle Cleanliness Validation System (PCVS) is a combination of a surface particle collection tool and a microscope based data reduction system for determining the particle cleanliness of mechanical and optical surfaces at LLNL. Livermore is currently constructing the National Ignition Facility (NIF), a large 192 beam laser system for studying fusion physics. The laser is entirely enclosed in aluminum and stainless steel vessels containing several environments; air, argon, and vacuum. It contains uncoated optics as well as hard dielectric coated and softer solgel coated optics which are, to varying degrees, sensitive to opaque particles, translucent particles, and molecular contamination. To quantify the particulate matter on structural surfaces during vendor cleaning and installation, a novel instrument has been developed to both collect surface particles and to quantify the number and size distribution of these particles. The particles are collected on membrane filter paper which is "swiped" on a test surface for a proscribed distance to collect sufficient particles to significantly exceed the cleanliness of the filter paper. The swipe paper is then placed into a cassette for protection from further contamination and transported to a microscope with x-y motorized stage and image analysis software. The surface of the swipe paper is scanned to determine both the background particle level of the paper, the cassette cover, and the portion of the paper which made contact with the test surface. The cumulative size distribution of the collected particles are displayed in size bins from 5 to 200 μm. The quantity of particles exceeding 5 μm is used to compute the IEST-STD-1246D cleanliness Level. Eight image analysis microscopes have been constructed for use with several dozen particle collection tools. About 30,000 cleanliness measurements have been taken to assure the clean construction and operation of the NIF laser system.

Keywords

Cleanliness, precision cleaning particles, MIL-STD-1246, digital image processing, cleanroom, particle size distribution.

Introduction

When the need to measure surface particle cleanliness was recognized in 1997, we surveyed existing techniques used in aerospace and microelectronic fabrication and determined that no existing technique satisfied all of our needs. We therefore developed the filter paper swipe technique to extract particles from surfaces, concentrate them one hundred fold, and then count the particles with a CCD camera equipped microscope. The particle collection technique can be seen as the logical extension of the "white glove" test. Details of the collection efficiency and issues associated with measuring particle cleanliness from ≤ Level 50 to ≥ Level 300 is described in sufficient detail that the technique could be reproduced by others interested in measuring similar cleanliness levels.

The NIF Laser and Optical Cleanliness

Particle and organic film cleanliness are important to the NIF laser system because they could lead to beam obscuration and scatter losses. The use of cleanrooms, as described in Federal Standard 209C[1], minimizes the settling of airborne particulate contaminants. The inside of the laser beamline is maintained at ≤ Class 1.

Large aperture optics on NIF have a cleanliness requirement of Level 50-A/10 as installed. Optical and structural surface cleanliness is further specified as initial cleanliness (immediately after cleaning) and as-assembled cleanliness. The NIF laser system particle cleanliness requirements are defined in Table 1. The particle cleanliness levels span from less than Level 50 to Level 300 (a dynamic range of 1,000:1 in particle concentration). Additional papers on the cleanliness issues associated with the construction and operation of the NIF laser systems can be found in the bibliography [2, 3, 4].

Table 1 Cleanliness Level requirements in the as-cleaned and as-assembled conditions for small optics (≤ 40 mm), large optics (400 mm), and structural surfaces.

	Surface cleanliness Level (as-cleaned)	Surface cleanliness Level (as-assembled)
Large optical surfaces	≤ Level 50-A/10	≈ Level 50-A/3
Small optical surfaces	≤ Level 100	≈ Level 100
Structural surfaces enclosing large optics	≤ Level 83-A/10	≈ Level 120-A/3
Structural surfaces enclosing small optics	≤ Level 300-A	≈ Level 300-A

Particle Cleanliness Levels

MIL-STD-1246C and IEST-STD-CC1246D[5] define surface cleanliness Levels for particles and thin-films. It has been found that the surface particles generally follows a $\log_{10}$ (cumulative concentration) versus ($\log_{10}$ particle diameter)^2 function. Each surface cleanliness Level is named for the largest particle size expected to be found in a 0.1 m^2 [1 ft^2] surface area. Thus a surface with a Level 100 distribution of contaminants, should have (on average) only one particle of ≥ 100 μm diameter on each 0.1 m^2 [1 ft^2] and an analytically defined number of smaller particles down to 1 μm diameter. For a particular cleanliness Level, the cumulative size distribution is given by the following equation and shown graphically in Figure 1.

