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»INVESTIGATION OF CLEANING TECHNOLOGIES AND VALIDATION PROCEDURES APPROPRIATE TO NEEDED CLEANLINESS FOR INSTRUMENTS USED IN THE SEARCH FOR LIFE«

John Vrublevskis

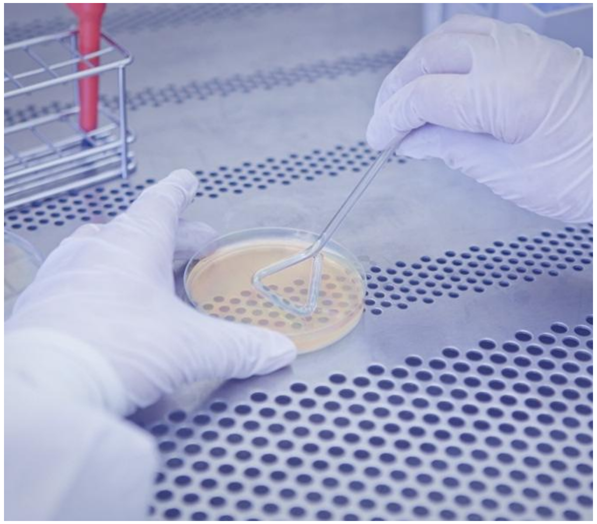
SEA (Systems Engineering & Assessment Ltd), Bristol, United Kingdom

Dr.-Ing. Udo Gommel
Guido Kreck

Fraunhofer IPA, Department Ultraclean Technology and Micromanufacturing, Stuttgart, Germany

Robert Lindner

European Space Agency (ESA)

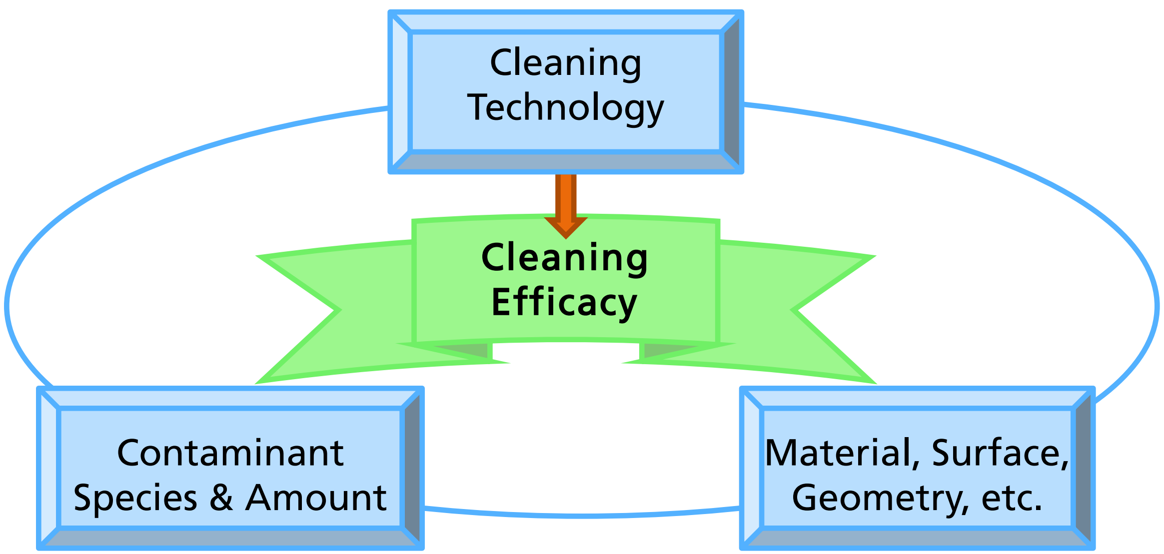


1: Introduction and Background

Cleanliness Requirements of „Search for Life“ Missions

- high reliablity & long life cycles of components
- „Planetary Protection“ aspects
- exobiology instrument requirements: avoid „false positive“ results caused by terrestrial cross-contamination

→ final cleaning of parts and components necessary



validation of cleaning efficacy is necessary:

→ determine influences

→ quantify cleaning efficacies

selection criteria of detection methods:

→ direct

→ quantitative

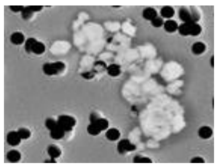
→ no sampling losses

→ 100% test

1: Introduction and Background

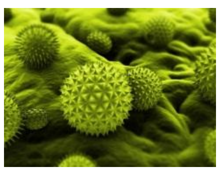
Relevant Contamination and Specific Cleanliness Requirements

Particles



abiotic & anorganic

- manufacturing process residues (dust from abrasive processes, abrading agents, …)
- dust from the environment
- extraterrestrial samples
- etc.



biotic

- bacteria
- spores
- flakes of skin & cell fragments
- etc.

