HOW PRODUCTION TECHNOLOGIES INFLUENCE SURFACE QUALITY OF ULTRACLEAN GAS-SUPPLY EQUIPMENT: INSPECTION METHODS FOR SURFACE EVALUATION

(Third of four parts)

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As linewidths continue to shrink, defects are caused by ever-smaller particles and amounts of organic and inorganic contamination. The monitoring, measuring, and identification of surface impurities has thus become an essential element of semiconductor technology. The particular technique used is dependent on the type, site, and time of the inspection. Specific considerations must, for example, be made in each of the following test situations:

- Product control at the component manufacturers (according to order specifications).
- Acceptance/product-entrance testing of individual component parts.
- Acceptance testing of the supply system.
- Process monitoring.
- Inspection of the end-product (chip).

In recent years inspection techniques have been further refined and developed, particularly for process monitoring and for the final inspection (function testing) of component parts. However, there remain hidden sources of contamination that cannot be detected and eliminated through the inspection process. Preventive measures must therefore be taken, with manufacturers' quality assurance resources extended to allow for these measures.[9,10,22,31]

This article, the third in a series on ultraclean gas-supply systems, is concerned with the inspection methods for assessment of surface quality (in terms of cleanliness, structure, and the like) in such systems.

INSPECTION PROCEDURES

To achieve a high level of surface quality, it is necessary not only to define requirements for the component but also to establish suitable inspection procedures (Tables I and II). With any quality assurance procedure, the goal must be to achieve the maximum quality with the minimum possible effort. While accurate measuring procedures are available for the purity verification required in semiconductor technology, these pro-

Measured value/ size	• Particle generation. • Water-soluble contaminants. • Surface roughness. • Other.
Precision/unity of measurement	• For example, limit value per surface, volume, or weight.
Frequency of measurement	• Single measurement. • Continuous individual measurements. • Random sample testing.
Test point	• Type and shape of component.
Time of measuring	• Previous component. Manufacturer's qualification. • On-line manufacturing process-monitoring acceptance. • Testing at manufacturer's premises. • Entrance control at user's premises.

Table I: Criteria to be considered when establishing inspection procedures for ultraclean gas components.

Prior unique production monitoring	• Destructive testing (e.g., REM, AES, SIMS). • Dissolving test. • Gas-retention properties. • 3-D reproduction. • Particle measuring. • Chemical analysis.
Continuous production control	• Electrolyte monitoring (chemical analyses, conductivity, etc.). • Tool monitoring (microscope). • Auxiliary equipment monitoring (chemical analyses). • Cleanroom (particle measurement). • Component (roughness).
Final inspection	• Particle measurement. • Leakage rate.

Table II: Suggested framework for inspection procedures.

Criterion	Test Procedure
Material purity	• Chemical analysis. • Metallographic section. • SEM of electropolished test sample.
Surface structure	• SEM. • Brush analyzer (Ra, Rz, steepness, 3-D reproduction, etc.). • Laser-optic probe (Ra, Rz, 3-D reproduction). • Interference-measuring instrument.
Water-soluble contaminants	• Rinsing test + ion chromatography. • Mass spectrometric measuring methods (e.g., SIMS [secondary-ion mass spectrometry]).
Particles	• SEM. • Replica + SEM. • Membrane filtering + microscope/SEM. • Laser-optic particle counter.
Particle identification	• SEM + EBM (electron-beam microprobe). • Electron spectroscopy.
HC residues	• Coulometry. • SIMS. • AES (Auger electron spectroscopy).
Gas-retention properties	• Gas chromatography.

Table III: Testing procedures used most frequently.

cedures are both elaborate and expensive and are thus not useful for continuous on-line monitoring or acceptance testing. Also, elaborate tests can be superfluous if comprehensive tests have already been carried out and if verification has been drawn up during the manufacturer's qualification sequence.

It is possible to greatly reduce and simplify the process of acceptance testing. As purity requirements become more stringent, manufacturers can build minimal additional tests on to their existing procedures for revealing and eliminating the sources of defects. The final quality of the component is verified by a documented inspection that follows external rules and specifications. Because such a final qualification should be performed only once, at this point more elaborate comprehensive tests can be carried out or verification drawn up.