$$\frac{Particles}{0.1m^2} = 10^{0.926\left(Log_{10}^2(Level) - Log_{10}^2(particle\ \ diameter\,[\mu m])\right)}$$

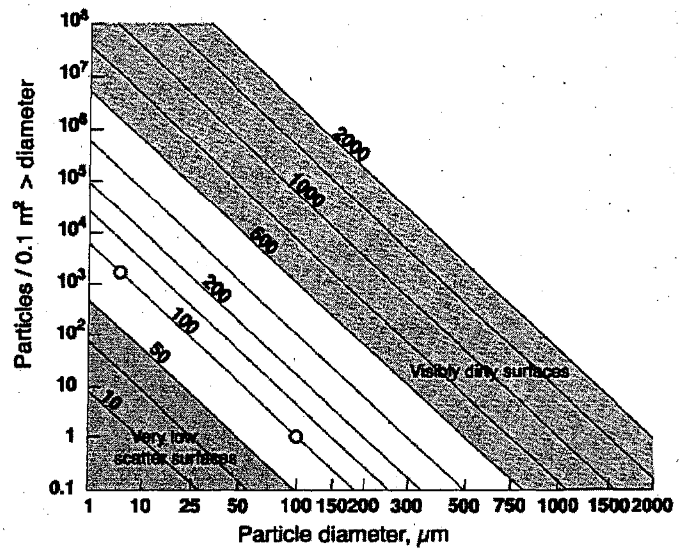

Figure 1 IEST-STD-1246D plotted on log-log^2 axes which results in a series of straight lines representing each cleanliness level. Cleanliness Level 100 allows only 1 particles / 0.1 m^2 [/ft^2] of 100 μm size or larger and simultaneously allows 1,785 particles / 0.1 m^2 [/ft^2] of 5 μm size or larger. These two points are shown as small circles (O) on the Level 100 cleanliness line.

Collecting Surface Particles

Surface Cleanliness Measurement

Although IEST-STD-1246D defines cleanliness Levels, it does not specifically define how to measure this quantity. Measuring the particle surface concentration can be done either directly or indirectly. Direct surface examination of very clean surfaces is difficult even with superior microscopic techniques. For example, a Level 100 surface has 1,785 particles (≥ 5μm) / 0.1 m^2 [/ft^2] or the equivalent of only 0.018 particles (≥ 5 μm) / mm^2. At this concentration it will require the examination of 56 mm^2 to statistically locate a single 5 μm particle. Since a count of only 1 particle is not statistically significant, at least 4 x 56 = 224 mm^2 will need examination to achieve a variance of 2 [$4^{1/2}$].

In contrast, indirect surface examination techniques, such as liquid surface flushing followed by filtration, will concentrate the particles onto a filter to be subsequently examined by a microscope. This process will achieve 50x increase in particle concentration and thereby 1) reduces counting time over direct surface examination, 2) improves counting statistics, and 3) increase particle concentration to significantly above the background contaminant level of the filter paper (the signal-to-noise level).

The authors have developed a dry surface wiping technique that utilizes a membrane filter to wipe [or swipe] a well defined area and then examine the filter under a microscope. Unlike the direct examination technique previously described [0.018 particles/ mm^2], the swiping distance can be adjusted (depending upon the cleanliness Level being examined) to achieve at least 1 particle (≥ 5 μm) / mm^2 in every microscope viewing area. Utilizing very clean filter paper with ≈ 0.1 particles (≥ 5 μm) / mm^2 and by adjusting the swiping distance from a few inches to several feet [see Table 2] it is possible to reliably measure particle cleanliness to below Level 50. The complete examination procedure and counting statistics are described in MEL98-012 Surface Cleanliness Validation by Swiping for NIF Components[6]. One advantage of the PCVS swipe tool compared to the ASTM F303 solvent flushing test is the ability to easily examine overhead and vertical surfaces.

Table 2 Swipe length needed to achieve a specific particle concentration ratio. In particular, the first column defines the particle cleanliness Level to be validated. The second column is the particle concentration at ≥ 5 μm diameter as defined in IEST-STD-1246D. The third column defines the minimum recommended swipe length. The fourth column defines the particle concentration ratio achieved with the minimum recommended swipe length.