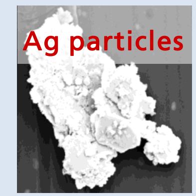
Molecular

filmic (organic & anorganic)

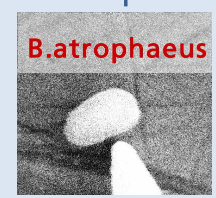
- residues from auxiliary production materials (cooling lubricants, preserving agents, …)
- fingerprints
- etc.

requirements:

- particulate (PAR): < 10 Particles/m^2



- biological (BIO): 0.03 spores/m^2



- organic (VOC): 1 ng/cm^2
  - Hexadecane
  - Diethylphthalate
  - Dimethylsiloxane
  - Toluene
  - Phenylethylamine

2: Available Cleaning Technologies

Cleaning Technologies Trade-Off

- ➔ Which cleaning technologies are available? (34 cleaning methods)
- ➔ Which cleaning technologies are most suitable? (Pre-/Main-/Post-Cleaning)
- ➔ How can I preserve the final cleanliness state? (Packaging Concept)

CLEANING METHODS

- **BLASTING CLEANING**
  - Compressed air blasting
  - Wet compressed air blasting
  - Pressurized fluid blasting
  - Low pressure water jet blasting
  - Elutriation blasting
  - Centrifugal blasting
  - Steam blasting
  - CO_2 pellet cleaning
  - CO_2 snow cleaning
- **MECHANICAL CLEANING**
  - Wiping
  - Brushing, Sweeping
  - Scraping/Abrading
  - Grinding
  - Beating off
- **FLUIDICS CLEANING**
  - Washing/Rinsing
  - Blowing off cleaning
  - Suction cleaning
  - Ultrasonic cleaning
- **CHEMICAL CLEANING**
  - Etching & Leaching
  - Chemical reaction
- **SOLVENT CLEANING**
  - Detaching & Acid-etching
- **THERMAL CLEANING**
  - Evaporating
  - Scarfing
  - Decomposing
- **SPECIAL CLEANING**
  - Hot vacuum purge
  - Plasma
  - UV light
  - LASER
  - Supercritical CO_2

3: Proof of Concept

Validating CO_2 Snow Jet Cleaning: Definition of Constraints

advantages:

- effective cleaning method
- removal of organic and particulate contamination
- universally applicable
- residue-free
- dry

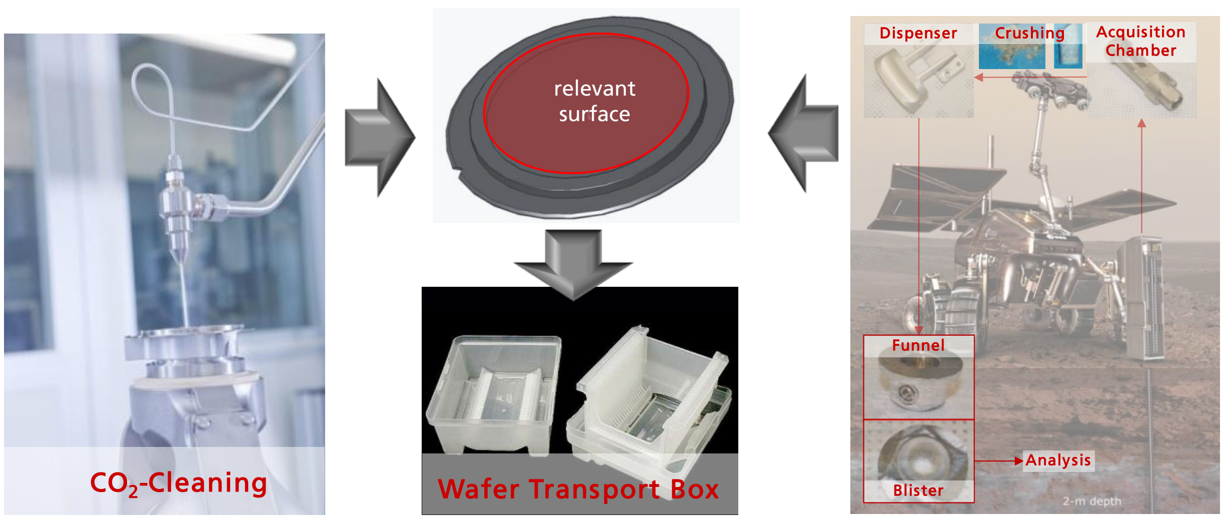
test sample:

