

Beyond Particle Counts: Cleanroom Contamination Monitoring

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Outline

- Particle counts: cleanrooms, controlled environments, gases, N₂:
 - ISO classes Air ≠ ISO Class CDA
- Particle ID: on substrates, filter autopsies: conducting particles
 - SEM-EDS whiskers, e.g. data center
- Molecular contamination due to **volatiles, semi-volatiles**, not removed long by filters
- AMCs, Airborne Molecular Contamination: ABCDM's..., ppmv, ppbv, pptrv, SEMI F21, IRDS: v/v units, parts per, vs. ISO 14644-8: µg/m³
- Sources of AMC's: Outside, process, leaks, people, outgassing, cleaning/sanitizing, reactions, excess energy (ions, electrons, UV, X-rays, heat)
- SMCs, Surface Molecular Contamination: Wafers, Substrates, Equipment, Optics
 - Photochemistry, haze, corrosion, wetting for wafers, microfluidics
 - Sub-monolayer, monolayer (adhesion failure), many layers: films vs particles
- Conversion of AMCs to: SMCs, particles, haze, acid + base reactions
- Corona Ionizers for ESD control: AMCs, can grow dendrites, make nanoparticles, imbalance
- Grab, real-time analysis: cost, specificity, speciation and sensitivity tradeoffs
- Standards, refs: SEMI F21, IRDS guidelines, ISO 14644, ASHRAE, IEST

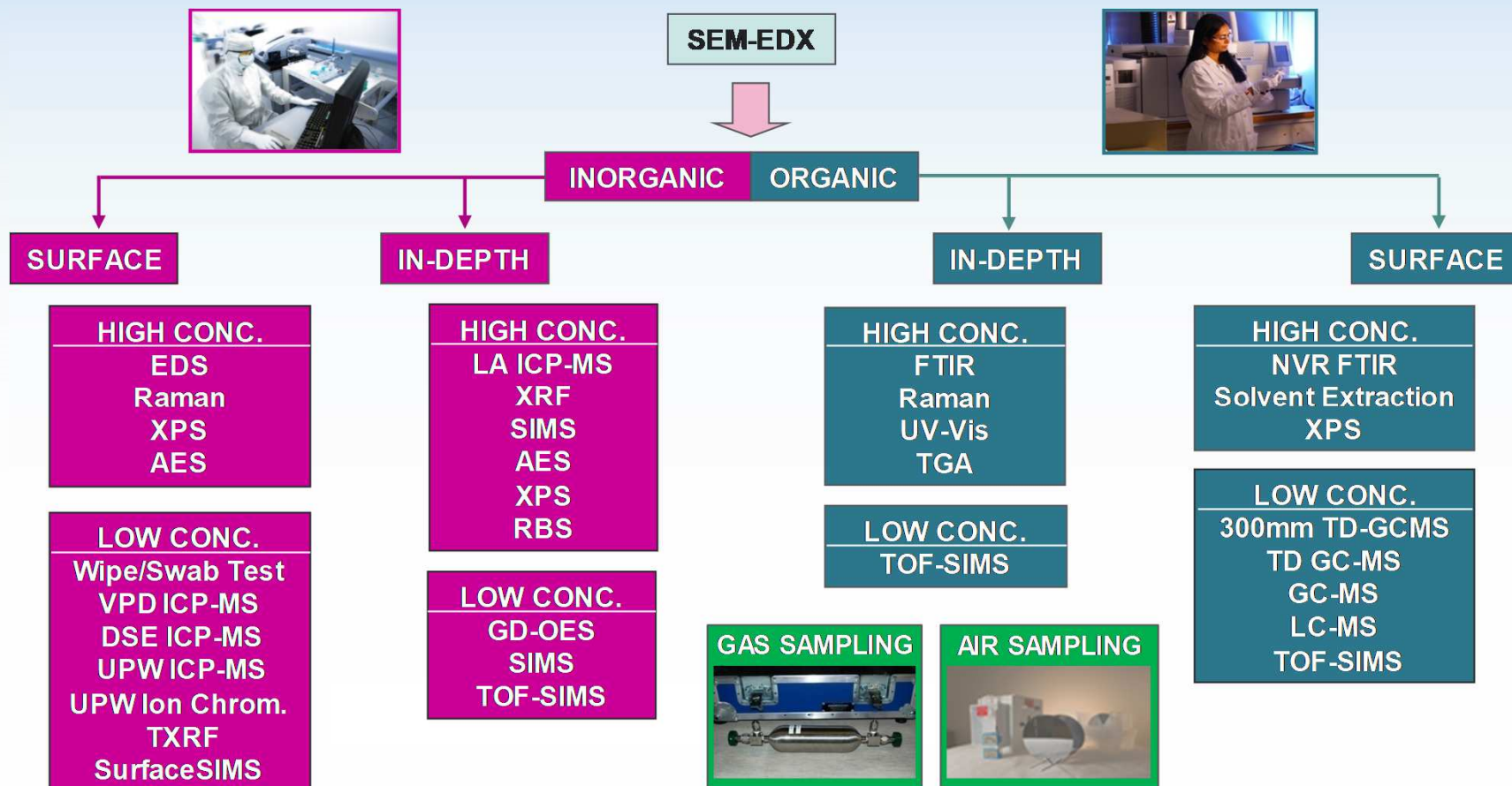
What is the Contamination and Where Is It?



The "Big Picture pie chart:

* Focus of this talk, but all contribute to total contamination control program

Contamination Identification Decision Tree



NEBB focuses on Air Purity: what is needed?