Table III lists the most important acceptance-testing procedures available. For comparable and consistent measured values to be obtained, precise parameters must be specified (e.g., sequence of measurement, length of measuring time, measured length, frequency of measurement/mean value formation, and measurement sensitivity).

MEASURING SURFACE ROUGHNESS

Brush Analyzer. Measuring surface roughness is one of the most important aspects of surface-quality testing.

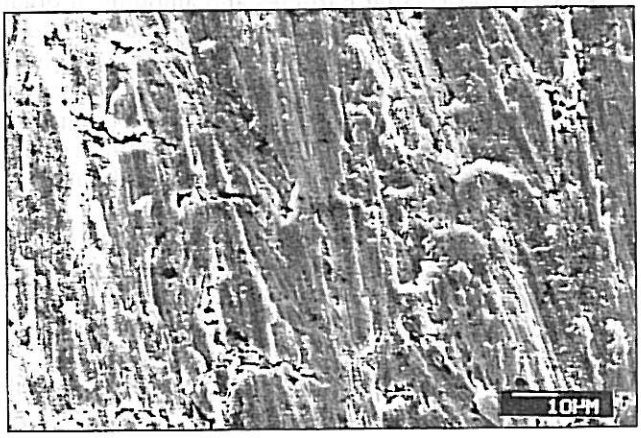

Figure 1: Cold-rolled surface with clearly marked microimperfections (roughness value: Ra = 0.24). (Measuring instrument: Hommel tester.)

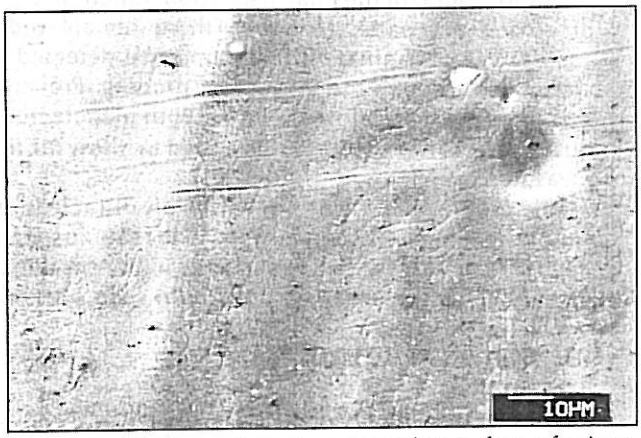

Figure 2: Electropolished surface that is free of microdefects (Ra = 0.45). (Measuring instrument: Hommel tester.)

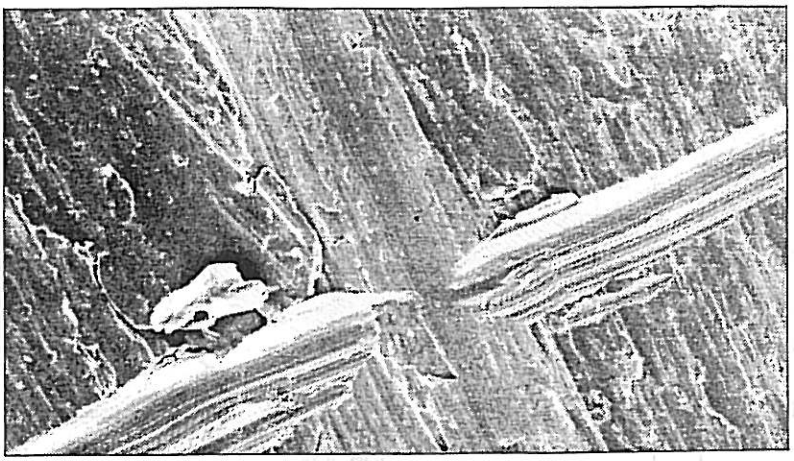

Figure 3: Surface damage caused by a defective measuring probe of a brush analyzer.