standardized test geometry for robot-assisted cleaning

- diameter of test substrate: 100 mm (≈ 4″ Wafer)
- materials, roughness, etc.: freely alterable

test environment:

- Class 1 cleanroom acc. to ISO 14644-1
- benchmark for clean environment
- laminar airflow
- controlled temperature and humidity



3: Proof of Concept

Particle Removal Efficacy - Approach

Test Environment

Flighthardware → Test Surface

↓

defined contamination

↓

analysis of cleanliness before cleaning

↓

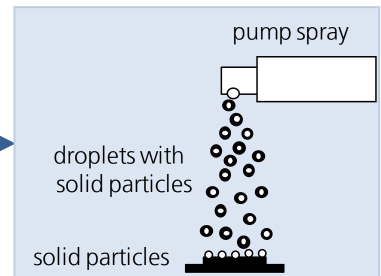
cleaning

↓

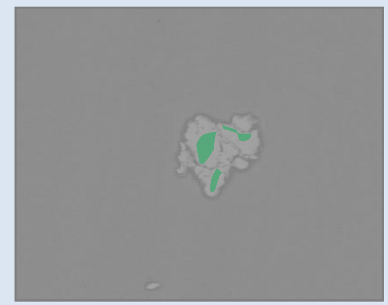
analysis of cleanliness after cleaning

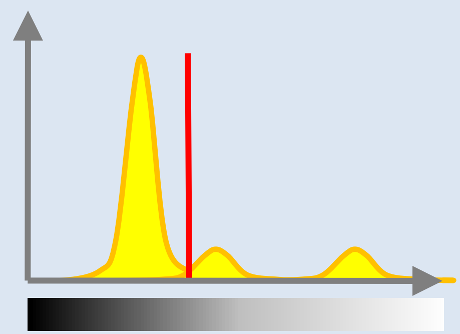
↓

cleaning efficacy



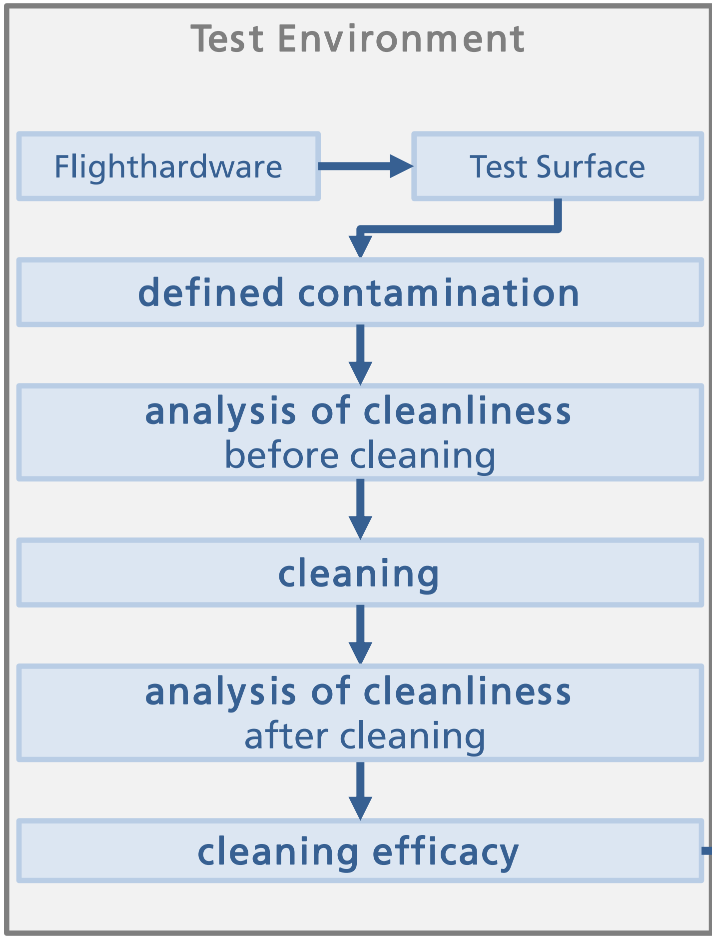
requirement:
➔ adequate material contrast between particles & surface