Depends on apps, people vs process

Purity ranges from 0-100%

For particles it is counts/m³, μm or nm size matters, concentration, % removal



ISO 14644-1, particle size in microns



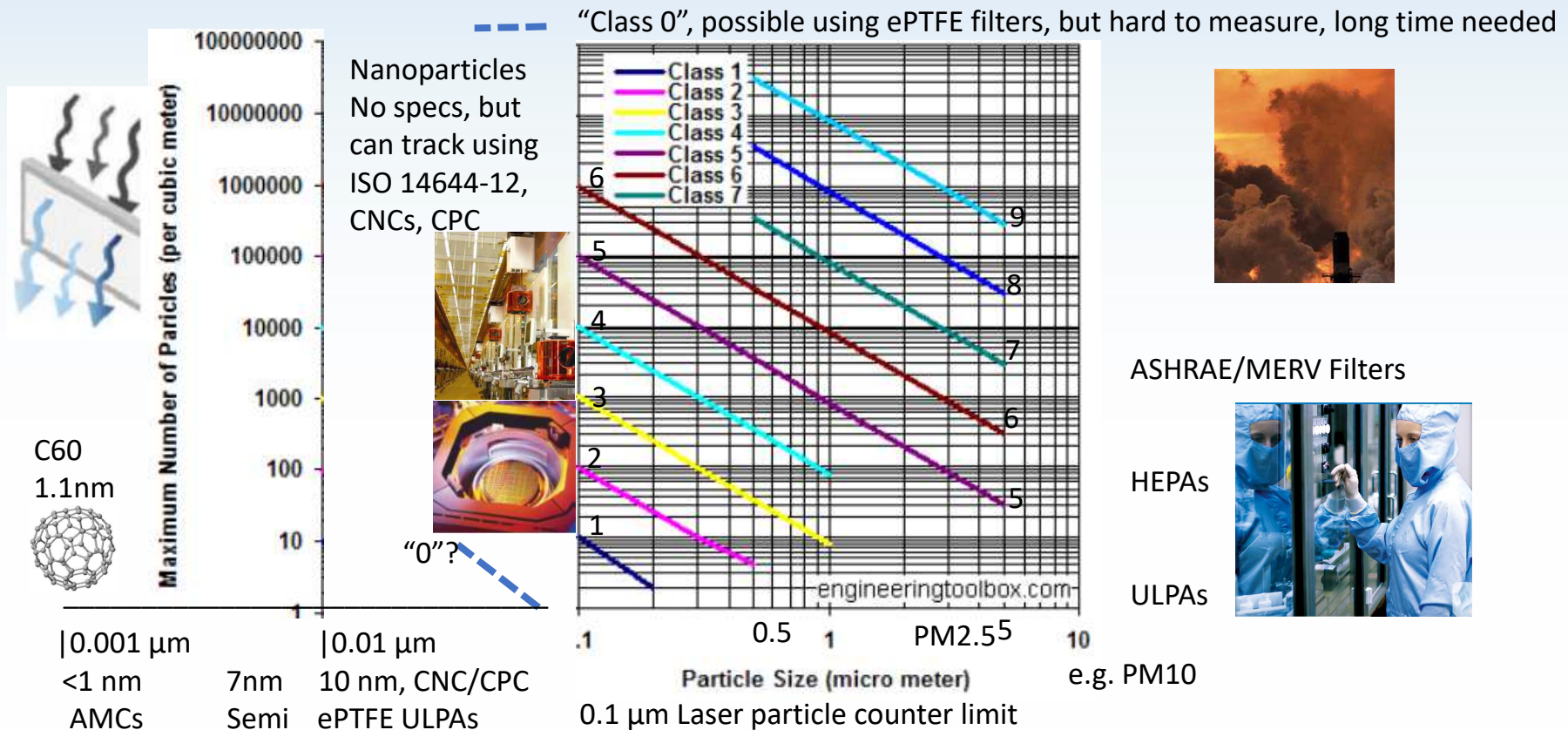
EUV lithography



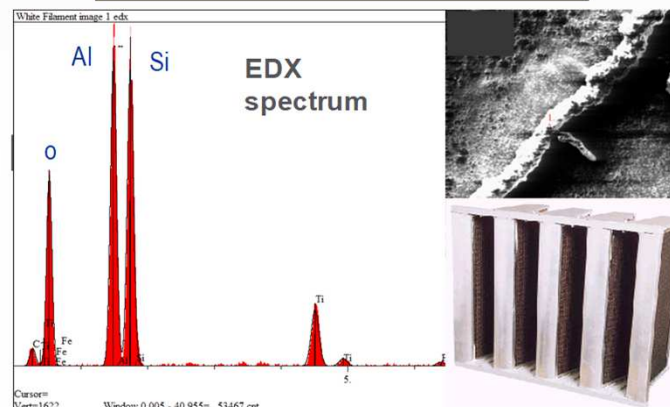
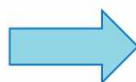
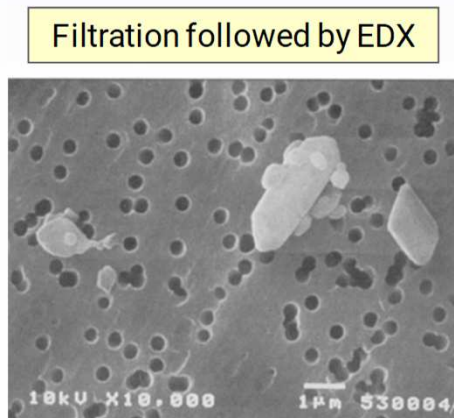
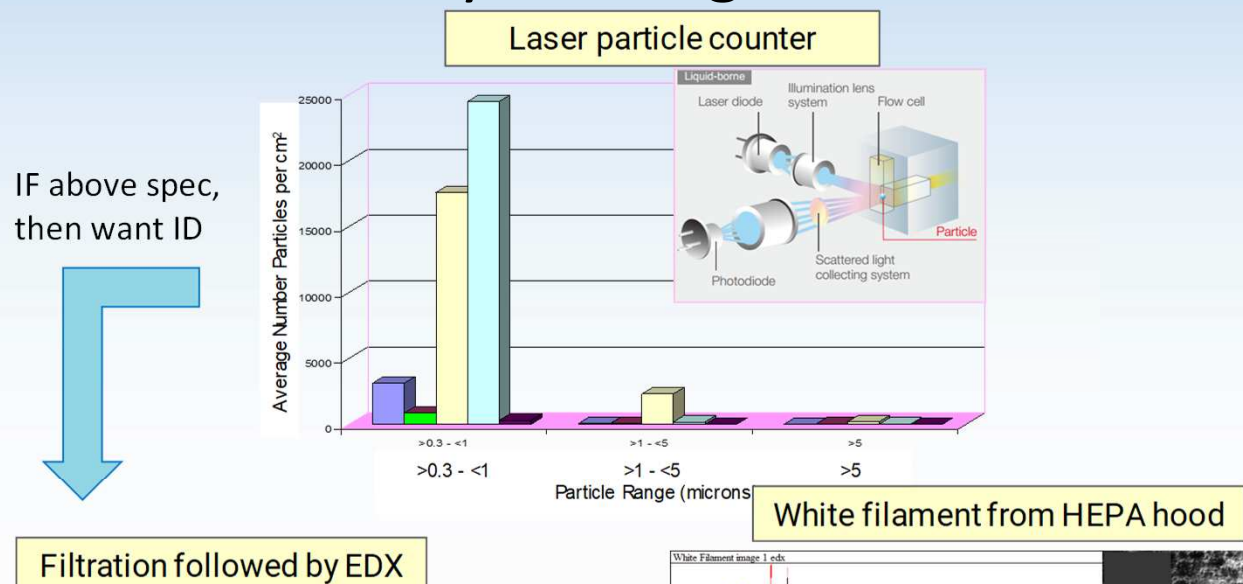
	ISO Class	Maximum Number of Particles in Air (particles in each cubic meter equal to or greater than the specified size)					
		Particle size					
		> 0.1 μm	> 0.2 μm	> 0.3 μm	> 0.5 μm	> 1 μm	> 5 μm
Mini-Env	ISO Class 1	10	2				
	ISO Class 2	100	24	10	4		
Semi Fabs	ISO Class 3	1,000	237	102	35	8	
	ISO Class 4	10,000	2,370	1,020	352	83	
Pharma	ISO Class 5	100,000	23,700	10,200	3,520	832	29
	ISO Class 6	1,000,000	237,000	102,000	35,200	8,320	293
	ISO Class 7				352,000	83,200	2,930
	ISO Class 8				3,520,000	832,000	29,300
	ISO Class 9				35,200,000	8,320,000	293,000

If ISO Class 1, 30 L/minute 0.1 μm laser particle counter: takes 33 min to sample 1m³ to get 10 particles
 ISO Class 0? No such class, yet! 10X cleaner: would take 330 minutes to count 10 particles, very slow!
 IRDS currently recommends ISO Class 1 for leading Semiconductors <10 nm devices (2017-2021)
 Ideally would count e.g. 5 nm particles: CNC counters can do this, but lower flow rates, 3 slpm max, ISO 14644-12. Can monitor for Nanoparticles, but no agreed upon numerical limits yet for sizes, counts etc.

Leading Semiconductor chips 7nm features.
So 3.5 nm particles matter, vs vs ISO counts 100nm+

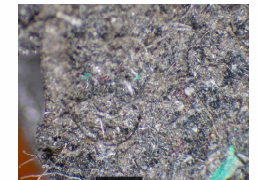


Particle Analysis using Laser Particle Counter



ID of particles is key, depending on process

- Example: DATA CENTER FAILURES, Zn whiskers: shed post filters
- Data Centers filtered, cooled, up-flow airflow from raise floor tiles to dissipate heat
- Electroplated or soldered parts can grow Zn, Sn, Ag whiskers: electrical shorts, failures of: satellites, pacemakers, data centers, but anywhere w electronics. E.g.
- 1 cm diameter black conductive adhesive, sampled underside Zn-plated floor tile: silvery
- Zn whiskers found 1-2 μm wide, **0.5 mm long**, in data center, high failures
- Touching adhesive on failed circuit boards w/high short rates, finds fibers: OK if insulating, e.g. lint, but disastrous if conductive. ISO 8 typical data center
- SEM-EDS finds 1-2 μm D, long Zn fibers present, up to almost mm => shorts



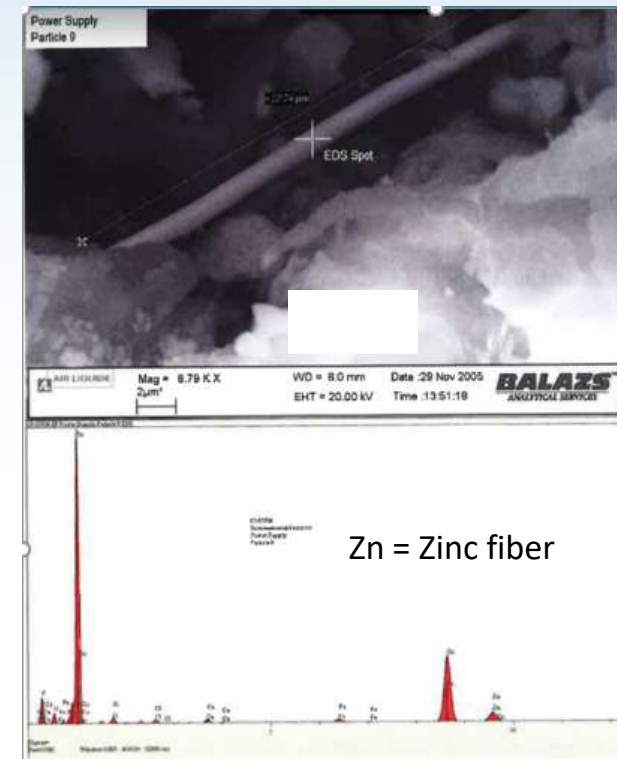
SEM Image + EDS spectrum

used w/permission, Phil Spate, Environmental Protocol Corp

This was datacenter with fine pitch circuit boards, shorts
Many other electronics now have fine pitch IC's
Including factory control centers

Overall, many contaminants were extracted from the board using sticky adhesive tapes, and since they are easily removed, this suggested they might also easily migrate and, if conductive, lead to shorts. The shorts will depend on the length of individual fibers, or for fibers that are too short, several fibers might more rarely combine to lead to a short. In addition to the Zn whiskers, that were the focus of this study, some other possibly conductive contaminants were detected, such as particles with Cu and Zn, possibly due to brass, but these were not fibrous.