Cleanliness Level	Particles / ft^2 ≥ 5 μm	Swipe Distance Inch	Particle Concentration Ratio
300	169,688	4	8:1
200	28,218	12	24:1
100	1,785	24	50:1
83 [Level 100 / 2]	908	48	100:1
50	166	144	300:1

Alternative Particle Collection Methods

ASTM F303 and F306[7] use a solvent to flush particles from 0.1 m^2 [1 ft^2] of surface area and then recovers the particles on a membrane filter. The particles are then manually counted with a microscope or with an image processing microscope. The particle concentration ratio is 54:1 assuming that 100 ml of clean solvent is used to collect particles onto a 47 mm membrane filter. This procedure is capable of measuring particle concentrations down to Level 86 assuming an initial filter cleanliness of ≤ 0.1 particle (≥ 5 μm) / mm^2, a signal-to-noise ratio of 3:1, and a fluid cleanliness of MIL-STD-1246 Level 50 [166 particle (≥ 5 μm) / 0.1 liter].

ASTM E1216[8] uses an adhesive tape to remove particles from surfaces and the tape is then microscopically examined to determine the particle concentration. Since the particle concentration ratio is 1:1, this method is only capable of measuring a surface cleanliness of Level 200 assuming a particle cleanliness of the tape of ≤ 0.1 particle (≥ 5 μm) / mm^2 and a signal-to-noise ratio of 3:1.

Pentagon Technologies[9] has developed the Q-III instrument that uses a moving gas stream to collect particles from a surface and deliver them to an airborne particle counter where they are counted. At its longest sampling time the instrument scans a 12 inch path that is 2 inch wide or 1/6 ft^2. This sampling distance allows the instrument to measure particle concentrations down to about Level 38 assuming a minimum count of 10 particles ≥ 5 μm and assuming an 80% collection efficiency.

Ernst[10] at Eastman Kodak has developed a sticky roller technique for collecting particles on surfaces. A 40.5 mm wide x 18 mm diameter roller is passed over a 0.1 m^2 [1 ft^2] area and then the particles are transferred to a sticky tape. The sticky tape is in turn examined using an image analysis microscope. The particle concentration ratio is 41:1 with samples taken from 0.1 m^2 [1 ft^2]. This particle collection procedure is able to measure particle concentrations down to Level 93 assuming an initial tape and roller cleanliness of ≤ 0.1 particle (≥ 5 μm) / mm^2 and a signal-to-noise ratio of 3:1.

The accuracy of all of these indirect particle collection techniques is highly dependent on their collection efficiency. Measuring collection efficiency is, however, not a standardized procedure and there are no standard dirty surfaces with which to repeatedly measure, using various particle collection methods, to generate statistics on particle collection efficiency.

Efficiency of Swipe Collection Method

Ideally a particle collection method should remove 100% of the particles above 1 µm from a surface. We've developed a procedure that repeatedly samples the same area of a surface and measures the particles removed during each swipe. The slope of the curve of particles/area, plotted on a Log-linear plot, represents the removal efficiency. As shown in Figure 2, a total of five separate locations were each swiped 10 times in sequence. For the first 4 swipes at each location, the swipe tool removed 85% of the surface particles during each swipe of a surface. After 4 swipes, the particle removal rate began to deviate from a constant percentage per swipe and after 6 swipes the particle concentration reached a steady-state level corresponding to Level 65 [0.004 particles (≥ 5 µm) / mm^2] which corresponds to the particle cleanliness level of the swipe paper.

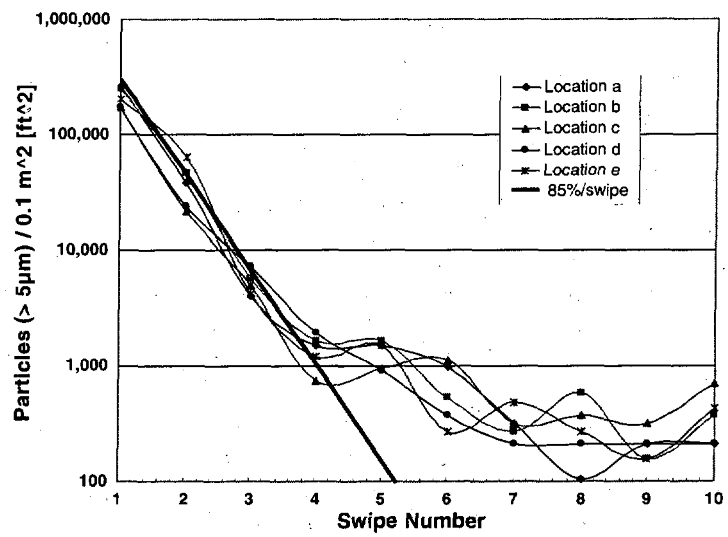

Figure 2 Swipe removal efficiency can be estimated by repeatedly swiping the same location and counting the swipe paper. During the first 4 swipes of the same surface, the particle removal efficiency remained constant at 85%.