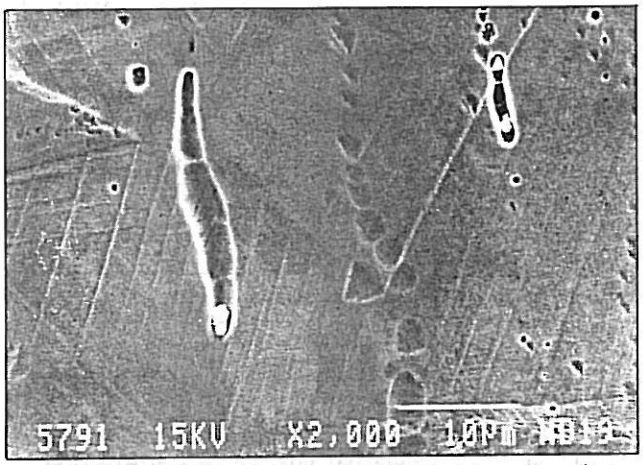

Figure 4: SEM photograph showing segregation lines exposed by electropolishing.

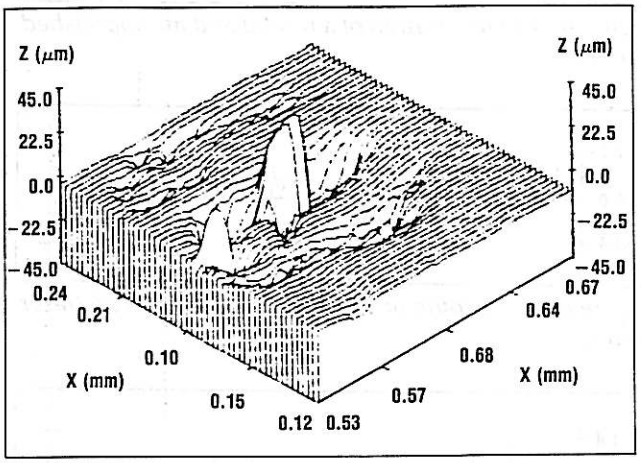

Figure 5: 3-D representation of surface shown in Figure 4, with defect sites clearly visible.

The roughness dimensions can be measured and recorded simultaneously with instruments that are transportable, economical, and easily operated. However, the reliability of these measurements leaves much to be desired. For one thing, microimperfections (e.g., exposed nonmetallic inclusion particles) are not reliably recorded. In an extreme case, this can lead to the acceptance of a surface that is full of serious microdefects (Figure 1), while a surface that is homogenous and free of microimperfections is rejected (Figure 2). The reason for this is that the probe tip of the brush analyzer is relatively coarse (diameter = 5 μm),[36,37] and as a result the sensitivity is low. Also, local damage to the surface can occur if surface-roughness measuring is performed incorrectly (Figure 3).[38]

Laser Measuring Instruments. A considerably better picture of the surface topography can be obtained with a laser measuring instrument, which provides a 3-D representation.[36] The high optical dispersion can permit detection and identification of, for example, nonmetallic inclusions exposed by electropolishing. Figures 4 and 5 show, respectively, an SEM photograph and a 3-D laser-scanner illustration. The disadvantage of the laser system is that the relatively large size of the probe prevents direct measuring of inner surfaces, so the component can only be tested destructively (as in SEM). Therefore, the 3-D representation is best used in conjunction with the SEM photograph in preliminary component qualification to provide comparative evaluations of larger surface areas.

Figure 6 shows a mechanically micropolished surface, and Figure 7 shows such a surface that has subsequently been electropolished. In each case, comparison of the 3-D reproduction with the true surface profile and roughness reading shows that the roughness measurements bear little relation to the true quality of the surface (Figures 8–11).

If different measuring instruments are used with the same test sample, different readings will be obtained (Table IV).[39] The higher dispersal of the laser optical measuring device (size of measuring laser beam = 1 μm) is clearly noticeable. The measuring parameters, which are adjustable according to each instrument, also play a part. To keep within reproducible limit values, such dimensions as forward speed, test length, and measuring frequency must be sacrificed for surface-roughness control.

IDENTIFYING AND ANALYZING SURFACE CONTAMINANTS

Dissolving Test. Dissolving tests, which take over where SEM stops (see next section), can be used to identify monolayers of water-soluble contaminants on a surface. This procedure is particularly important for the detection of dopant poisons. The water-soluble contaminants are dissolved in deionized water, and the ions are subsequently determined chromatographically. The dissolving treatment is done at 60° to 80°C.