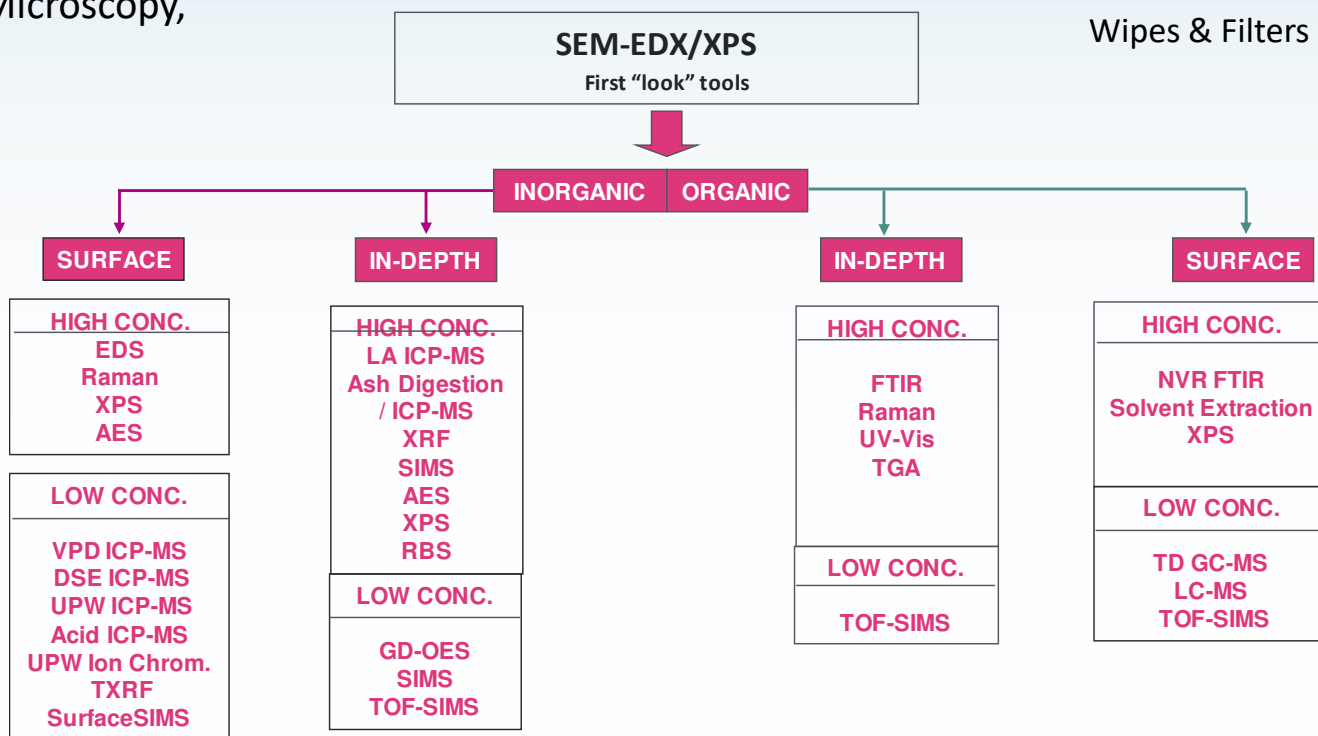
Overall, this study demonstrated that a large number of Zn fibers were easily removed from the floor tile, and some Zn fibers of similar morphology were also found on the power circuit board.



Many methods for analysis of particles, films

Take history first!
Visual, Microscopy,
color

Sampling is key:
B&W Swabs
Adhesive stubs
Wipes & Filters



Particle control key to cleanrooms, but what else?

- If particles over specs: Can ID on filters - “autopsy” on surfaces
- AMC’s=volatile compounds pass thru filters: can affect products
- Acids corrode, affect all electronics
- Acids + Bases make haze, cameras, lasers, optics, lithography
- Bases affect thin film processes, photoresists
- Organics can make haze, delamination of next layer, affect wetting
- Dopants, B, P can effect electrical properties at ppbv or lower
- Corona Ionizers react w AMC’s after HEPA/ULPA to make deposits, nanoparticles, imbalances
- Odors, Toxics, IH, NIOSH, OSHA, EPA –
 - Not covered here, but also very important: ppm-ppb.
 - Process may be much more sensitive vs people

Small Particle Capture Mechanism

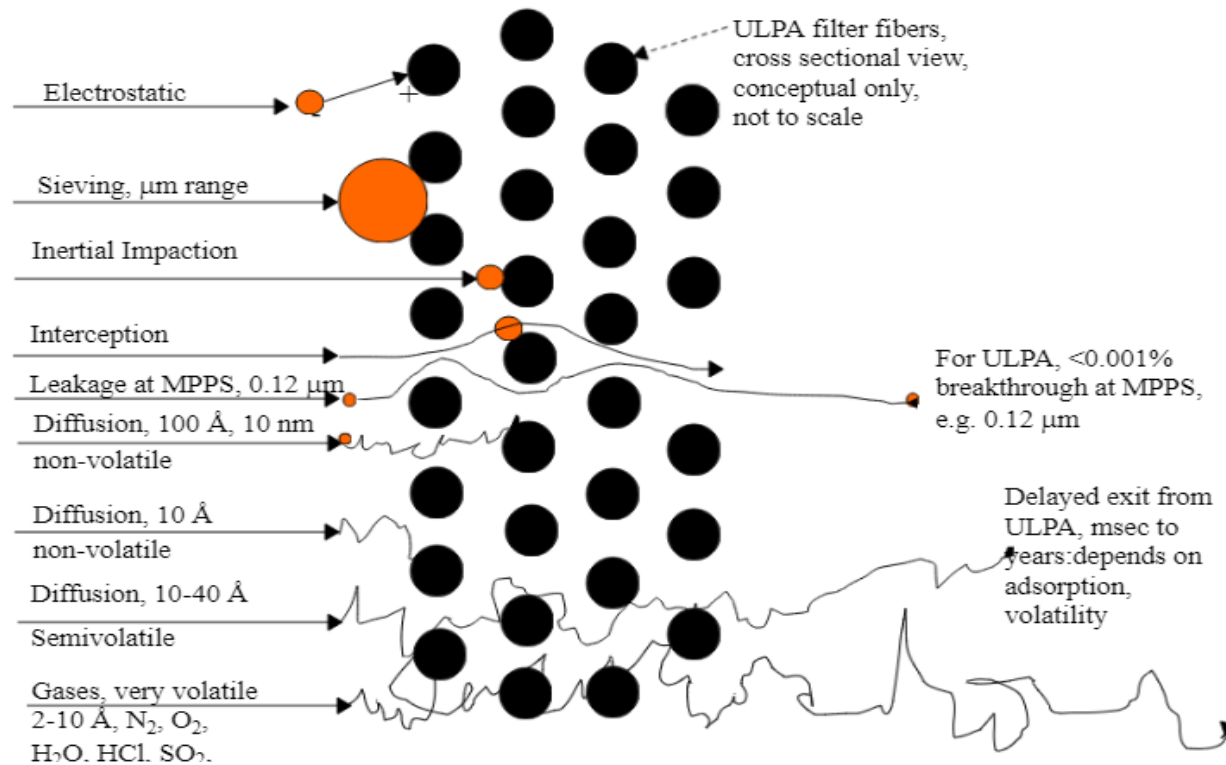
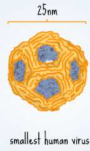
Red Blood cell 7-8 μ m