The Swipe Tool Design

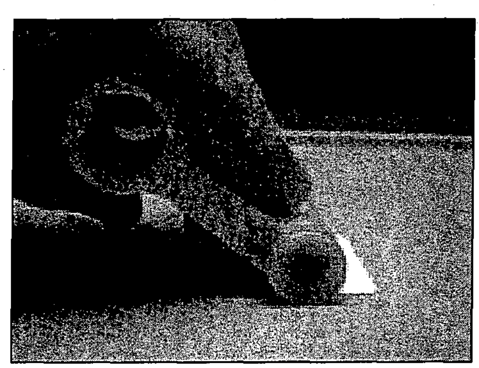

Figure 3 Photograph of swipe tool and swipe paper. The cellulose based swipe paper is available from two commercial vendors.

The swipe tool, in its third generation design (see Figure 3 and Figure 4), consists of a Delrin plastic swipe paper holder, a clamp to firmly hold one end of the swipe paper, an elastomer cushion that applies a uniform pressure to the back of the swipe paper, and two rollers that prevent excessive force from being applied to the swipe paper which might lead to tearing of the paper. A vertical force of 0.5 kg [1 pound force] is sufficient to compress the elastomer sponge and efficiently collect particles from relatively smooth surfaces. The preferred surface finish should be ≤ 1.6 μm [63 microinch] however, surfaces with twice this roughness can be swiped without damaging the surface of the swipe paper.

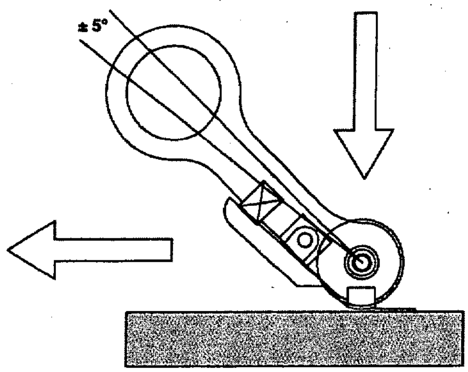

Figure 4 Outline of swipe tool indicating the small square sponge that backs-up the swipe paper. The swipe paper is held in place by a clamp. The swipe tool is held at 45° and gently rocked up and down as it is drawn across the surface. The downward force is about one pound which is regulated by rollers on each side of the swipe tool and the hardness of the sponge.

Swipe Paper

The swipe paper is a commercially available cellulose membrane filter that has been cut to a 2 inch x 1.5 inch shape. Although most membrane filter paper is not purchased to a surface cleanliness specification but rather to a pore size specification, the paper has been found to have a very repeatable cleanliness from lot to lot and between manufacturers. Typically, the paper has fewer than ≤ 0.1 particle (≥ 5 μm) / mm^2 which is equivalent to Level 153. Interestingly, it is possible to use this paper cleanliness level to verify the particle cleanliness of surfaces up to 50 times cleaner because of the particle concentration process inherent in swiping a surface.

Swipe Cassette

The particle collection procedure consists of placing a swipe paper into the swipe tool, swiping (wiping) a surface for a defined swipe length, placing the swipe paper into a clean protective cassette, counting the particles, and estimating the background contaminant level of the swipe paper with the PCVS microscope described below.

The swipe cassette [see Figure 5] is an injection molded polycarbonate housing and cover designed to protect the swipe paper from further contamination and allows the paper to be examined through the transparent cover.

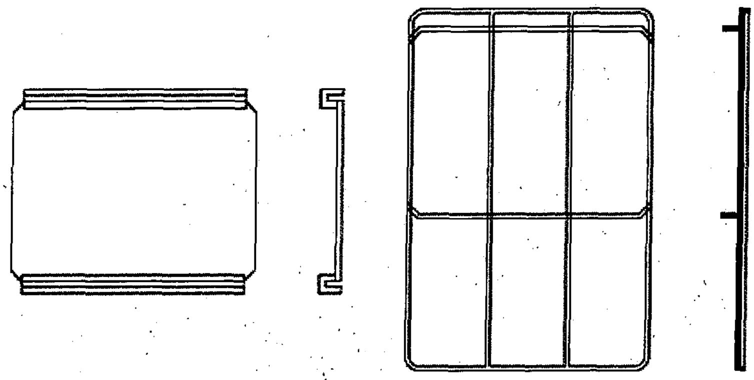

Figure 5 Schematic of swipe cassette. The cover protects the swipe paper from environmental contamination and holds the paper to maintain a flat field-of-view for the microscope.

