

CLEANROOM SUITABILITY OF MATERIALS AND EQUIPMENTS



Newest innovations for semiconductor industry from Saxony and Japan for better energy efficiency

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Outline of the presentation

- Brief introduction
- Materials
 - Particle emission
 - Cleanability, chemical resistance
 - Outgassing
- Production equipment
 - Procedure and assessment
 - Examples
 - Data base
- Documentation of certification results

Technology and Cleanliness Trends

Clusters »Electronics«, »LifeScience«, »Others«

MARKET		TECHNOLOGY	RESULTING CLEANROOM REQUIREMENTS
ELECTRONICS	Semiconductor	Status quo: ■ manufacturing of 300 mm wafers ■ line widths of approx. 65 nm Forecast: ■ manufacturing of 450 mm wafers ■ smaller line widths (approx. 32 nm)	Up to 200 mm fabs: ■ ISO Class 2 ballroom fabs 300 mm fabs: room-in-room-concepts: ■ minienvironments: ISO Class 2, surrounding: ISO Class 5 ■ AMC/ESD-control is highly necessary ■ trend: higher local air cleanliness classes
	Flat Panel	■ Thin-Film Transistor Liquid Crystal Displays (TFT-LCDs) ■ Organic Light Emitting Diode (OLED) ■ flexible panels ■ Field Emission Displays (FED)	Requirements similar to semiconductor industry: ■ particles can cause shorts between layers ■ ESD is critical ■ AMC is not that critical
	Hard Disks	New generation of high density HDDs: ■ increases of areal density of 100% per year ■ heads fly closer to disk: current distance read-write heads averages 20 nm ■ velocity 4.000 – 12.000 rpm ■ Giant Magnetoreistance Technology (GMR)	Requirements similar to semiconductor industry: ■ parts are manufactured in ISO Class 1 to ISO Class 6 ■ critical areas (e.g. head assembly): ISO Class 1 & 2 ■ norm disk drive assembly in ISO Class 5 ■ trend: probably more use of minienviromemts ■ AMC/ESD-control is highly necessary
	Solar	■ sunlight falling on silicon shakes loose electrons and create positive and negative charge ■ more efficiently cells, e.g. thin film technology or alternative contact methods	■ status quo at present: ISO Class 6 to greyrooms ■ more efficient contact methods (e.g. with thousands of tiny metal dots) requires cleanrooms like in chip industry

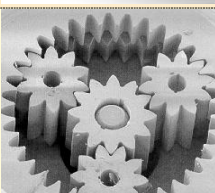
Technology and Cleanliness Trends

Cluster »LifeScience«

MARKET		TECHNOLOGY	RESULTING CLEANROOM REQUIREMENTS
LIFE SCIENCE	Pharmaceuticals	 Products: ■ tablets, capsules, liquids packaged, injectables Critical factors to control: ■ viable particles, bacteria, viruses (> 0,3 µm) ■ toxicity of pharmaceuticals itself, e.g. chemotherapeutics or cytostatic drugs	Room-in-room-concepts ■ minienvironments, high grade of automation ■ aseptic filling process (e.g. antibiotics): GMP A/B ■ surrounding space: GMP C/D or ISO Class 8 (without GMP classification; possible with automated, capsulated processes)
	Med. Dev.	 Products: ■ scalpels, implants, artificial hip joints, contacts etc. Critical factors to control: ■ viable particles, bacteria, viruses (> 0,3 µm)	Requirements similar to pharmaceutical industry: ■ minienvironments, high grade of automation ■ critical area (e.g. implant manufacture): GMP A/B ■ surrounding space: GMP C/D or ISO class 8
	Bio technology	 Fundamental approach: ■ molecular / cellular manipulation Biopharmaceuticals: ■ drug / agents for gene therapy ■ moving from small scale to large scale manufacturing	Requirements similar to pharmaceutical industry: ■ FDA requires the production to be in compliance with GMP ■ controlled cleanroom areas to protect employees and users
	Food	 General objectives: ■ improve shelf life ■ decrease food poisoning incidences / food-borne illness (e.g. caused by E. coli, salmonella, listeria) ■ decrease product recalls	Requirements similar to pharmaceutical industry: ■ cleanroom technology is necessary when no final thermal treatment can be accomplished ■ requires production to be in compliance with GMP