Smallest Bacterium 0.4 μ m, 400nm

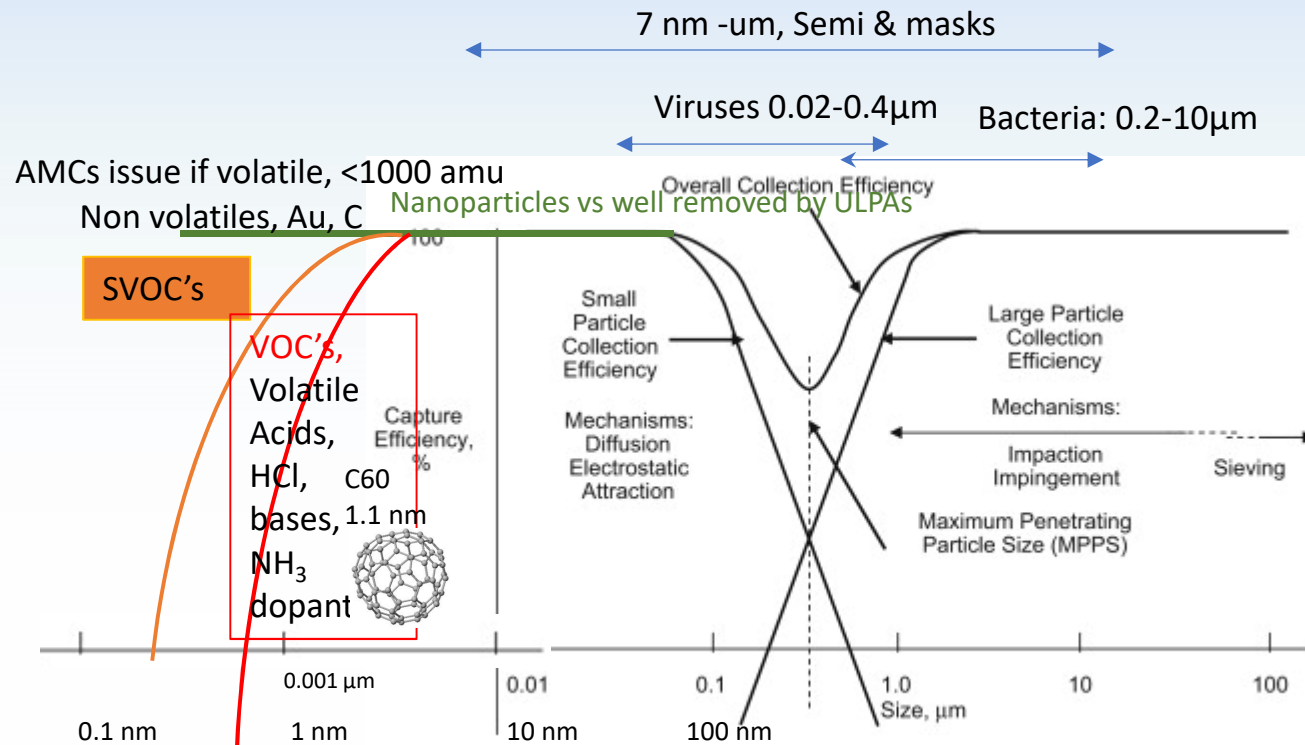


Smallest Virus 25 nm



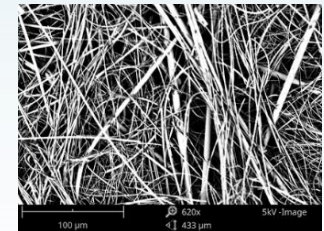
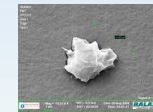
Volatility, not size is key for < 0.5 nm: Au (197 amu) vs Hg (201) vs Xe (131), Ar etc

Bridging AMCs, Nanoparticles, Particles, Filtration

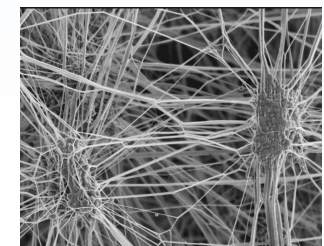


0% retention VOCs, gases

Roger W. Welker, in [Developments in Surface Contamination and Cleaning: Detection, Characterization, & Analysis of Contaminants](#), 2012 (bottom right of graph only)



Borosilicate glass HEPA



ePTFE media,
W. L. Gore & Assoc.

AMC Sources on the Manufacturing Floor

FILTER DEFINITION

- HVAC: Heating, Ventilation & Air Conditioning system
- MUA: Makeup air - air added to fab to make up for that lost to exhaust & leakage
- HEPA: High Efficiency Particulate Air filter
- ULPA: Ultra Low Penetration Air filter, used in cleanest cleanrooms. More efficient than a HEPA filter

Local and regional pollutants

Outside Air = Dirty, AMC > ppbM

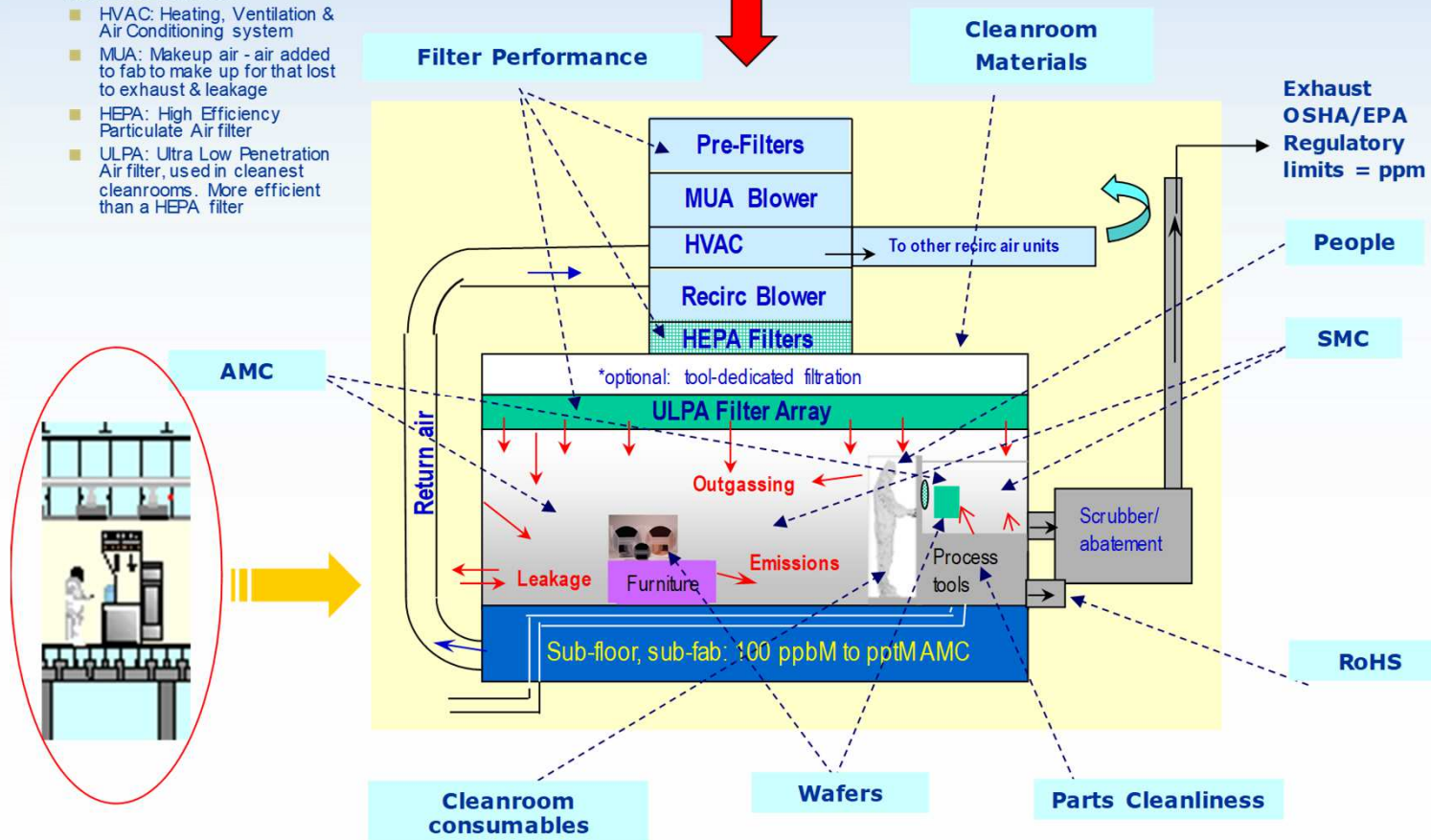


Table 1. SEMI F21-1016 Classification (pptr = pptrM = pptrv)

AMC classification, based on totals found, must be less than:

Contaminant	1	10	100	1,000	10,000
Acids	MA-1	MA-10	MA-100	MA-1,000	MA-10,000
Bases	MB-1	MB-10	MB-100	MB-1,000	MB-10,000
Condensables	MC-1	MC-10	MC-100	MC-1,000	MC-10,000
Dopants	MD-1	MD-10	MD-100	MD-1,000	MD-10,000
Metals	MM-1	MM-10	MM-100	MM-1,000	MM-10,000

SEMI F21 is Classification only, does not say what levels are needed for each process step

Effects of Molecular Contamination

- AMC/SMC can cause corrosion of process wafers, circuit boards, tools, etc.
- AMC/SMC can cause the formation of particles or films that affect adhesion, wafer bonding, electrical conductivity, wetting, microfluidics failures, spotting, and can cause delamination, opens, shorts, leakage currents, and electroplating defects
- Semiconductors
 - AMC/SMC can cause doping errors, nucleation errors, ALD failures, lithography defects, polysilicon or salicide failures. Can affect gate oxide integrity
 - Hazing of wafers, instruments, optics, lenses, lasers, masks/reticles (can be \$100K each)
- As feature sizes get smaller & AMC remains unchanged the effects of the AMC become amplified.
- AMC odors can cause health concerns with employees, but see OSHA, NIOSH, EPA, EHS, especially ppmv, some ppbv
- If AMC controlled at ppbtrv and ppbv levels for process control, fab will be much safer also as a benefit.