Investigations have shown that a single dissolving treatment does not suffice to record all contaminants

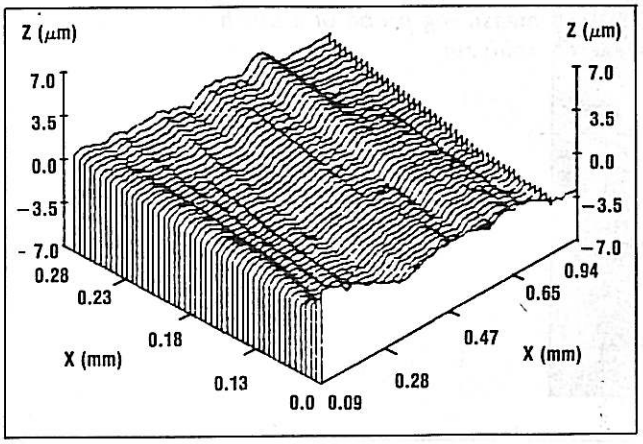

Figure 6: 3-D illustration of a turned and micropolished surface.

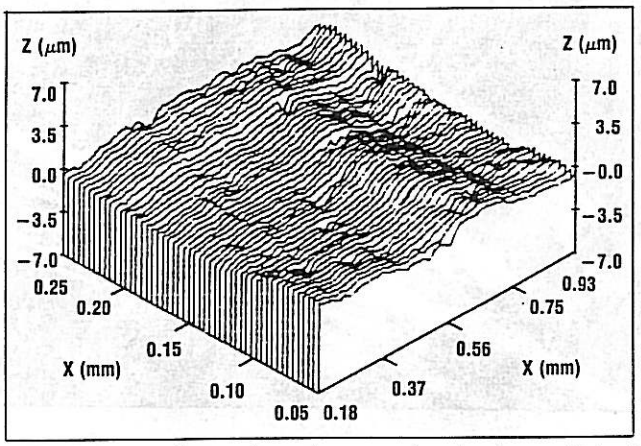

Figure 7: 3-D illustration of a turned and micropolished surface that has also been electropolished.

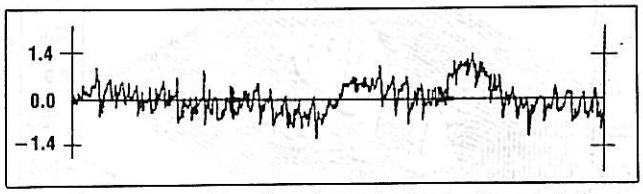

Figure 8: True profile of surface shown in Figure 6 (laser scanner).

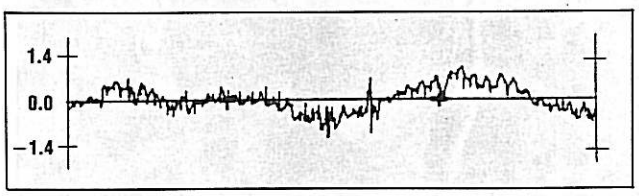

Figure 9: True profile of surface shown in Figure 7 (laser scanner).

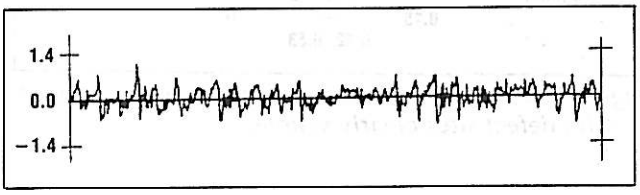

Figure 10: Surface roughness of surface shown in Figure 6 (laser scanner). Roughness measurement: Ra = 0.23.

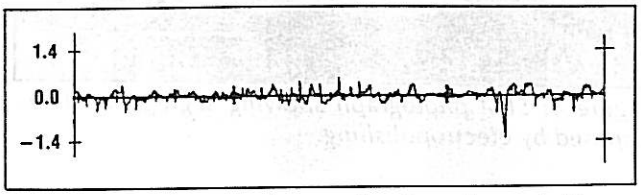

Figure 11: Surface roughness of surface shown in Figure 7 (laser scanner). Roughness measurement: Ra = 0.12.