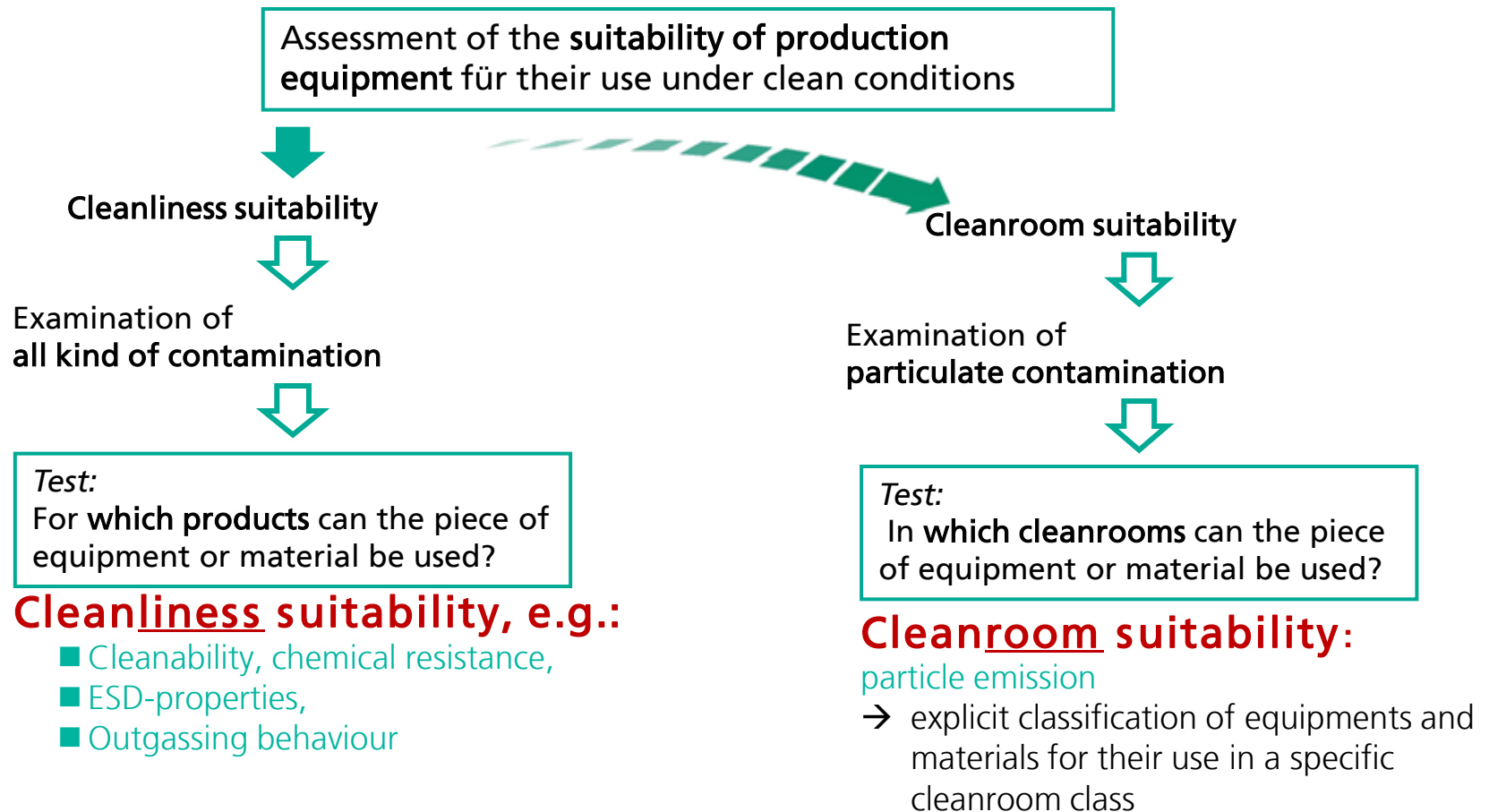
Technology and Cleanliness Trends

Cluster »Others«

	MARKET	TECHNOLOGY	RESULTING CLEANROOM REQUIREMENTS
OTHERS	Aerospace 	Products: Aerospace Structures and Components Critical Contamination: ■ particulate & molecular contamination over the lifetime of equipment as well as during construction ■ interplanetary contamination by microorganisms	■ cleanrooms for construction of large aerospace structures and components (Standard "Air Force Technical Order 00-25-203"), e.g. for spacecrafts, space stations and satellites ■ ISO Class 5 to ISO Class 8
	Automotive 	Hard particles (> 50 µm) critical for: ■ Antilock Brake System (ABS) ■ all hydraulic systems, valves etc. Particles results from the Assembly: ■ joining, personnel, environment	Clean Production in: ■ cleanrooms of ISO Class 8 and less critical sterile rooms ■ cleanrooms without special filter techniques
	Micro-mechanics 	■ typical applications/products: diodes, resistors, transistors, motors, gears, valves etc. ■ high-end applications: down to 0,1 µm particle specification less restrictive components down to 0,5 µm particle spec.	■ cleanrooms of ISO Class 5 to ISO Class 8
	Optics 	Products: ■ lenses, waferstepper optics, optical diodes Critical Contamination: ■ particles with sizes of 0,3 µm to 20 µm ■ AMC	Minimize Deposition on Optics: ■ cleanrooms of ISO Class 5 and less critical

Source: market data mainly taken from "McIlvaine" studies

„Cleanroom suitability“ vs. „Cleanliness suitability“



Production equipment and materials

Today's situation

- **Production equipment and materials:**
~ 40 % influence upon the products' cleanliness

problem

- worldwide **hardly any specifications available:**
most times: mere demand for cleanroom suitability

Method used up till now

- Selection of materials determined by:
Empirics, previous experience, hearsay, appearance
- ➔ Wrong material selection: **loss of production**

Aids

- ➔ Development of a test standard to determine the **cleanliness suitability of materials**
- ➔ Aim: **material data base**



„Halbleiterindustrie-zugelassene“ Werkstoffe	
Metalle bzw. Metalllegierungen	Elastomer-Kunststoffe
beschichtungs- / freie Legierungen: z. B. V2A (1.4301, 1.4303), V4A (1.4571) Stähle (diverse Legierungen): z. B. austenitische Stähle, säure- / korrosionsbest. Ti (Titan) Mo (Molybdän) Al (Aluminium) Div. Alulegierungen z. B. AlMg- oder AlSi-	Ti FKM (Kalrez) FPM (Viton) EPDM (Ethylenpropylenkautschuk) FEP (Tetrafluorethylen, Hexafluorpropylen)
„NICHT-Halbleiterindustrie-zugelassene“ Werkstoffe	
Metalle bzw. Metalllegierungen	
beschichtungsfreie Eisenwerkstoffe beschichtungsfreie Gussstähle beschichtungsfreie Schwarzstähle Buntmetalle und Buntmetalllegierungen: Cu (Kupfer) Zn (Zink) Sn (Zinn); Lötwerkstoffe bilden Ausnahme Bronze Rotguss Messing	Galvanische Beschichtungen: Zn (Zink) Ni (Nickel) Cr (Chrom) Cu (Kupfer) TiN (Titanitrid) Aluminiumlegierungen mit Anteilen von: Cu (Kupfer) Pb (Blei) Mn (Mangan) Zn (Zink)

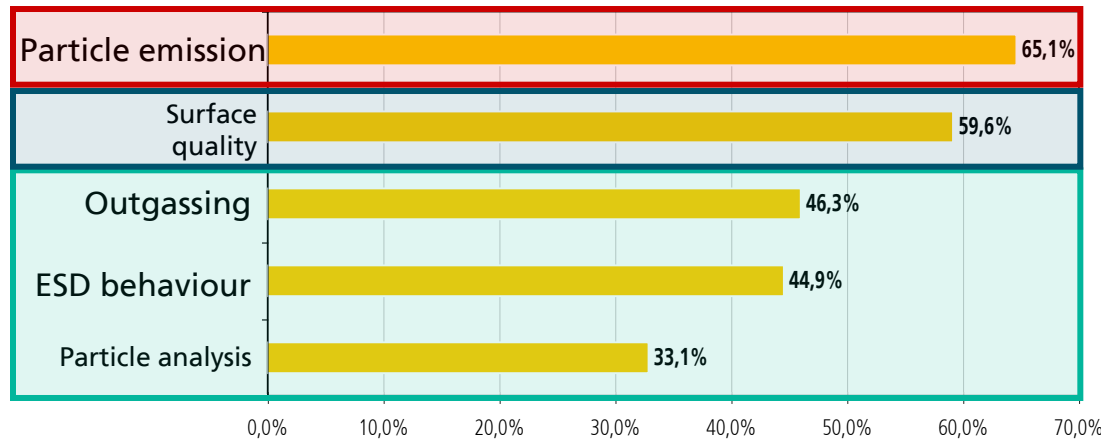
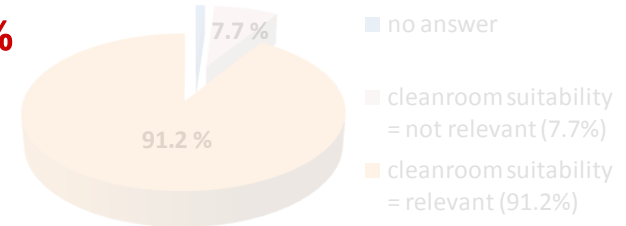
Relevance for the industry?

■ Market analysis of Fraunhofer IPA »Cleanroom suitable materials« (2003):

- Survey with **2700 companies**, quota of return: **11 %**

■ Main results:

- **Very high relevance** of "cleanroom suitability"
- Prioritisation of different **contamination factors**



Most important:
particle emission

Second most important:
surface quality

Next prioritisation:
outgassing, ESD, ...