3: Proof of Concept

Particle Removal Efficacy - Results



Material	Run	Cleaning Efficacies for the Particle Sizes in %			
		≥ 1 µm	≥ 5 µm	≥ 10 µm	≥ 50 µm
Aluminium Al 6061	1st	99.41%	99.97%	99.96%	100%
	2nd	97.71%	99.94%	100%	100%
Titanium Alloy Ti-6Al-4V	1st	99.97%	99.99%	100%	100%
	2nd	99.87%	99.95%	99.97%	100%
Aluminium Alodine 1200	1st	99.99%	100%	100%	100%
	2nd	99.99%	99.99%	100%	100%
Aluminium Black Anodized	1st	100%	100%	100%	100%
	2nd	100%	100%	100%	100%
Steel AISI 304	1st	100%	100%	100%	100%
	2nd	100%	100%	100%	100%
	3rd	100%	100%	100%	100%

- ➔ Steel (R_a≈0.8): zero count twice
- ➔ Titanium Alloy (R_a≈0.5): very good
- ➔ Aluminium Alloy (R_a≈0.5): > 97.71%
- ➔ Aluminium Black Anodized* & Aluminium Alodine 1200* (R_a≈2.4): very good

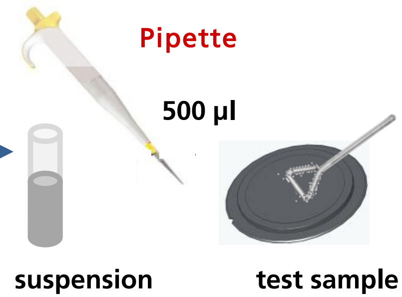
*Notice:
incompatibility to the cleaning method

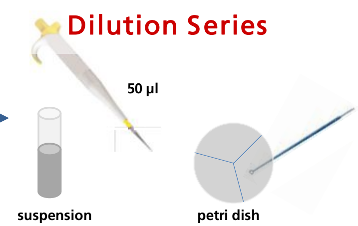
3: Proof of Concept

Spore Removal Efficacy - Approach

Test Environment

- Flighthardware → Test Surface
- defined contamination
- analysis of cleanliness before cleaning
- cleaning
- analysis of cleanliness after cleaning
- cleaning efficacy





- ➔ Is it not possible to determine spore concentrations directly?
- ➔ Spores (Bacillus Atrophaeus) provide too little contrast to enable direct counting using SEM

3: Proof of Concept

Spore Removal Efficacy - Approach

Test Environment

Flighthardware → Test Surface

defined contamination

analysis of cleanliness before cleaning

cleaning

analysis of cleanliness after cleaning

cleaning efficacy

organism	dilution factor	material	applied CFU/test surface	test: CFU/test surface before cleaning	test: CFU/test surface after cleaning	maximum R-value with cleaning Parameter Set 1	maximum R-value with cleaning Parameter Set 2
spores of *B. atrophaeus*	100	Aluminium Al 6061	6.E+05	film	film	**n.d.**	**n.d.**
spores of *B. atrophaeus*	100		6.E+05	film	film	**n.d.**	**n.d.**
spores of *B. atrophaeus*	100	Aluminium Black Anodized	6.E+05	film	film	**n.d.**	**n.d.**
spores of *B. atrophaeus*	100		6.E+05	film	film	**n.d.**	**n.d.**
spores of *B. atrophaeus*	100	Aluminium Alodine 1200	6.E+05	film	film	**n.d.**	**3.5**
spores of *B. atrophaeus*	100		6.E+05	film	306	**3.3**	**3.4**
spores of *B. atrophaeus*	100	Steel AISI 304	6.E+05	film	14	**4.6**	**6.1**
spores of *B. atrophaeus*	100		6.E+05	film	0.5	**6.1**	**6.1**
spores of *B. atrophaeus*	100	Titanium Alloy Ti-6Al-4V	6.E+05	film	3	**5.3**	**5.3**
spores of *B. atrophaeus*	100		6.E+05	film	1	**5.8**	**5.5**

organism	dilution factor	material	applied CFU/test surface	test: CFU/test surface before cleaning	test: CFU/test surface after cleaning	maximum R-value with cleaning
spores of *B. atrophaeus*	10^0	Steel AISI 304	6.E+05	film	0.5	**6.1**
spores of *B. atrophaeus*	10^0		6.E+05	film	0.5	**6.1**