Reading the Swipe Paper

PCVS Microscope Design

The PCVS microscope is conventional in concept but unconventional in implementation. Its purpose is simply to repeatedly examine many small portions of the swipe paper surface and count all particles ≥ 5 μm in equivalent diameter. The instrument must also function in two distinct modes; 1) as a research tool where the precise cleanliness of the sampled surface is needed as part of an investigation to correlate cleanliness with optical damage, and 2) as a production QC tool where it is simply necessary to verify (or validate) that a surface is cleaner than a predetermined surface cleanliness level. These two modes are accommodated by adjusting the length of the swipe as well as adjusting the number of images captured on each swipe paper.

The microscope is equipped with an x-y motor driven stage to move the cassette beneath a 6-power objective lens. Each captured video frame is 640 pixel x 480 pixel and represents approximately 1 mm^2. The particles removed during the swipe collection procedure creates a band that is roughly 12 rows (≈ 12 mm) wide by 50 columns (≈ 50 mm) long and could seemingly require 600 video images to be captured and processed. However, additional rows of images are required as the physical centerline of the paper may not coincide with the centerline of the x-y stage due to minor operator misalignments. Also, an additional eleven rows are captured to measure the cleanliness of the un-swiped portions of the paper. This allows the swipe paper background cleanliness level to be monitored and subtracted from the portion of the paper containing the collected particles, thereby significantly improving the counting accuracy. Despite the large number of video images needed to count the particles on a swipe paper, the scanning process is accomplished in approximately 5 minutes using custom software written using LabView[11].

Image Analysis Procedure

After each image is captured it is processed 1) to enhance contrast by performing a background subtraction, 2) converted to a binary image by thresholding the gray-scale image, 3) then particle outlines are closed 4) donut images are filled-in, and finally 5) pixels within an enclosed area are counted and converted to an equivalent circular diameter. Three enclosed pixels are needed to identify a 5 μm diameter particle.

After all images from a single swipe paper are counted, the number of particles are sorted into bins of 5 μm to 200 μm in 5 μm increments. The quantity of particles exceeding 5 μm is used to compute the IEST-STD-1246D cleanliness Level. The other 19 bins above 5 μm are not specifically used to compute the cleanliness level but are preserved along with the swipe description information in a database record formed after each swipe cassette is read. The measured size distribution rarely follows the exact shape of the MIL-STD-1246 size distribution due to the source of the contamination, as will be discussed in the following section.

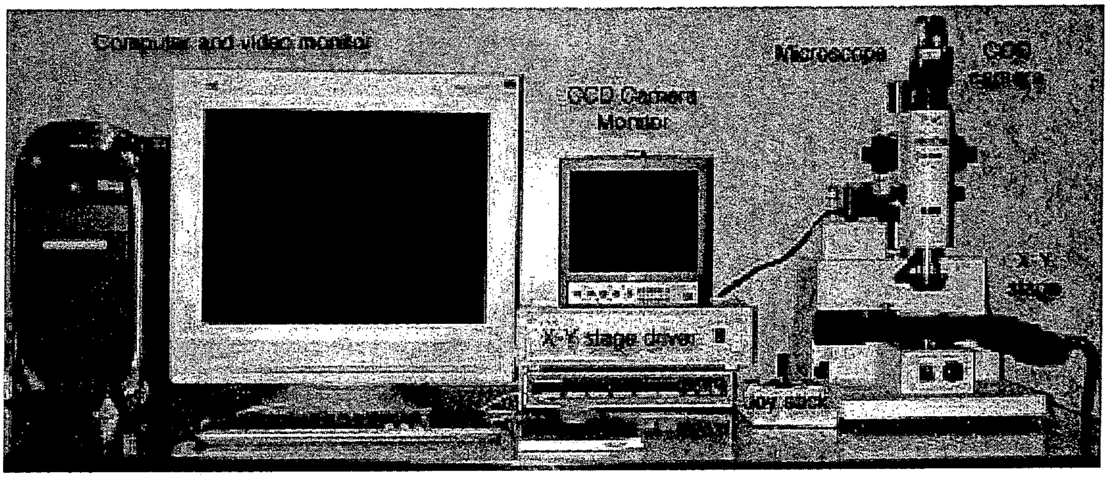

Figure 6 Illustration of the PCVS system. The custom assembled system includes a microscope (right) with 6-power objective lens and motor driven x-y stage. The small video monitor displays the image from the CCD camera whereas the large monitor displays the digitally processed image and provides the user interface for the PCVS instrument.