➔ **Panel of experts:** establishment of an **industrial consortium CSM®**

➔ **Aim: industrially applicable method** to determine the cleanroom suitability of materials

Production equipment and materials in cleanrooms

source: literature research and more than 600 qualification tests

Representative stress of materials

Normal forces:	10 N	$< F < 2500 \text{ N}$
Velocities:	0,1 m/s	$< v_{rel} < 2,5 \text{ m/s}$
Accelerations:	0,1 m/s ²	$< a < 10 \text{ m/s}^2$
Material pairings:	not specifiable	
Material surfaces:	not specifiable	

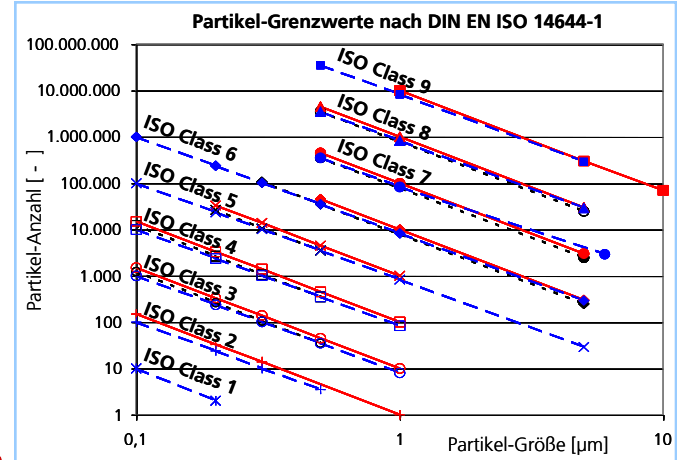
Wafer-Magazine z. B. Polypropylen (PP), Perfluoralkoxypolymer (PTFE)	Rollen für Transportstrecken z. B. Polyoxyethylen (POM), Polycarbonat (PC)	Hochbelastete Gleitsysteme z. B. Aluminium eloxiert, Polyamid 6 (PA6), V4A-Stahl
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Particle sizes:	0,1 µm - 5,0 µm
Particle concentration:	0 - 100.000 particles/min

Requirements for industrial application

- Should be used for **different industries**
- Assessment: in accordance to **intern. accepted air cleanliness classes**
 - Relation to **airborne particles**

Handhabungstechnik z. B. V4A-Stahl, Elastomere	MEMS-/Prozessanlagen z. B. Polystyrol (PS), Acryl- Butadien-Styrol-Copolymer (ABS)	Substratgreifer z. B. Polyetheretherketon (PEEK), Polytetrafluorethylen (PTFE)
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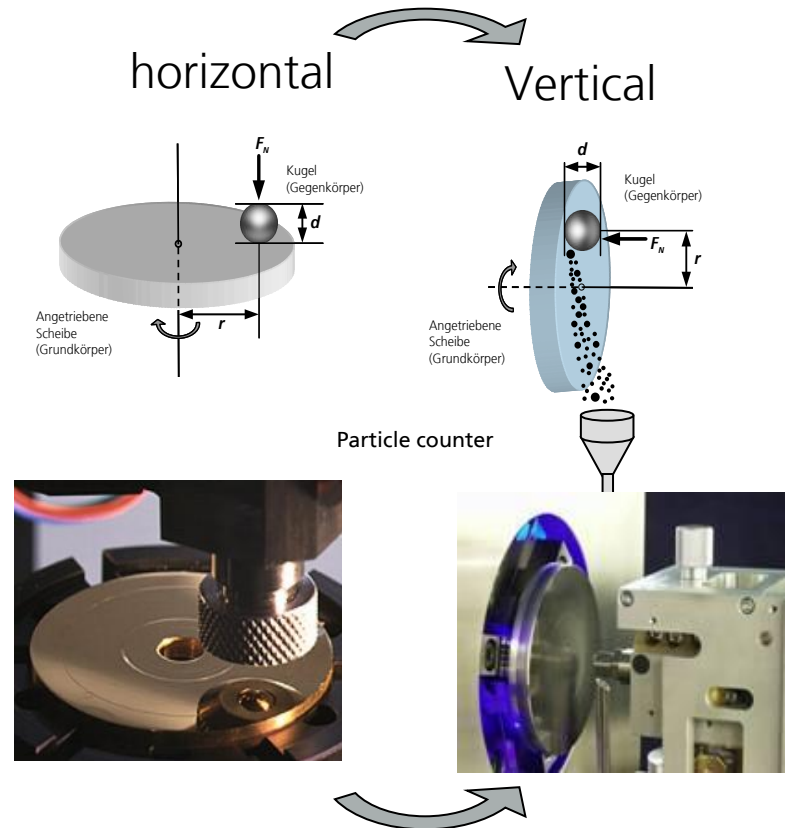
Particle emission

Innovative, creative method for the assessment of materials:

- Small-scale test according to tribological „Pin-on-disk“-technique
- **Turning of the test arrangement**
- **Measurement of emitted particles** with particle counter



Test stand:
Material Inspec



Particle analysis

New innovative analysis for the

assessment:

→ **Conventional view**

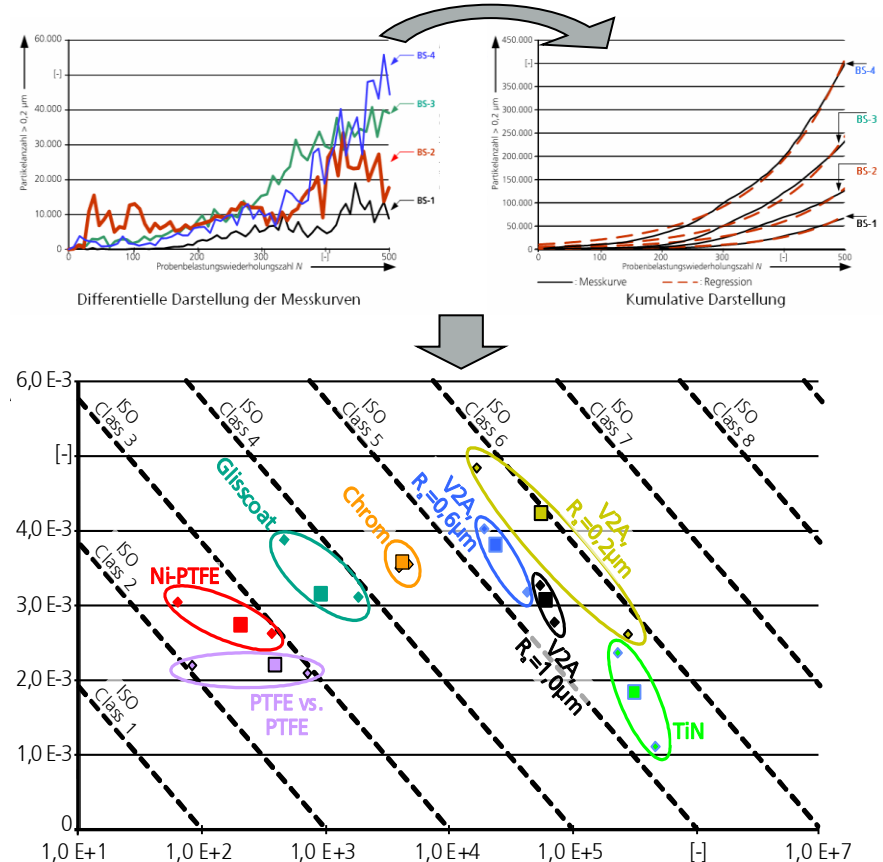
of particle emission: differentiell

→ **Mathematical change of view:**

- cumulation
- non-linear regression
- coordinate transformation

Résumé from these new conceptions:

→ **explicit classification of the cleanroom suitability of materials**

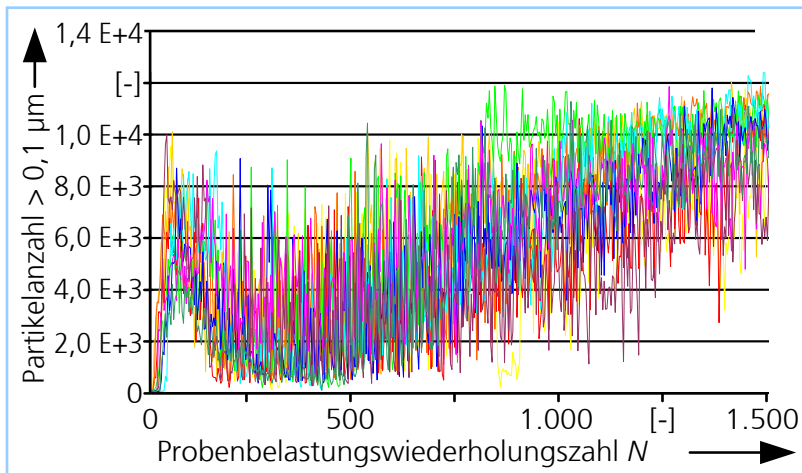


Particle analysis – example of test series

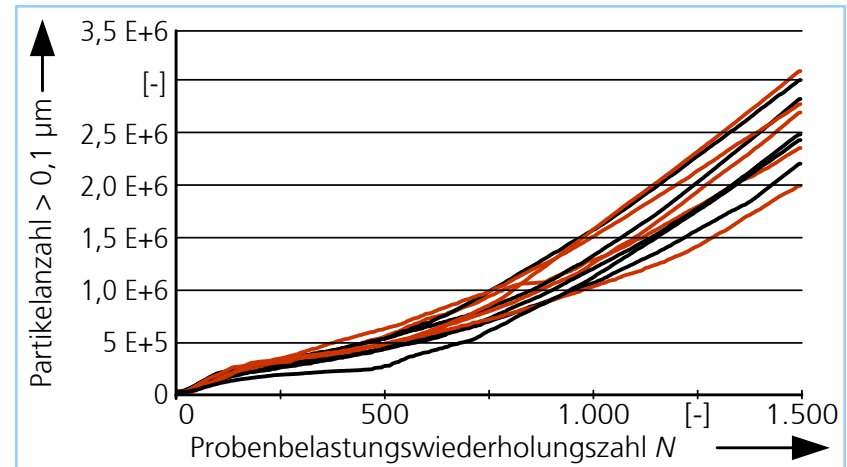
Two representative material pairings

- Material pairing 1 = **metallic sample: V2A ↔ 100Cr6**
- Material pairing 2 = **high efficiency polymer: PEEK ↔ PEEK and PEEK ↔ 100Cr6**

Measurement values of V2A ↔ 100Cr6

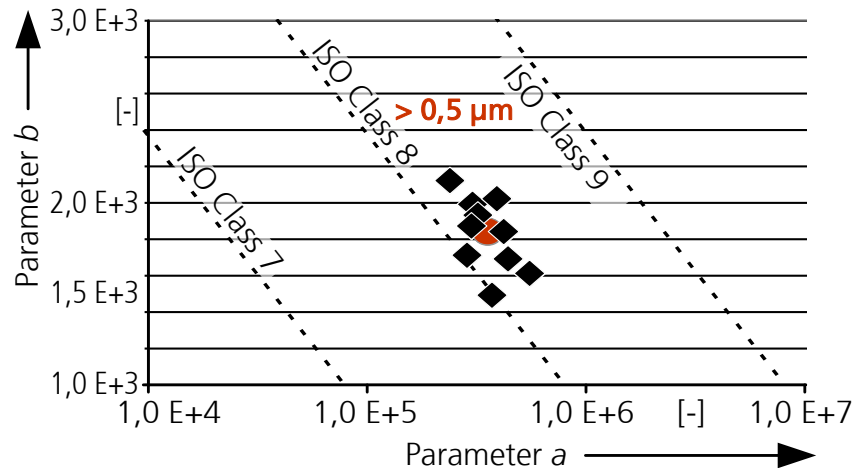
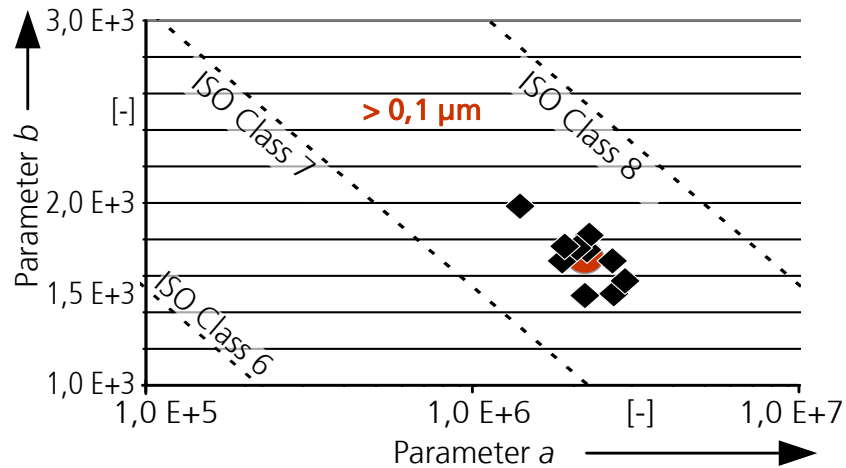