Spore Reduction:

- ➔ Log 6: Stainless Steel (R_a≈0.8)
- ➔ Log 5: Titanium Alloy (R_a≈0.5)
- ➔ Log3/n.d.: Aluminium alloy samples (non-coated, R_a≈0.5)
- ➔ n.d.: Aluminium alloy samples (coated, R_a≈2.4)

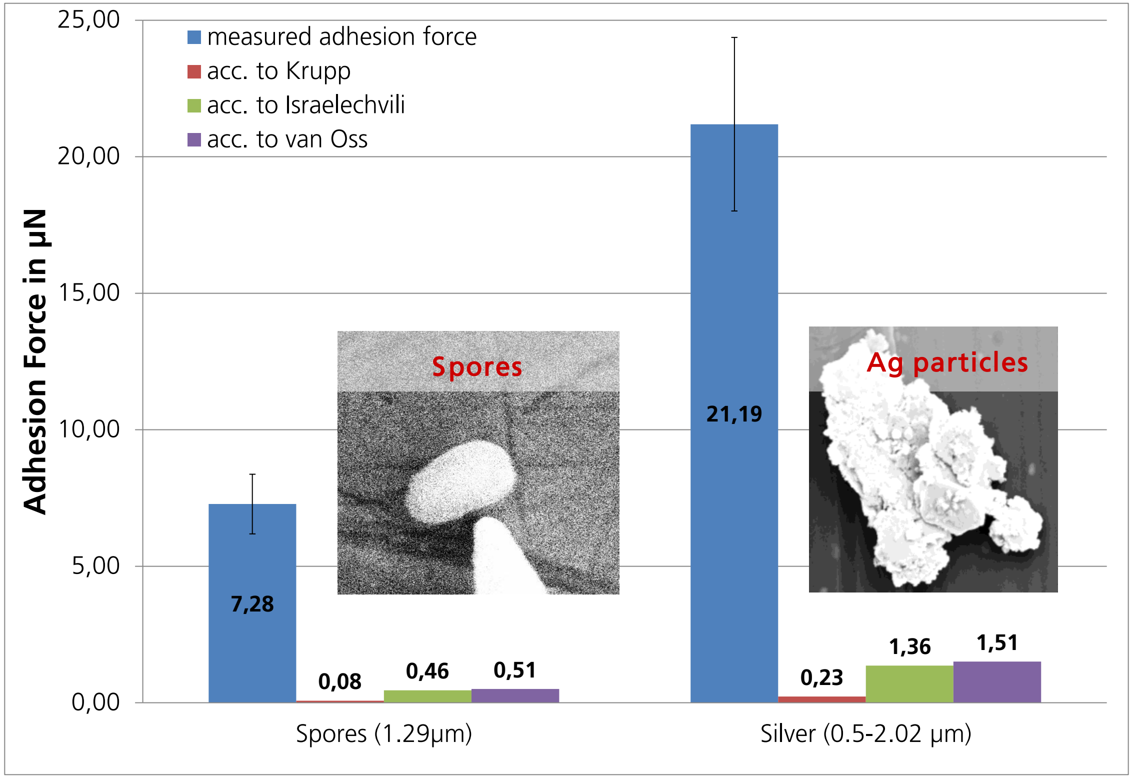
sea Fraunhofer IPA esa

3: Proof of Concept

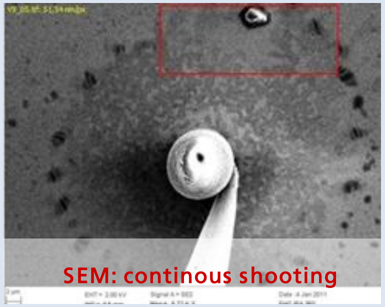
Transfer of Particulate Results

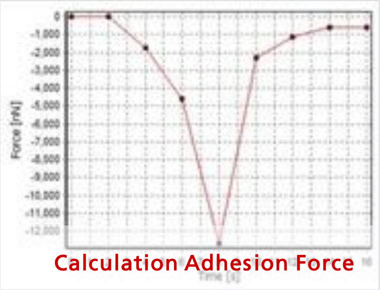
»Adhesive forces are the cause of all surface effects leading to contamination and influencing cleaning«