Surface Treatment	Brush Analyzer				Laser Measuring Instrument	
	1		2		3	
	Ra	Rz	Ra	Rz	Ra	Rz
Turned	0.69	3.50	0.42	2.3	0.84	8.75
Turned + micropolished	0.57	3.22	0.52	2.8	0.92	9.56
Turned + micropolished + electropolished	0.12	0.49	0.43	2.1	0.78	10.96

Table IV: Surface-roughness measurements obtained using brush analyzers and laser measuring instruments from various manufacturers (Ra and Rz in μm).

Table V: Measurements obtained from dissolving tests of differently treated inner surfaces of valves. The values show the soluble contaminants that can be recorded at every step of immersion.

Valve Treatment	Dissolving Treatment (μg anions/valve)				Specific Surface Contamination (μg/cm²)
	1 hr	2 hr	3 hr	Total (1+2+3 hr)	
Normal processing	28	35	6	69	1.5
Electropolishing + dissolving	5.5	1	<0.1	6.5	0.14
Electropolishing + ultrasonic treatment	0.4	<0.1	<0.1	0.4	<0.01

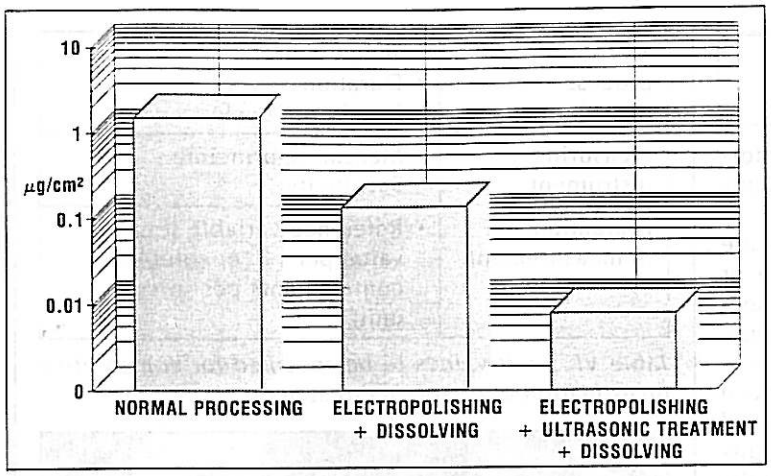

Figure 12: The influence of electropolishing and final cleaning (acid treatment followed by DI water rinsing and additional ultrasonic treatment [US + dissolving]) on surface purity (monolayers of soluble contamination in µg/cm²).

reliably. If salts that are not easily dissolved are present (e.g., phosphates and calcium sulphates), the rinsing treatment must be continued until no more ions are dissolved. Investigations have shown that three separate 1-hour dissolving treatments are sufficient to capture all the ions from high-purity surfaces (Table V). Table VI shows the limit values that must be specified for comparative investigation. Figure 12 shows the effects on surface purity of normal polishing, electropolishing plus dissolving, and electropolishing, ultrasonic treatment, and dissolving, respectively.

Scanning Electron Microscopy. Scanning electron microscopy (SEM) has become one of the most important methods of surface examination, since it shows the true picture of the surface structure and can disperse this up to the size range of the material structure.[37] When SEM is used in combination with energy-dispersive analysis, it is possible to identify and analyze particles down to 0.1 µm in size (Figures 13 and 14).

As noted earlier, SEM has reached its limits when contaminant monolayers must be identified, and mass spectrometric measuring procedures must be brought in. If contaminants appear in higher local concentrations, however, SEM can recognize them (Figures 15 and 16).

A disadvantage of SEM is that larger test pieces or inner surfaces can be tested only by destructive means. By making replicas (negative impressions of the surface), it is possible to illustrate the structural frameworks of

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these surfaces nondestructively; however, such information as the type and size range of contaminants becomes lost.