Differential values;
Only tendencies to be recognized



Cumulative values;
Exponential curve to be recognized

Particle analysis – influence of particle size



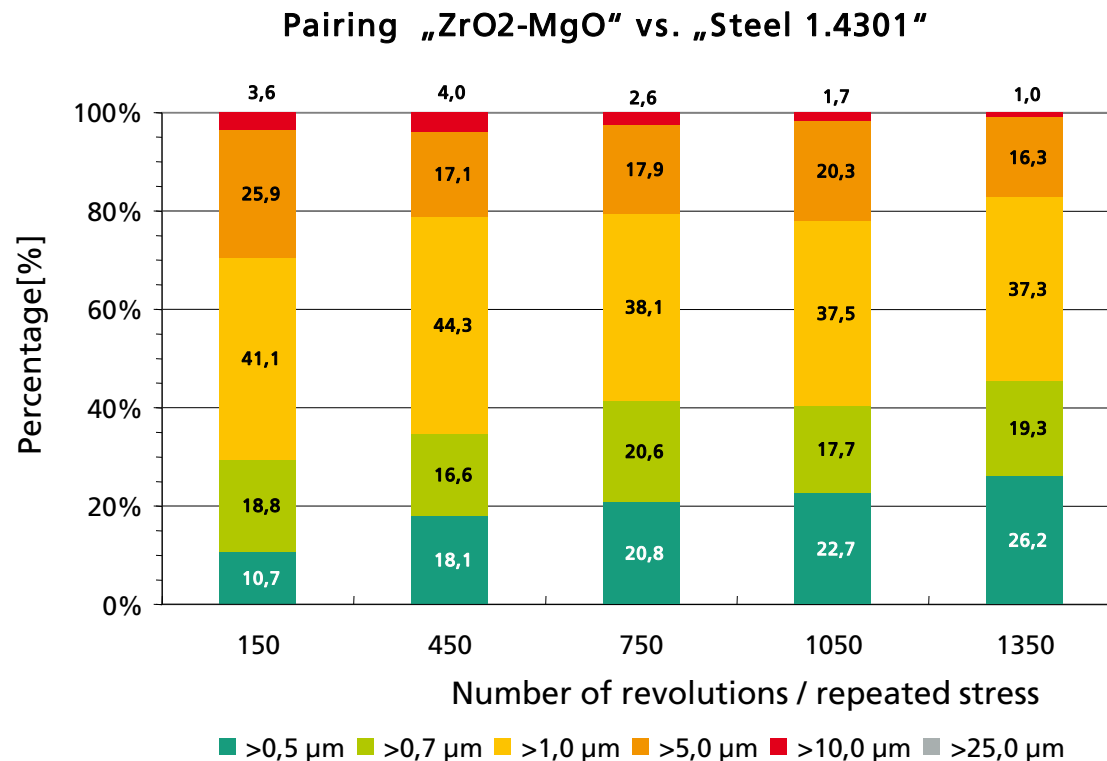
V2A ↔ 100Cr6

◆ single measurement curve

● Curve of mean values

Assessment is sensitive to different particle sizes

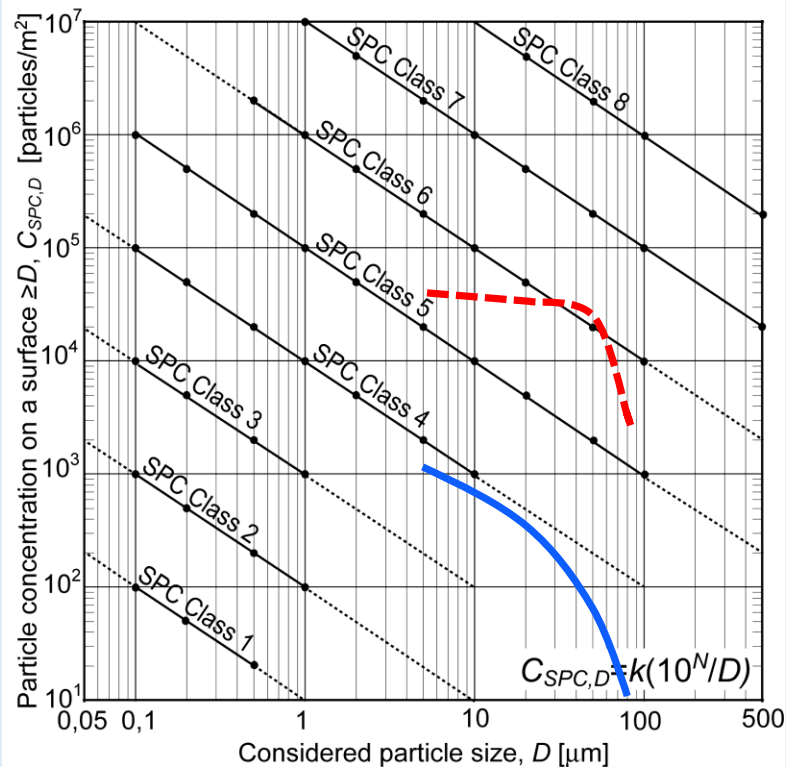
Particle analysis – influence of dynamic processes



Assessment will be influenced by **dynamic wear**:

- particle sizes
- cleanroom classification
- friction coefficients
- stress parameters
- wear parameters

"Cleanability of Surfaces" – Assessment & Grading



Assessment of cleaning efficiency
acc. to ISO 14644-9

Detected particle size [µm]	Mean particle concentration BEFORE cleaning [particle / cm ²]	Mean particle concentration AFTER cleaning [particle / cm ²]	Cleaning efficacy per detected particle size
≥ 5	41677	1142	97,26%
≥ 20	39639	384	99,03%
≥ 50	23042	65	99,72%
≥ 80	4417	11	99,75%
Cleaning efficacy (mean value)			98,94%

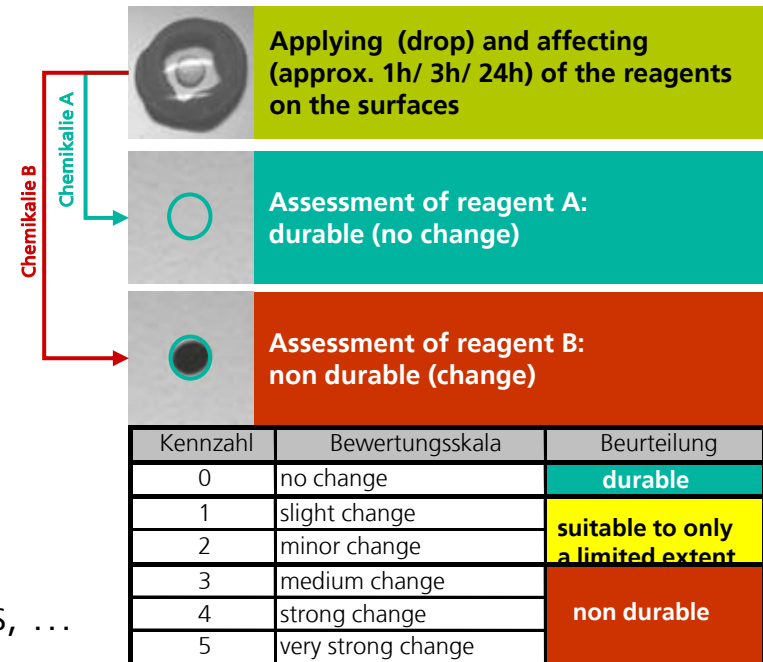
Detected particle size [µm]	SPC-class BEFORE cleaning	SPC-class AFTER cleaning	SPC-cleaning efficacy
≥ 5	6	4	2
≥ 20	6	4	2
≥ 50	7	4	3
≥ 80	6	3	3

Grading into SPC-classes
acc. to ISO 14644-9

"Resistance of Surfaces to Liquids" – Assessment

Surfaces have to be resistant against reagents used to clean, process or sanitize.

- Selection of **representative reagent**
- Mechanism during residence time:
Diffusion process
- Material samples:
should have plain and even surface
- **Assessment of changes on surface:**
formation of cracks, swelling, contraction, blisters, ...