Hauser G. (2008). Hygienic Manufacturing Technology Weinheim: WILEY-VCH Verlag GmbH & Co. KGaA



Particle Adhesion Measurement





3: Proof of Concept

Organic Removal Efficacy - Approach

Test Environment

Flighthardware → Test Surface

↓

defined contamination

↓

analysis of cleanliness before cleaning

↓

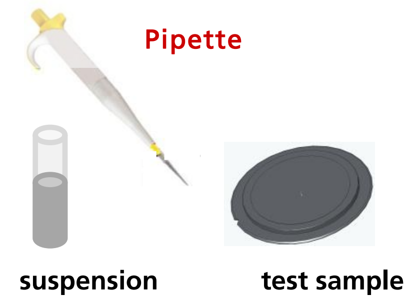
cleaning

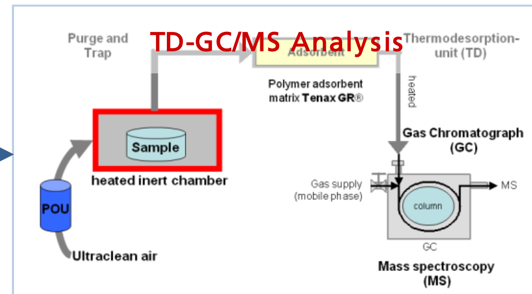
↓

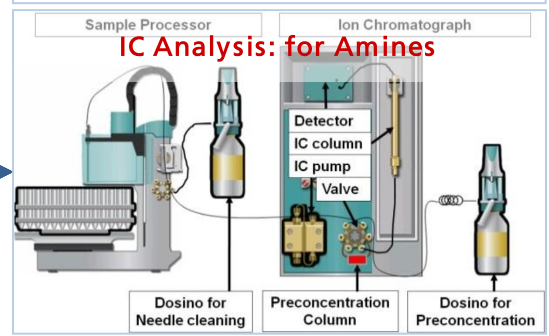
analysis of cleanliness after cleaning

↓

cleaning efficacy







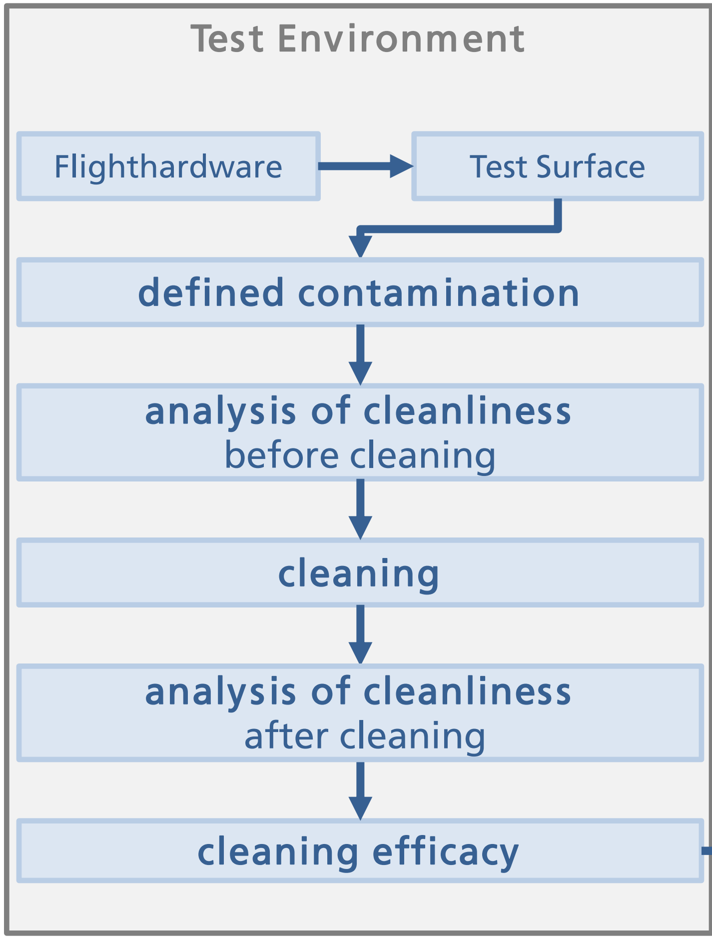
analysis methods:
- ➔ no direct methods available