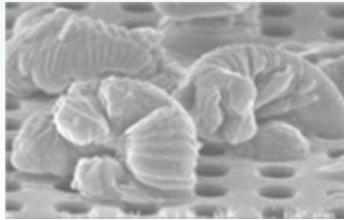
Other issues for filters

- Gel seals: can be bad, drippy, off ratio, solvent swelled
- Polyurethanes: can have organo phosphate fire retardants
- Silicone gels: S, Sn, amines poison Pt catalyst?
 - Drips
 - Outgassing of silicones, even for years,
 - esp. if acid excursions, depolymerize polydimethylsiloxanes to make volatiles cyclic $D_x = [(CH_3)_2SiO]_x$, $x = 3, 4, 5, 6, 7, 8, 9, 10, 11, 12...$
- Potting compounds: can crack, separate from housing = bypass leak , plasticizers can separate, drips, can have Organo Phosphate fire retardants that outgas for years
- Aerosol leak tests: loading of filters especially ePTFE ULPA in some cases
- Fan failure, FFU mounts break, SW, bearings, outgassing, overheating, lubricants, circuit breaker, fan belts failure if pressurized plenums, corrosion
- Possible biofouling, especially if wet, %RH upsets, water drips

AMC: Molecular Acids (MA)

- Effects of Molecular Acids

- ✓ Corrosion of Cu, Al and other Films
- ✓ Corrosion of Process and Test Equipment and the Facility



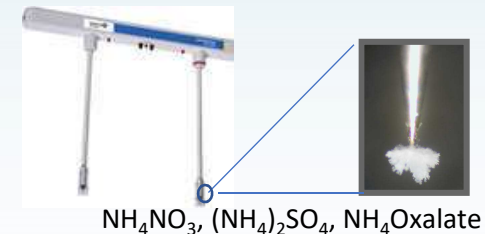
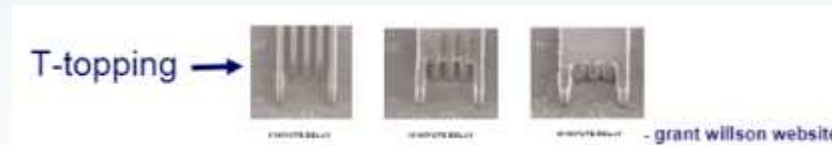
Al corrosion due to Cl
(used with permission)

- Reaction of acids with bases to form ionic particles on different surfaces, $(\text{NH}_4)_2\text{SO}_4$ and NH_4Cl
- Hazing of wafers, optics, masks and metrology tools
- Unwanted silicon wafer doping due to HF attack on borosilicate glass in HEPA filters – this causes filter degradation that can release boron into the cleanroom
- HF can attack SiO_2 surface critical for thin gate oxides

AMC: Molecular Bases (MB)

■ Effects of Molecular Bases

- ✓ The presence ammonia, amines and amide at only ppb levels has been observed to interact with chemically-amplified photoresists and lead to lithography defects such as “T” topping

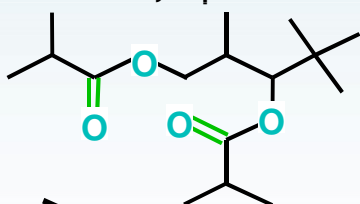


- ✓ The reaction of molecular bases in air with acid vapors has led to the formation of ammonia salts and subsequent hazing of particle/droplet formation of wafer and optics surfaces
- ✓ High ambient base levels in cleanroom air or clean dry air (CDA) can rapidly degrade filter lifetime, leading to premature filter failures and yield losses
- ✓ Reactions between molecular gases and acids in the air may lead to “Fuzz ball” formation on air ionizer tips and large particles shedding into the cleanroom

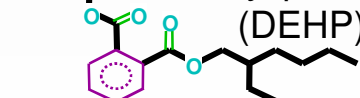
Molecular Condensable (MC) = Organics

Plasticizers

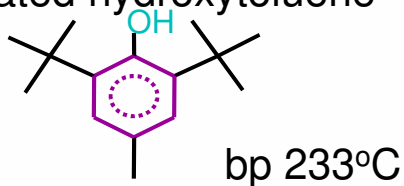
(Texanol isobutyrate)
TXIB, bp 280°C



DOP, bp 384°C
dioctyl phthalate
(DEHP)

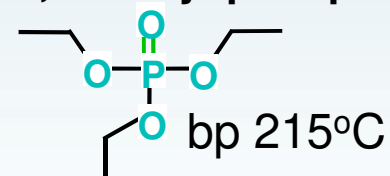


Antioxidants: BHT
Butylated hydroxytoluene



Organo Phosphates

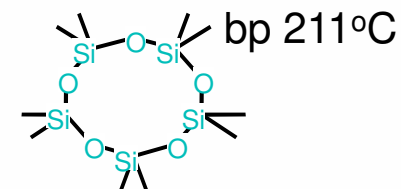
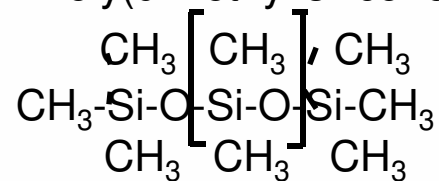
TEP, Triethyl phosphate



If Si, S, P etc: "Refractories" can deposit
onto UV optics, Corona Ionizer tips

Typical Silicones (siloxanes)

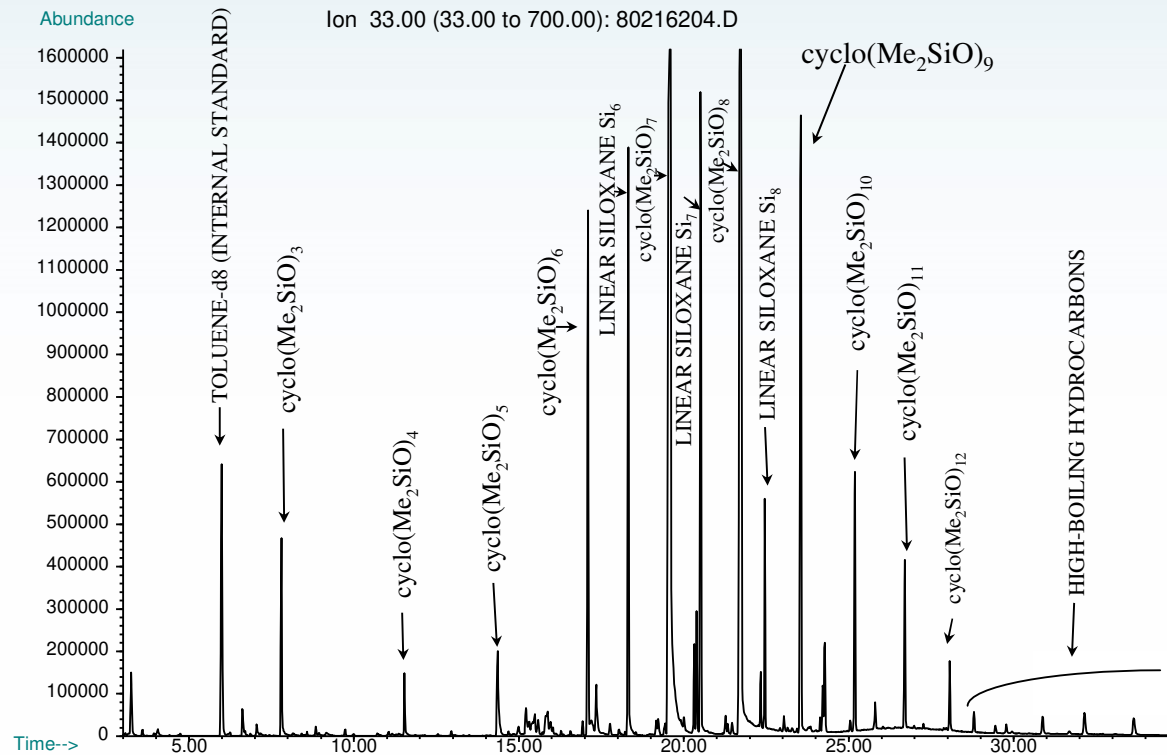
Poly(dimethyl silicone)



Silicone HEPA Gel Seal Outgassing onto Silicon Wafer

Dynamic headspace GC-MS of organic compounds desorbed at 300°C for 1/2 hours from wafer exposed to Silicone HEPA gel sealant "A". Outgassing collected onto wafers at 50°C for 24 hours. Balazs internal R&D. Sample tested after 1 year cure!