Validation for equipment manufacturer and end-user:

Assessment of chemical resistance of used materials in acc. to

ISO 2812-1 „Paints and varnishes - Determination of resistance to liquids “

Outgassing behaviour of equipment materials

Suitability of equipments to be used in **AMC-controlled environments**?

→ emission behaviour of equipment has to be determined

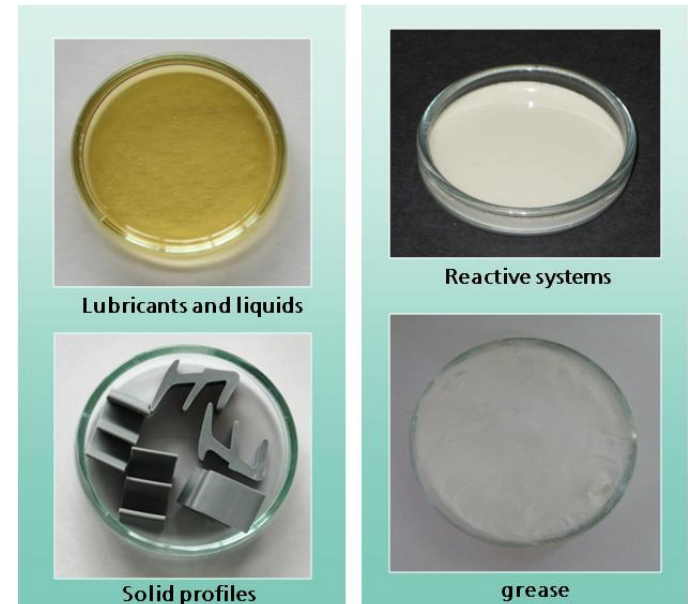
- Examples of **critical materials**:

- Lubricants and sealants,
- Surface treatments, coatings /paints
- Plastics and elastomers
 - harmful for process/product:
fogging on lenses, contact failure, ...

- Examples of **critical compounds**:
Amines, Phthalates, Organophosphates,
Siloxanes, Dopants, Total VOC and others

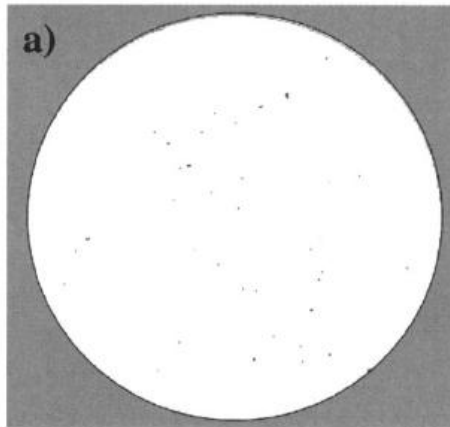
- **VOC**: Volatile Organic Compounds

- **TVOC**: Sum of **a**ll volatile organic compounds

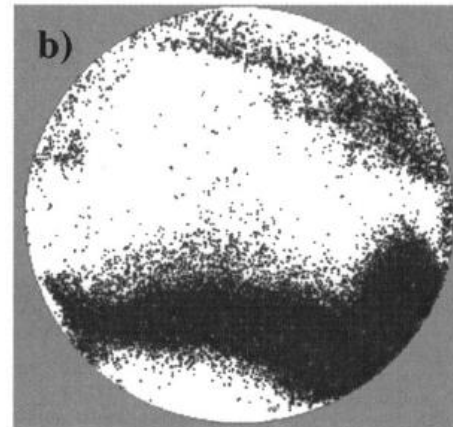


Example: Waferprocessing and TDH

- Time Dependend Haze (TDH) as contamination onto silicone wafers.
TDH: mainly caused by cleanroom atmosphere
- Example: Wafer stored for 4 months in atmosphere enriched with acetone:
Increase of LLS by factor of 15 000 !



Initially:
cleaned wafer



After 4 months:
TDH-range 0.12 to 0.24 μm

LLS =
Localized
Light
Scatters

N. Münter, W. Storm, T. Müller, B. O. Kolbesen "Analysis of Time-Dependent Haze on Silicon Surfaces" Journal of The Electrochemical Society, 150(3) G192-G197 (2003)

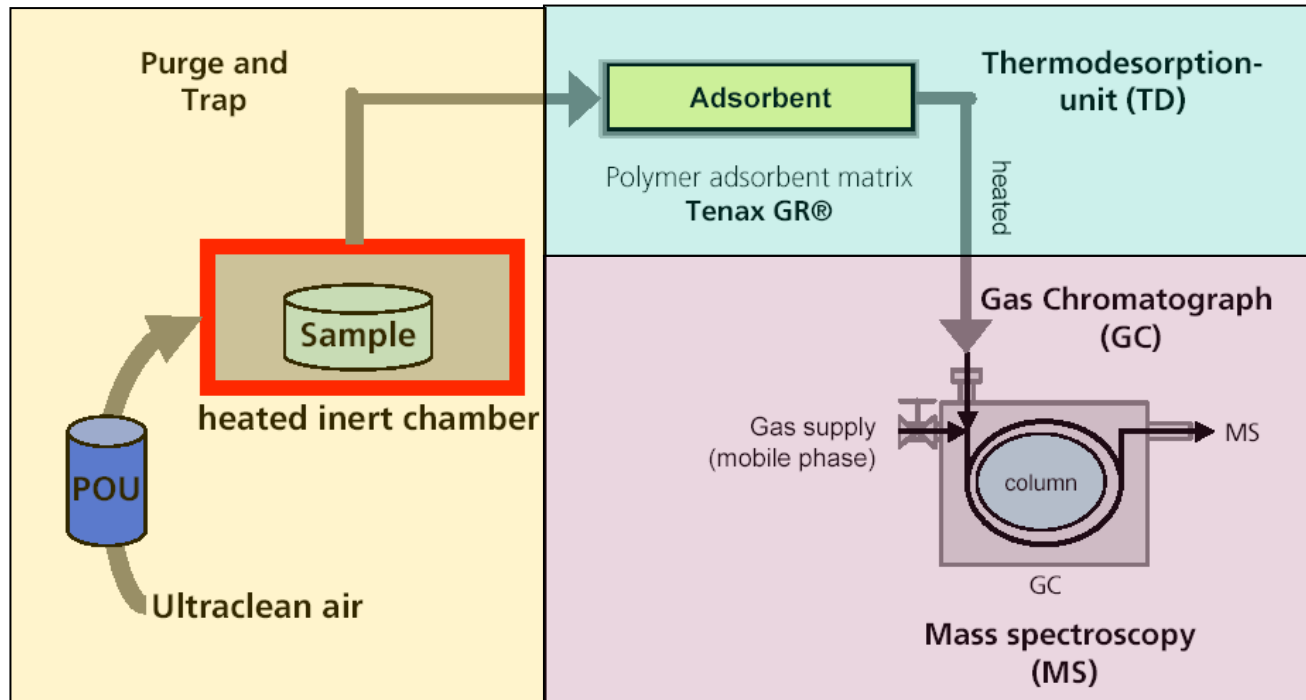
Fundamentals in VOC-measurements

– Emission chamber measurements by TD-GC/MS

■ Purge and Trap TD-GC/MS:

Thermo desorption coupled with gas chromatography and mass spectrometer, emission chamber and adsorbent tubes:

Purge and Trap TD-GC/MS



Material classification acc. to VDI 2083-17 (draft)

Example: representative material sample at 23 °C

■ Example: TVOC-determination $m_{\text{TVOC}} = 306 \text{ ng}$

Normalisation

$A_m = 0,001 \text{ m}^2$
 $t = 60 \text{ min}$
 $m_{\text{TVOC}} = 306 \text{ ng}$

$A = 0,001 \text{ m}^2$
 $t = 1 \text{ s}$
 $m_{\text{TVOC}} = 0,085 \text{ ng}$

$A = 1 \text{ m}^2$
 $t = 1 \text{ s}$
 $\text{SER}_m = 8,5 \cdot 10^{-8} \text{ g/m}^2\text{s}$

$$\text{SER}_m = \frac{m_{\text{TVOC}}}{A_m \cdot t}$$



Hereby applied:

SER_m
 m_{TVOC}
 A_m
 t

= surface specific material emission rate at room temperature 22 +/- 1 °C in $\text{g}/(\text{m}^2 \cdot \text{s})$
= mass of the total volatile organic contamination of the material sample in g
= surface of the material sample m in m^2
= duration of the sampling in s

Material classification acc. to VDI 2083-17 (draft)

Example: representative material sample at 23 °C

The material classification in **AMC_m(or)-classes** is done by using the value **TVOC_{norm}** based on the ISO-AMC classes according to ISO 14644-8:

$$TVOC_{norm} = \frac{SER_m \cdot A_{norm}}{V_{norm} \cdot n_{norm}}$$

Hereby applied (see also previous slides):

V_{norm} = standardized theoretical chamber volume of 1 m³

A_{norm} = standardized material surface area of 1 m²

n_{norm} = standardized purge gas flow of 1/s

$TVOC_{norm}$ = standardized TVOC in g/m³

$$AMC_m(or) = \log(TVOC_{norm})$$



$$SER_m = 8,5 \cdot 10^{-8} \text{g/m}^2\text{s}$$

$$TVOC_{norm} = 8,5 \cdot 10^{-8} \text{g/m}^3$$

$$AMC_m(or) = -7,1$$

ISO-AMC Class	Concentration in g/m ³
0	10 ⁰
-1	10 ⁻¹
-2	10 ⁻²
-3	10 ⁻³
-4	10 ⁻⁴
-5	10 ⁻⁵
-6	10 ⁻⁶
-7	10 ⁻⁷
-8	10 ⁻⁸
-9	10 ⁻⁹
-10	10 ⁻¹⁰
-11	10 ⁻¹¹
-12	10 ⁻¹²

Conversion of the material-specific ISO-AMC_m-class into the ISO-AMC_{CR}-class of cleanrooms

Materials with

- high proportionate material surface area &
- high TVOC emission
 - play major relevance regarding the SER_m-values
 - major relevance for ISO-AMC-classification

Most notably relevant are:

- Large surface areas
 - flooring / walls / ceilings (filter systems)
 - air conditioning technology, ...
- Possible high TVOC-sources:
 - sealants / adhesives / greased systems, ...



Indicator for its success: developed with, and accepted by the industry

- AMD Saxony Manufacturing GmbH 
- Carl Zeiss Semiconductor Manufacturing Technologies AG 
- COLANDIS GmbH 
- DESY-Deutsches Elektron-Synchrotron 
- Festo AG & Co. KG 
- Forbo Flooring GmbH 
- Gerflor Mipolam GmbH 
- Grenzebach Maschinenbau GmbH 
- INA-Schaeffler AG 
- Infineon Technologies AG 
- Qimonda AG, Dresden 
- KARDEX Organisationssysteme GmbH 
- Klüber Lubrication München KG 
- KUKA Roboter GmbH 

- Océ-Technologies B.V. 
- Parker Hannifin GmbH, EMD HAUSER 
- Philips Semiconductors GmbH 
- rap.ID Particle Systems GmbH 
- Schott AG 
- Schunk GmbH & Co. KG 
- Sto AG 
- Sieghard Schiller GmbH & Co. KG 
- Siltronic 
- Sika Deutschland GmbH 
- SKF Linearsysteme GmbH 
- Uhlmann Pac-Systeme GmbH & Co. KG 
- Unaxis Balzers AG 
- M+W Zander Holding AG 
- VAT Vakuumventile AG 

Measurement Procedure to classify Equipments for their Cleanroom Suitability (particle behaviour)



Test environment determination

Decontamination of specimen

Definition of test parameters

Localis. specimen's weak points

Classification measurements

Statistical verification



- Classification:
production equipment is suitable for its use in cleanroom class »X«

Particle Emission Test of a Robot System

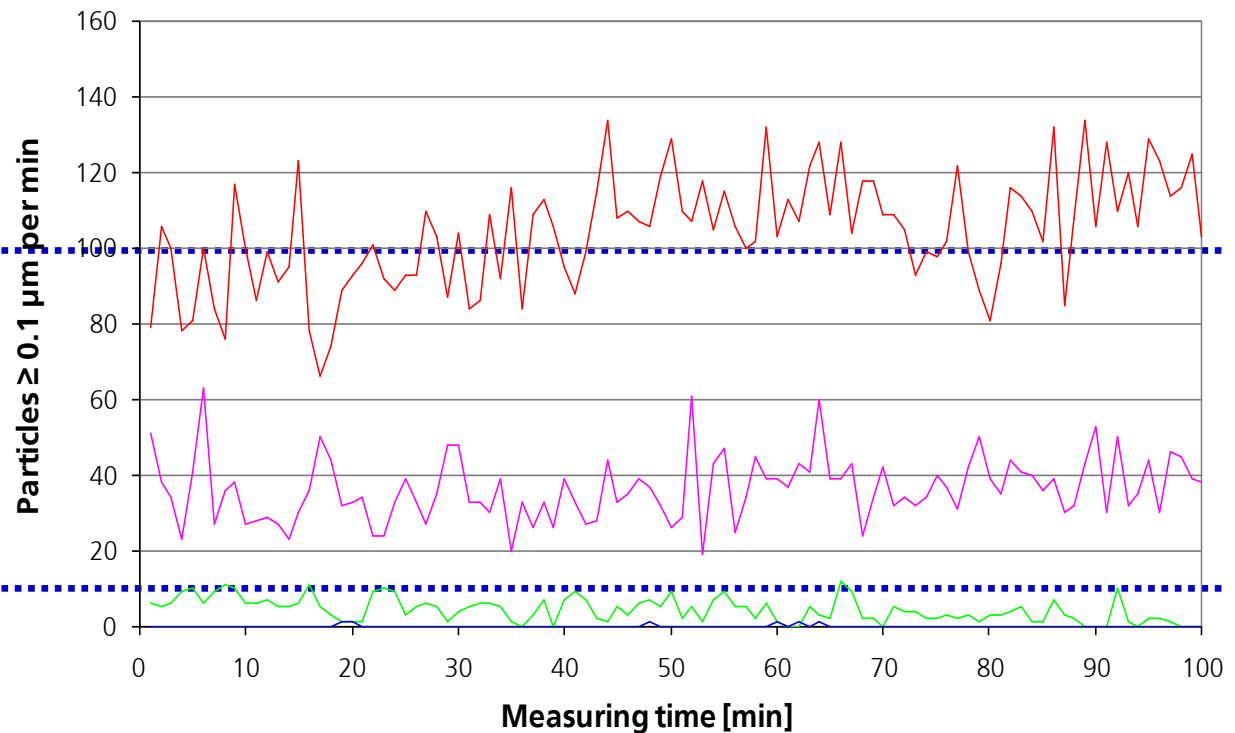
– Interpretation of Measurement Values

- Measurement point 1
- Measurement point 2
- Measurement point 3
- Measurement point 4

ISO CLASS 3?