TD-GC/MS:
- ➔ organics
- ➔ sensitivity and selectivity

IC (complementary):
- ➔ less sensitive than TD-GC/MS
- ➔ possibility to analyze amines

3: Proof of Concept

Organic Removal Efficacy - Results



Organic Removal Efficacies				
	Toluene	DMP	Hexadecane	PDMS
Aluminium ($R_a \approx 0.5$) Al 6061	97%	99%	99%	99%
Aluminium ($R_a \approx 2.4$) Black Anodized	92%	100%	99%	97%
Aluminium ($R_a \approx 2.4$) Alodine 1200	94%	98%	97%	99%
Titanium Alloy Ti-6Al-4V ($R_a \approx 0.5$)	98%	96%	98%	98%
Steel ($R_a \approx 0.8$) AISI 304	98%	98%	99%	99%

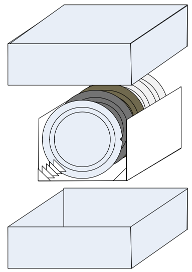
contamination in µg per material	Steel AISI 304 ($R_a \approx 0.8$)			Aluminum Al 6061 ($R_a \approx 0.5$)		
calculated contamination	42	420	4200	42	420	4200
detected contamination prior cleaning first elution	24.3	149.2	5012	17.5	470.0	4918
detected contamination prior cleaning second elution	7.5	13.2	118.6	8.3	11.6	900.5
detected contamination after cleaning	6.3	13.1	8.1	9.2	7.3	7.3
elution efficacy [%]	69.24	91.16	97.63	52.37	97.52	81.69
cleaning efficacy [%]	74.17	91.19	99.84	47.31	98.44	99.85

TD-GC/MS & IC Analysis:

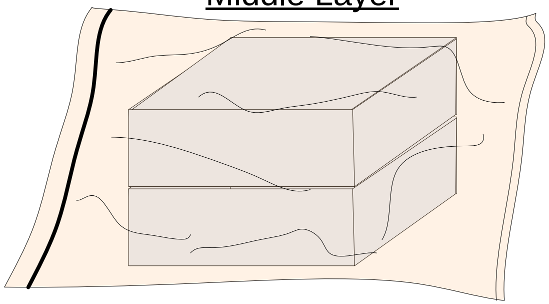
→ 92% (worst) to 100% (best)