Particle Size Distribution Compared to IEST-STD-1246D

The data in the 20 particle size bins collected from each swipe can be used to compare with the size distribution given by IEST-STD-1246D. Figure 7 shows 20 size distribution curves selected from our database of nearly 30,000 swipe readings which were collected during August 2000. The readings were selected randomly but in the range between Level 83 and Level 100 and represent surfaces immediately after precision cleaning using high pressure DI water. The curves indicate that nearly all of the surfaces show fewer larger particles than would be expected from the IEST-STD-1246D curves. This finding is similar to what Welker[12] has observed; and, it is generally expressed as an increase in the slope of the curve above the value 0.926 found in the Section entitled: Particle Cleanliness Levels.

Welker[12], for example, reported using undulation, low pressure spray, and ultrasonic agitation to remove particles from surfaces and then counted the particles using a liquid particle counter. He found that the nature of the size distribution depended on the material being tested and on the precise particle removal technique. In general, undulation gave a coefficient slightly larger [typically 1.0] than the value of 0.926 found in IEST-STD-CC1246D, low pressure spray resulted in a higher value [typically 1.33], and ultrasonic agitation resulted in an even larger coefficient [typically 1.63].

In contrast, Tribble[13] found that the average IEST-STD-CC1246D coefficient measured at several aerospace cleanroom facilities was 0.383 for surfaces contaminated by airborne settling. A coefficient smaller than 0.926 indicates a higher number of larger diameter particles than predicted by IEST-STD-CC1246D. Tribble further states that the normal 0.926 coefficient is more applicable to precision cleaned surfaces where cleaning processes are more effective at removing larger size particles.

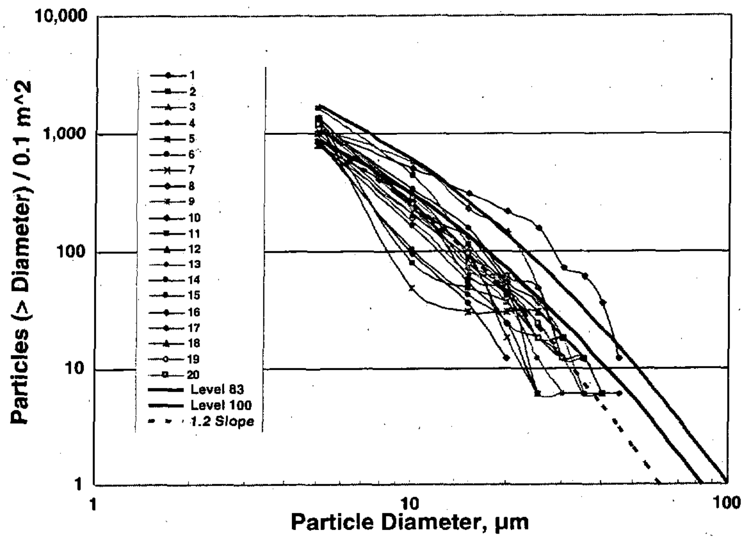

Figure 7 Size distribution of 20 randomly selected swipes from the PCVS database. The upper solid curve (red) represents Level 100 whereas the lower solid curve (blue) represents Level 83 (Level 100/2). All of the surfaces were precision cleaned before the swipe measurement was taken. The data show a higher slope than the value of 0.926 found in IEST-STD-CC1246D and a value of 1.2, shown as the green dashed line) appears to fit the data. This increase in slope, is typical of precision cleaned surfaces where the cleaning processes are more effective at removing larger particles.

Conclusions

To quantify the particulate cleanliness of structural surfaces during vendor cleaning and installation, a novel instrument has been developed to both collect surface particles and to quantify the number and size distribution of these particles. The instrument has sufficient collection efficiency and signal-to-noise ratio to reliably measure particle cleanliness from below Level 50 to above Level 300, a dynamic range of 1,000:1. The system is composed of a simple "swipe" collection tool and a microscope equipped with a CCD video camera connected to a custom programmed video image processing system. At this time, it has been used to make over 30,000 particle cleanliness measurements.

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