Silicones affect wetting, cleaning, form SiO_2 on ionizer tips, haze UV optics, lasers, inspection tools especially 193 nm optics, photomasks/reticles, can contribute to thin film ellipsometry thickness errors



Wafer Exposure

Experiment

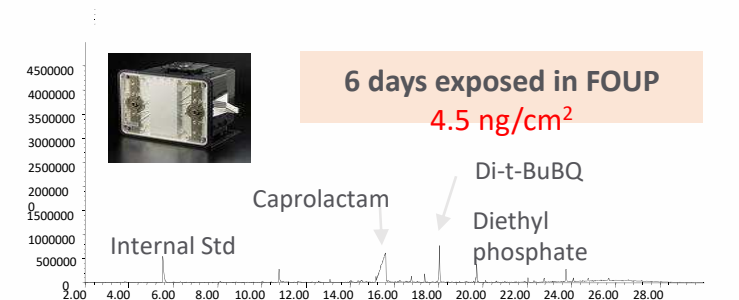
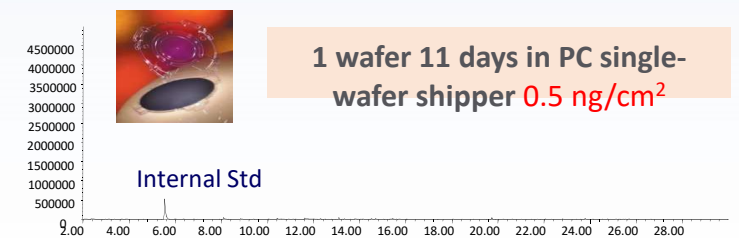
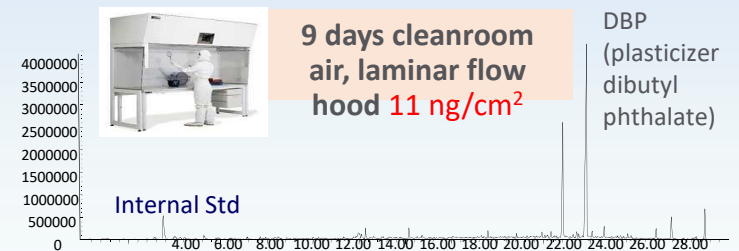
- Organic-free wafers exposed in various environments. Wafers analyzed using TD GC-MS, SEMI MF 1982-1103 Method-B

Results

- Individual shipper provided the best protection – less surface area exposed to the wafer

Conclusion

- Individual shippers provide excellent protection from outside AMC
- FOUP outgassing/carryover issues possible; especially for hot wafers
- Keep wafer exposure in LFH to a minimum

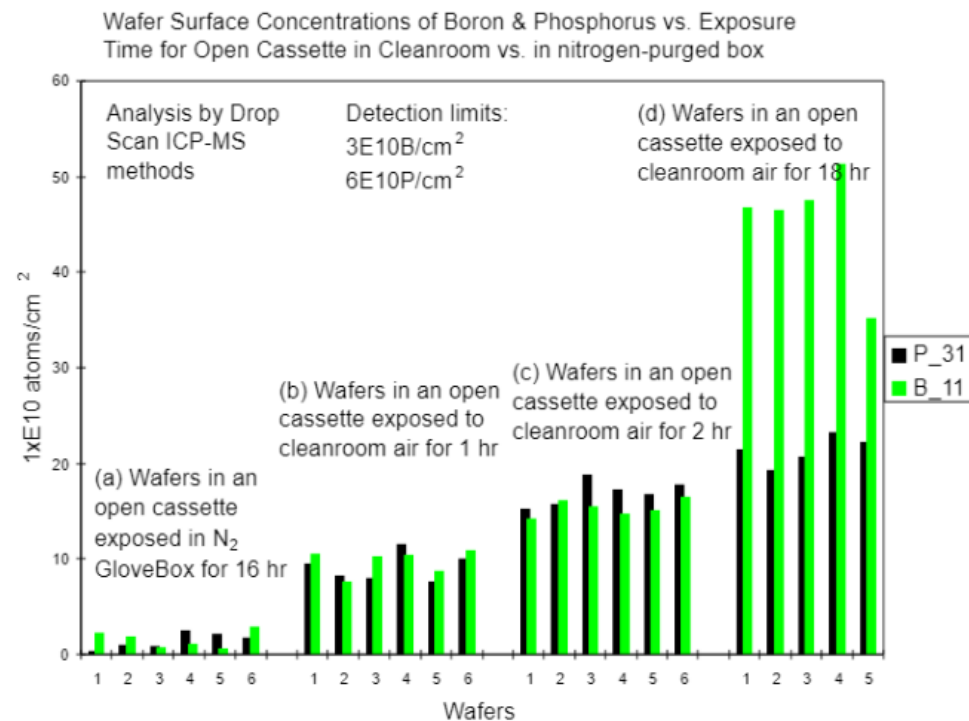


MD: Dopants

Boron likely from HEPA filter, borosilicate glass, exposed to trace HF, forms volatile BF_3 or similar compounds
P likely from fire retardant used in urethanes, such as foams, potting compounds, gel seals

B, P primarily issue for Si Semiconductor Front end fabs. Organop may affect ionizers, UV lasers

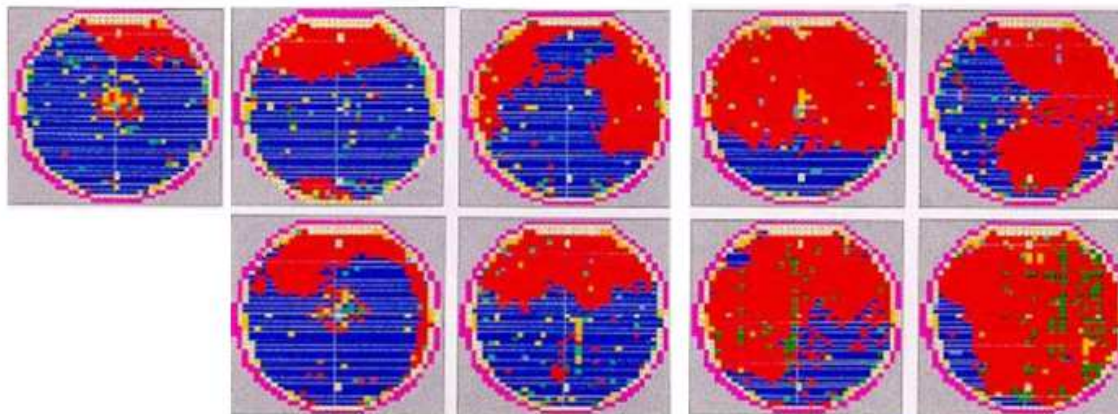
B & P on wafers exposed to air



MD Dopants, esp B from ULPA's, P retardants

Organophosphates Cause Doping Problems

■ Electrical Test Yield Maps



Wafer 23

Wafer 24

Wafer 25

Blue die

Passed

Red die

Failed, n-doped by Phosphorous

Kumar, Ahmed, Camenzind – Micro

- **A new method for Microcontamination Analysis & Control**
 - **Ionizer Tip Analysis: AMCs can react with ionizer tips**
 - **$\text{NH}_3 + \text{Ionizer} = \text{NH}_4\text{NO}_3$ Nanoparticles, IBM, Vaughn Gross**
 - **Silicones + Ionizer, energy, heat, UV, electrons, ions $\rightarrow \text{SiO}_2$**



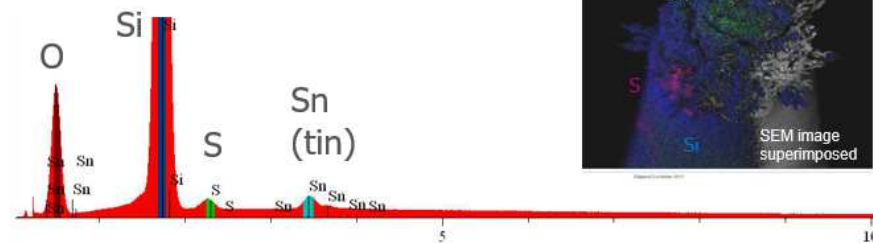
Ionizer Dendrite Analysis

■ Problem indicators

- Dendrites on the tip (see picture)
 - Dendrites can fall from tips
 - Voltage changes as dendrite/ SiO_2 quantity increases
- Excessive nanoparticles in the area if AMCs
- Rapid dendrite formation after tip replacement
- More dendrites in certain areas of fab



Dendrite SEM/ EDS Analysis



Ionizer EDS analysis: 99% SiO_2 , trace S, Sn (tin), from AMC's

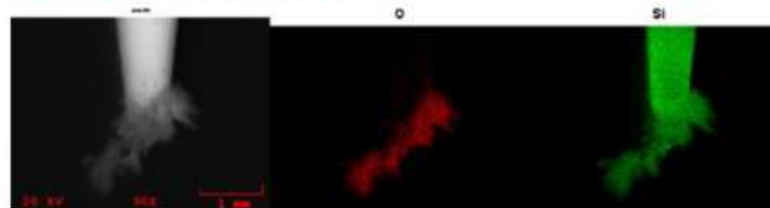
Case Study: Silicon Tip vs. Titanium

- SiO_2 deposits on Ti tip as well: Si in deposits from AMC, not Si tip!