→ no appreciable difference in organic cleaning efficacy for different materials

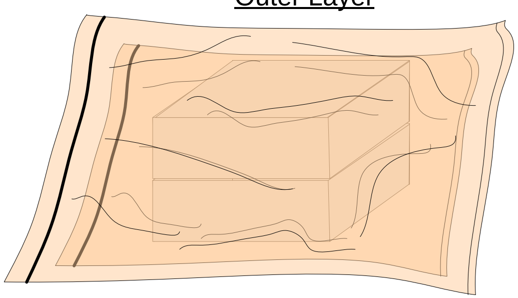
4: Packaging Concept



Middle Layer



Outer Layer

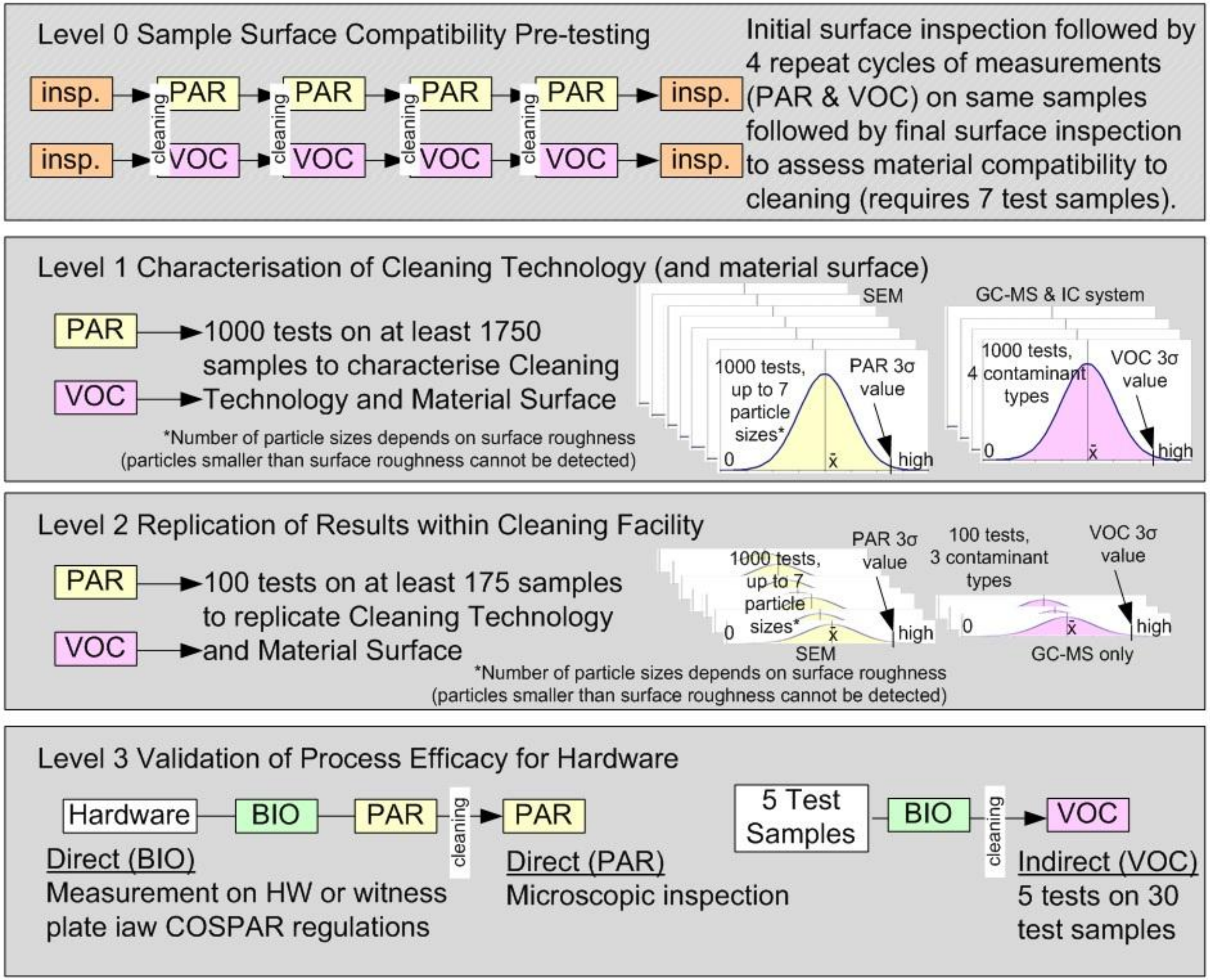


- based on techniques and materials from the semiconductor industry
- inner layer
  - cleaned polycarbonate holder
  - small contact area
- middle/outer layer
  - ultra-clean PP Ziploc bags with ESD protection
  - no contaminants generated during heat sealing
  - PP: low levels of outgassing & shedding

➔ long term storage samples were prepared

➔ level of contamination on these samples will be measured after storage periods of
  - 6 months
  - 12 months
  - 18 months
  - 2 years

5: Detailed Test Design



6: Summary

- requirement: high cleanliness levels of parts & components (PAR/BIO/VOC)
  - → cleaning and selection of suitable cleaning technologies
  - → quantitative assessment of cleaning technologies
- cleaning validation approach:
  - particles: automated SEM analysis with tracer contaminants
  - spores: plating with Agar
  - transfer of particle results: measuring particle adhesion forces
  - organics: combination of TD-GC/MS and IC analysis
  - → suitability demonstrated:
    quantitative assessment of CO_2 snow cleaning efficacies
- preserve cleanliness state: packaging concept
  - standard wafer equipment (optimized in regard of PAR, VOC and ESD)
  - longterm storage tests: assessment of recontamination potential
- statistical hedging: detailed test plan
  - → repetition test series required

7: Acknowledgements

- The Authors would like to thank Thomas Rohr, Nadine Fritz and Gerhard Kminek of the European Space Agency as well as Markus Keller of Fraunhofer IPA for their assistance throughout the study.