Mass Spectrometric Measuring. These methods are used to analyze surface deposits that are in one or relatively few monolayers (nm range).[37,40] Auger electron spectroscopy (AES) and secondary-ion mass spectrometry (SIMS) are the most commonly used procedures, although in both procedures inner surfaces can be tested only by destructive means. Also, because of the instruments' high sensitivity, readings can be distorted when cutting work is carried out prior to the preparation of samples. For these reasons, AES and SIMS have not gained general acceptance as methods of examining components.

Hydrocarbon Measuring. Hydrocarbon (HC) measuring can be used to test the surface for hydrocarbon compounds, such as greases, oils, residues from organic solvents, and the like. Mass spectrometry or coulometric measuring methods can be used, with the same limits valid as with AES and SIMS. Here, too, measurements taken from the component are meaningless; despite the effort involved, the results do not bear weight because of the many influencing factors. HC measurement is suitable for controlling or optimizing cleaning procedures; test samples specially adapted to the testing methods can be used.

Measuring process (dissolving)	• Temperature. • Duration. • Number of cycles.
Measuring instrument	• Measuring principle. • Sensitivity.
Evaluation of measurement	• Reference variable (total value per valve; soluble contaminants per surface unit).

Table VI: Limit values to be specified for comparative investigation of dissolving treatments.

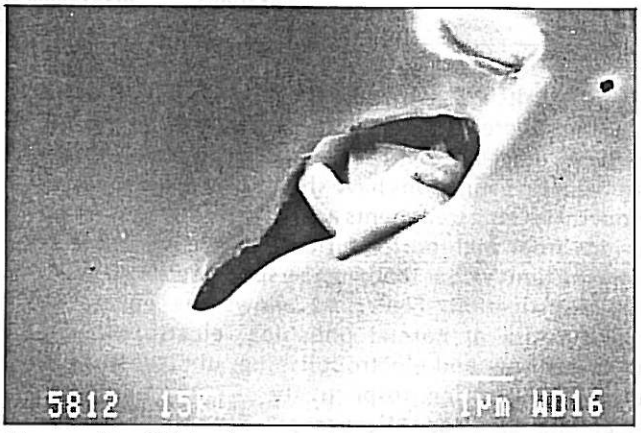

Figure 13: SEM photograph showing a nonmetallic inclusion particle, 1–3 μm diam, exposed by electropolishing (8000×).

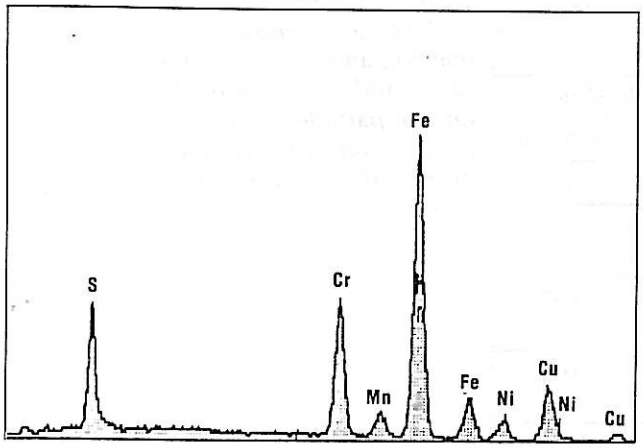

Figure 14: Energy-dispersive analysis of the nonmetallic inclusion particle shown in Figure 13. It can be concluded that we are mainly dealing with manganese and copper compounds.

Figure 15: SEM photograph showing residue of cleaning agents left on a surface following electropolishing (2300×).

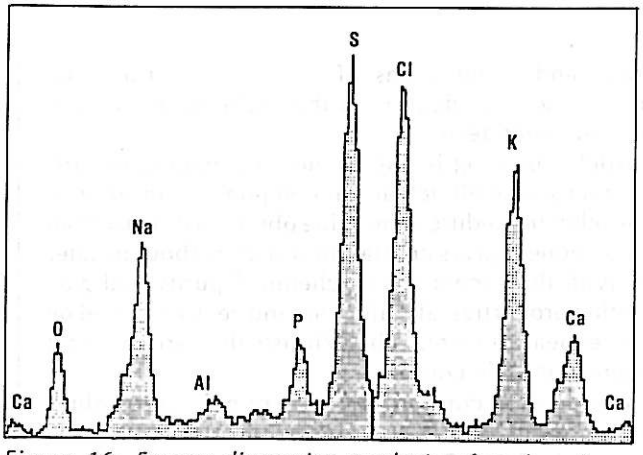

Figure 16: Energy-dispersive analysis of surface layer depicted in Figure 15. The chemical values (phosphorus, sulphur, chlorine, and calcium) indicate that the layer is residue from cleaning agents (electrolytes, water).