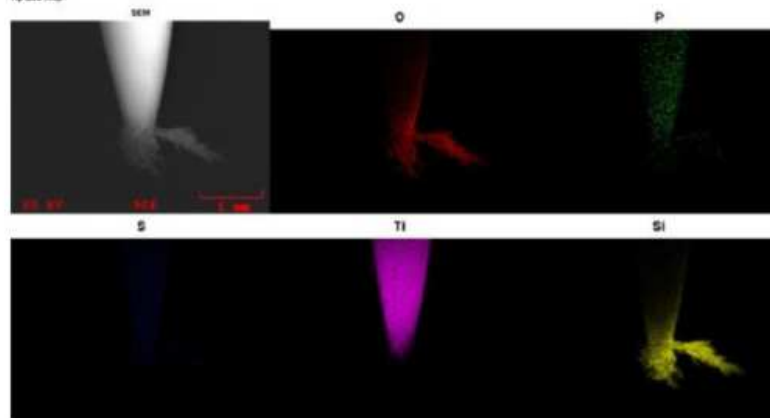
Optical photo:

EDS Elemental Map:

Si tip



Ti tip



**tips from
same room,
in same fab,
same SiO_2
deposits!*

AMCs + Ionizers

- Large AMC literature
 - ISO 14644-8, SEMI.org, IEST.org, Controlled Environments
 - <https://irds.ieee.org/> International Roadmap for Devices and Systems
- Large ESD literature
 - ESDA.org
 - SEMI.org
 - ITRS.net
 - J. Electrostatics
 - Patents, supplier info
 - IEST RP-CC-022.2 Electrostatic Charge in Cleanrooms
 - ANSI/ESD STM 3.1 (IEC 61340-4-7) – Ionization
- Fuzz/deposits issues known for over 25 years
 - Murray, K.D.; Gross, V.P., "Ozone and Small Particle Production by Steady State DC Hood Ionization: An Evaluation," 11th Annual EOS/ESD Symposium, 1989 (IBM)
- No standards to interrelate AMC, ESD, SMC, particles (IRDS)
- High Voltage Corona Ionizers do form ozone, an oxidant AMC compound
- Soft X-Ray ionizers can also form Nano aerosols

Ionizer issue added to the 2017 IRDS International Roadmap for Devices and Systems (<https://irds.ieee.org/>) in the Yield enhancement subsection (https://irds.ieee.org/images/files/pdf/2017/2017IRDS_Yield.pdf):

4.2.3. NEW AMC TOPICS RELATED TO INTERACTIONS OF AIR IONIZERS WITH AMC

Corona based air ionizers may be used to neutralize charge to prevent electrostatic discharge damage and electrostatic particle attraction to wafers, masks or other surfaces.

The hot emitter tip, UV light, energetic electrons and ions formed can interact with AMC's (Airborne Molecular Contaminants) that have Si, S, P, B, Cl, Sn or other selected elements to make nm non-volatile particles such as oxides, and larger deposits or dendrites on the tips up to millimeters, that can throw the ionizer out of balance or lead to ionizer faults. While presumably rare, if this happens, the deposits call attention to the unusual presence of specific excess AMC's that can react with also with other energy sources to, not only deposit onto ionizer tips, but possibly onto other surfaces, due to interaction with excess energy such as 193 nm lithography, lasers and inspection tools or hot surfaces. The compounds might degrade optics, masks, scanners, or other surfaces. Analysis of the ionizer tip deposits by SEM-EDS, ICP-MS (especially for boron) or other methods can indicate what elements are present in air, aiding selection of methods to look for possible sources of a limited number of contaminants, instead of large sections of the periodic table. This method can be used to more rapidly find some AMC issues for which no other test is currently available, such as ppbv and higher leaks of some hydrides, silicones, silanols, halogens, organometallics, O=C=S, TEOS, organophosphates, ammonia, acids, etc.

MM: Now more volatile forms for metals

Elemental Coverage

ALD precursor availability vs Periodic table



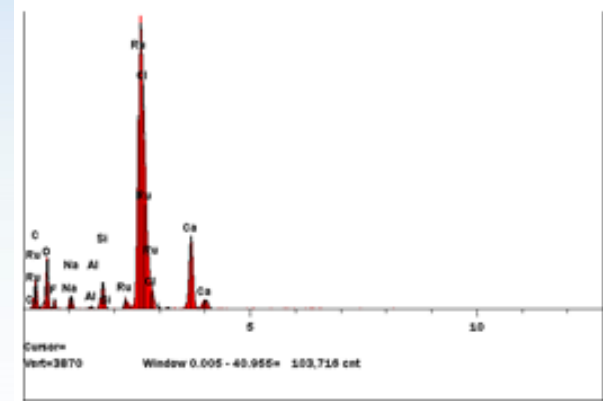
Study Scale																Study Scale									
IA												IIIA		IVB		VB		VIB		VIIB		VIII			
H	He											B		C		N		O		F		Ne			
Li	Be											Al		Si		P		S		Cl		Ar			
Na	Mg	IIIa	IVa	VA	VIA	VIIa	[-----VIIIa-----]		IB	IIB	Ga		Ge		As		Se		Br		Kr				
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	In		Sn		Sb		Te		I		Xe			
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	Hg		Tl		Pb		Bi		Po		At			
Cs	Ba			Hf	Ta	W	Re	Os	Ir	Pt	Au	Pt		Pb		Bi		Po		At		Rn			
Fr	Ra			Rf	Db	Sg	Bh	Hs	Mt	Uun			Uuq												
		La		Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu								
		Ac		Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr								

Film etch by-products can also get into Air, FOUP, EFEMs

Example of volatile precursor, not expected in room

- A Ru compound was being used. Noticed dark spots near area on white walls. Using conductive adhesive sampling, found Ru compound gets into air, ends up doing RT CVD onto room walls, surfaces, thus contamination risk
- EDS suggests perhaps RuOx or similar on wall, 66% w/w Ru, Ru:O ratio approx. 1:1 atomic

Many new organometallics, volatiles being used and can form volatile byproducts transported on wafers, FOUPs, tools...



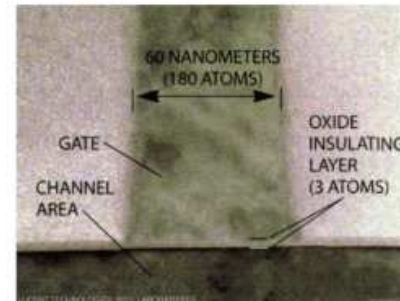
El.	Line	Intensity (c/s)	Atomic %	Conc	Units	Error 2-sig	
C	Ka	29.12	14.057	3.742	wt. %	0.160	
O	Ka	58.06	28.691	10.173	wt. %	0.307	
F	Ka	10.97	4.714	1.985	wt. %	0.138	
Na	Ka	17.84	2.058	1.049	wt. %	0.057	
Al	Ka	4.34	0.256	0.153	wt. %	0.017	
Si	Ka	45.55	2.230	1.388	wt. %	0.047	
Cl	Ka	305.95	11.158	8.767	wt. %	0.115	
Ca	Ka	145.77	7.225	6.418	wt. %	0.122	
Ru	La	385.77	29.611	66.327	wt. %	0.778	
			100.000	100.000	wt. %		Total

kV 20.0
Takeoff Angle 35.0°
Elapsed Livetime 75.4

Sources of Molecular Metals &/or Particles

■ Most metals still originate as particulates

- ✓ From tools, floors, spills, ECP, CMP, ULPA leaks, corrosion, robotics
- ✓ Metals may transport via air or surface contact
- ✓ In the future, more metals/oxides may be used for contacts, salicides, electrodes, gates. Hence, there is a greater awareness to Co, Ni, Ru, RuO₂, Pt, Ir, Hf, Zr, Gd, Sc, and La



Fe on the wafer surface prior to gate oxidation has been correlated with GOI degradation