ISO CLASS 2

~~ISO CLASS 1~~



Particle Emission Test of a Robot System

– Statistical Analysis (as Example on MP1)

Determination of **probability** to exceed particle class limits

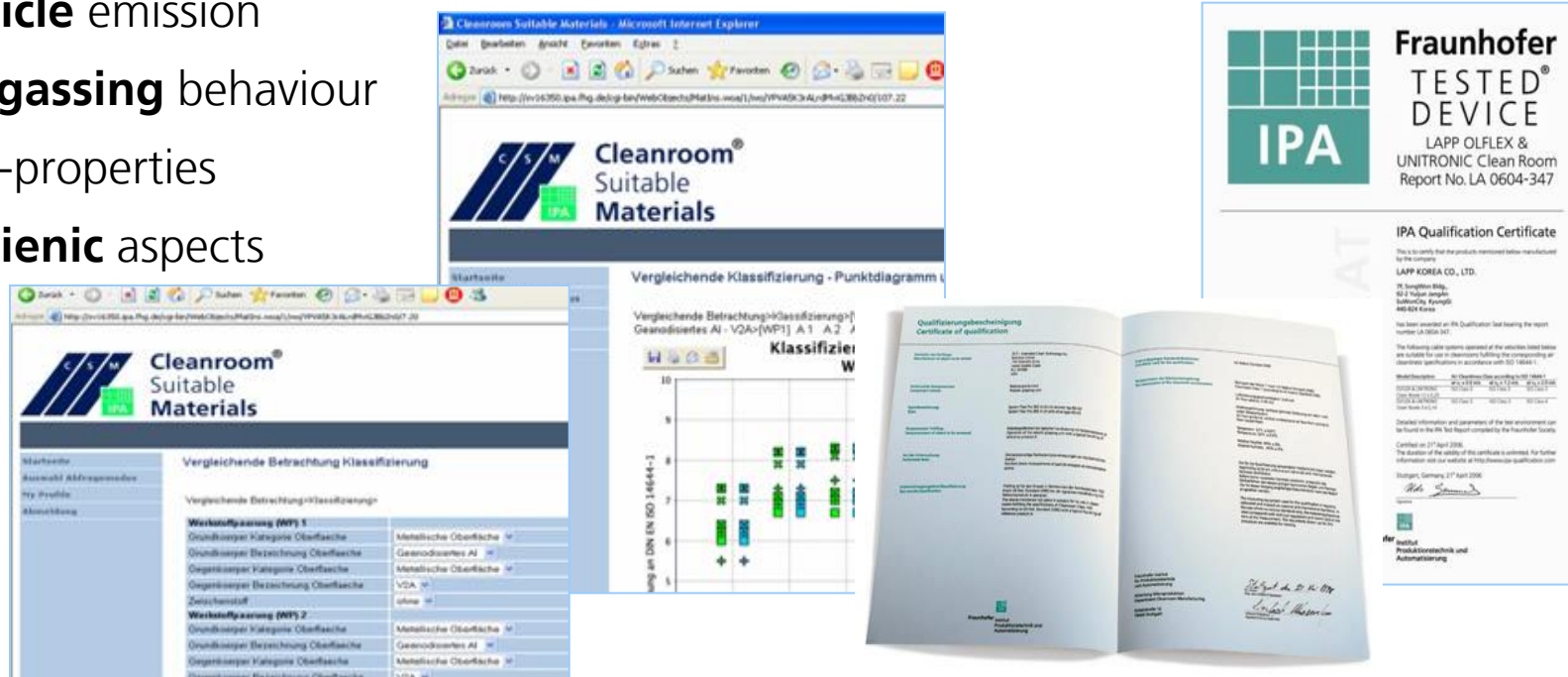
- at single measurement points
- in acc. to class limits of ISO 14644-1 (or any other air cleanliness standard)
- classification with Poisson- and Student-t-statistics

Statistical parameters		Measurement Points			
		MP1	MP2	MP3	MP4
Mean values for the detection size [particles / cft]	0.2 μm	51.7	0.0	3.2	0.0
	0.3 μm	20.1	0.0	1.3	0.0
	0.5 μm	3.5	0.0	0.0	0.0
	5.0 μm	0.0	0.0	0.0	0.0
Standard deviation for the detection size [particles / cft]	0.2 μm	19.3	0.2	2.3	0.2
	0.3 μm	13.7	0.2	1.4	0.1
	0.5 μm	2.9	0.2	0.0	0.0
	5.0 μm	0.0	0.0	0.0	0.0
Air Cleanliness Class [ISO 14644-1]		4	1	3	1
Limiting values of particles in corresponding air cleanliness class for the detection size [particles / cft]	0.2 μm	67	0.0	7	0.0
	0.3 μm	29	0.0	3	0.0
	0.5 μm	10	0.0	1	0.0
	5.0 μm	0.0	0.0	0.0	0.0
Probability of exceeding limiting values for the detection size [%]	0.2 μm	<0.1	3.0	1.7	3.0
	0.3 μm	<0.1	3.0	4.5	1.0
	0.5 μm	<0.1	3.0	<0.1	<0.1
	5.0 μm	<0.1	<0.1	<0.1	<0.1
Statistical certainty of keeping within the required limiting value for the given air cleanliness class [%]		>99.9	97.0	95.5	97.0

Knowledge-based expert system: internet database

(www.ipa-csm.com & www.tested-device.com)

- **Particle** emission
- **Outgassing** behaviour
- **ESD**-properties
- **Hygienic** aspects



So far: approx. **150 materials** and **700 equipments** in databases
ToDo: continuous “filling” of database

Summary

- **Procedure for the assessment** of the cleanroom suitability must be **exactly standardized** to achieve **comparability**.
- Factors to be considered:
 - Low contamination patterns regarding **particle** generation
 - Minimal **outgassing** behaviour and **ESD-properties**
 - **Cleanable, chemical resistant** surfaces
 - Avoiding contamination by usage of appropriate **materials**
- Material & equipment classification is a help
 - for **users**: to find the appropriate equipment
 - for **equipment manufacturer**: to optimize their equipment
 - for a **consistent system**: cleanroom environment / equipment

Contact Information

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www.ipa.fraunhofer.de/cleanroom

www.ipa-qualification.com

www.tested-device.de