Test equipment	• Filter (type, number, filter capacity/retention property, and arrangement). • Tubing (section, geometry, rigid or flexible). • Length of tubes (from filter to measuring instrument). • Connection of test component (fittings). • Inner-surface quality.
Measuring parameters	• Medium. • Flow rate. • Pressure. • Test volumes. • Duration of test. • Frequency of testing. • Test procedures (prior rinsing, background measuring, load cycles, etc.).
Test evaluation	• Reference variables.
Measuring instrument	• Measuring principle. • Limit values. • Detection sensitivity.

Table VII: Variables that influence particle-counting results.

Flow rate	5–100 L/min
Test pressure	0.2–150 bar
Sensitivity of measuring instrument	0.02–0.2 μm
Medium	High-purity inert gas to toxic/corrosive process gas mixtures
Filtering	Low-pressure membrane filter to high-pressure ceramic filter

Table VIII: Current ranges of limit values for particle counting during component testing.

MEASURING GAS-RETENTION PROPERTIES: GAS CHROMATOGRAPHY

The gas-retention property of a surface or of a whole component can be determined using gas chromatography. During this procedure the length of time needed to exchange one gas for another is gauged. This measurement depends on the following variables:

- Type of gas measured.
- Gas pressure.
- Flow speed.
- Proportion of component volume to gas throughput.
- Frequency of measurement.
- Limit values of contaminants.

With these variables, the component influences of wetted inner surfaces, dead volume, and surface quality can be recorded integrally.

PARTICLE COUNTING

Particle counting has been thoroughly reported on in many publications.[41–51] However, the reports have been concerned mainly with cleanroom inspection, filter

Measured Value Test Pressure: 135 bar Medium : Argon	Flow Rate			
	30 L/min		40 L/min	
	Particle Size		Particle Size	
	>0.1 μm	>0.2 μm	>0.1 μm	>0.2 μm
Background	6.0	1.6	6.0	1.6
Outset measurement (test duration: 1 hr)	1.8	<UG	12.2	1.0
After the first 5 cycles (test duration: 1 hr)	20.0	9.0	3.1	<UG
After the second 5 cycles (test duration: 1 hr)	16.5	7.8	3.8	0.2
After 50 cycles (test duration: 20 hr)	5.3	1.2	2.8	1.2
After 50 cycles (last hr)	0.3	0.0	5.9	4.3

Table IX: Influence of load cycling (i.e., complete opening and shutting of the valve) and of flow-through rate on particle release (mean value [particles/cu ft] measured on a fully opened valve).

testing, and comparisons of measuring instruments; almost none have dealt with the problems of component-inspection tests.

Particle counting is one of the most important variables for users of ultraclean gas components. In contrast to the other procedures, the value obtained directly from the component gives crucial information about its later use. With the exceptions of chemical purity and gas-retention properties, all values are indirectly covered by particle measurement. Table VII lists the variables that influence particle counts.

To maintain a comparable level of measured values among manufacturers, the influencing variables have been standardized. Table VIII uses some typical parameters to show the inspection ranges applied to the performance of these tests. A specific example of the influences of the load-cycling and flow-rate variables is given in Table IX.

CONCLUSION

There are a multitude of testing procedures available for surface evaluation. A quality assurance system, mutually agreed on, can make it possible to achieve maximum quality with minimum effort. The more thoroughly the prior production monitoring is carried out, the more precisely the control tests accompanying the processing can be adjusted to the running sequence. The final inspection of the component can then be limited to measuring leakage rate and particle release.

(The reference listing will appear in a future installment of this series.)

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