■ Degradation of air ducts

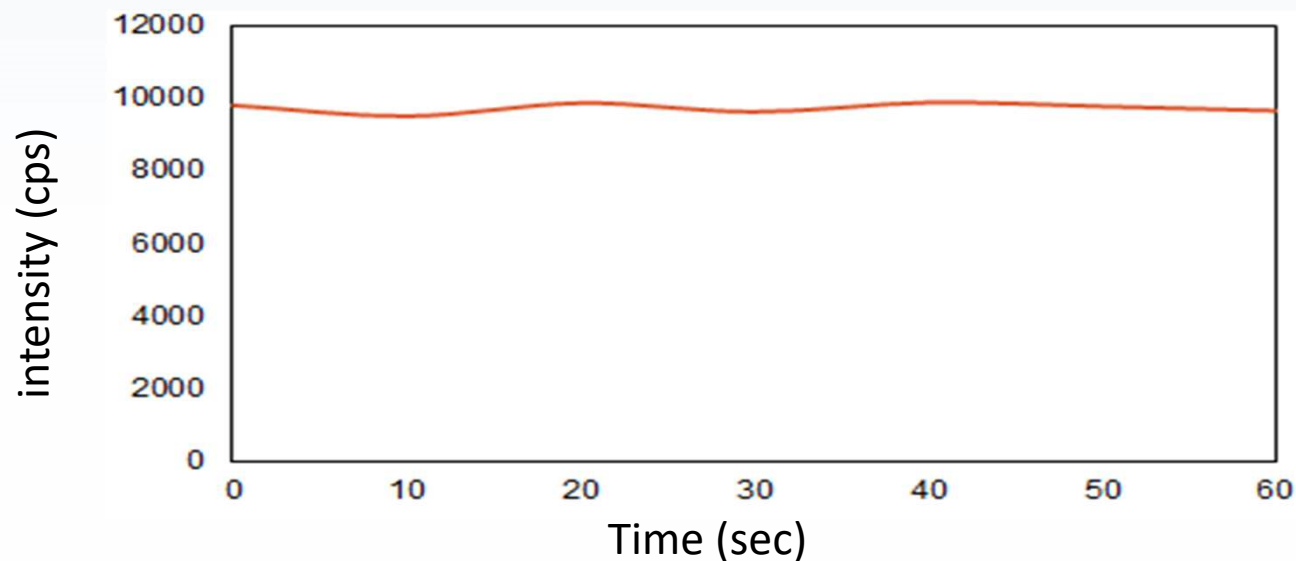
- ✓ Corrosion of passivated (chromium oxide films) stainless steel by HCl resulting in emission of Fe, Cr, and Ni into the airstream
- ✓ Corrosion of galvanized (Zn coated) stainless steel by H₂SO₄ resulting in Zn and Fe emission in airstream



SEM image of corrosion pits and deposits on ventilation duct

Nanoparticle Analysis Fundamentals

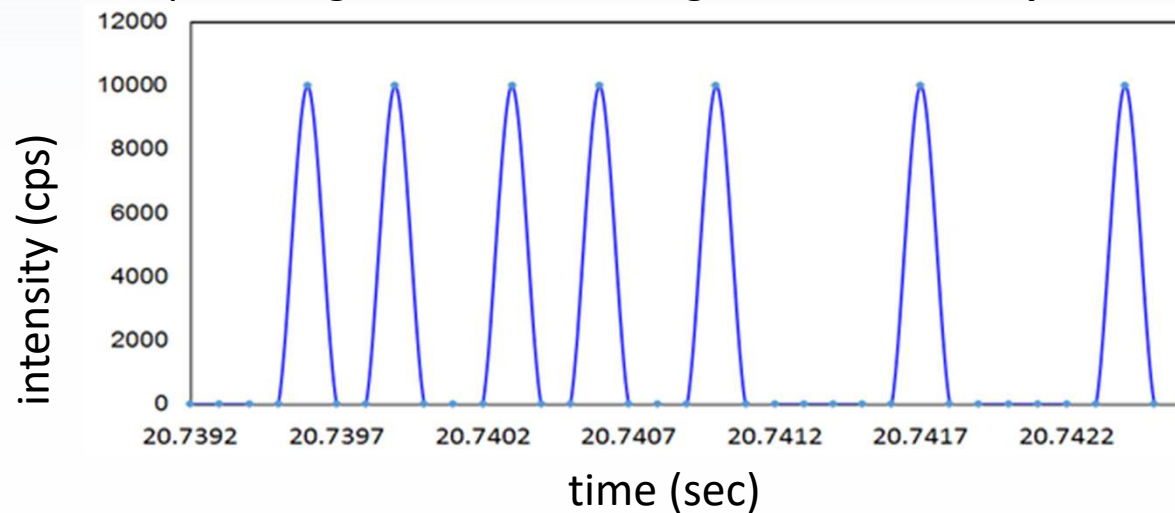
ICP-MS has been used regularly for the determination of trace metals in UPW, process chemicals and extraction solutions from OEM tool parts. The detection limits are often as low as sub parts per trillion (ppt). Since these liquid samples are atomized and ionized into the plasma continuously, producing a constant signal intensity, only total elemental concentration is determined.



Nanoparticle Analysis Fundamentals

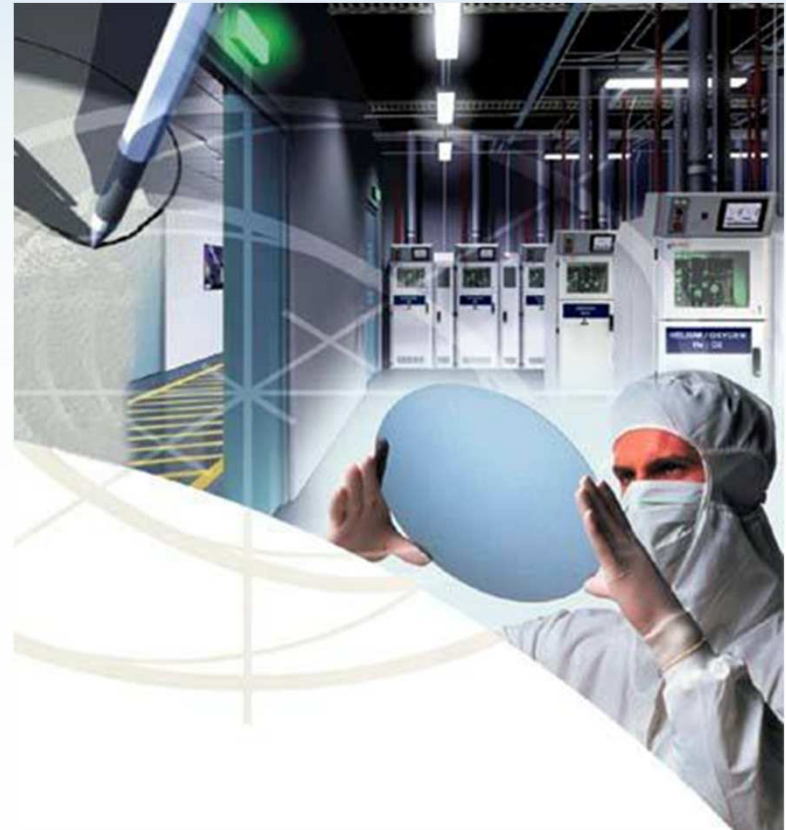
Using time resolved data acquisitions with short dwell times, the transient signal of each individual nanoparticle can be differentiated from the steady-state signal generated by dissolved ionic species. This unique feature coupled with fast electronics enables ICP-MS to characterize nanoparticles down to single nm sizes, producing results in the form of particle concentration, particle median size, and size distribution information.

Each nanoparticle gives a transient signal, collected at **point/0.1 ms**



Typical Nanoparticle Analysis - Figures of Merit

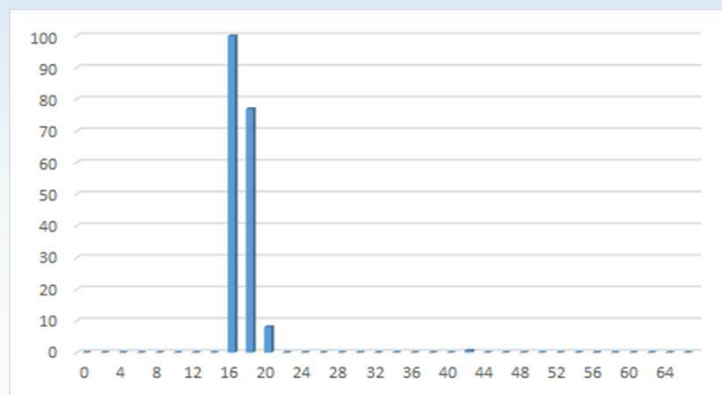
- Particle Concentration (number of particles/mL)
- Ionic Concentration (dissolved concentration; ng/mL)
- Particle Size Distribution
- Median Particle Size; nm
- Elemental Composition of Particles
- BED (background equivalent particle diameter; nm)



UPW Size Distribution: Fe vs. Si

(similar concentration of particles but different median particle size)

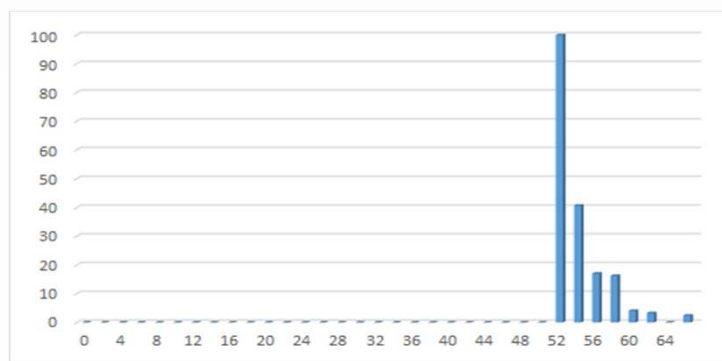
Normalized
frequency



Fe

(75953 particles/mL)

Normalized
frequency



Si

(62420 particles/mL)

FOUP Analysis They provide protection from air, but.....

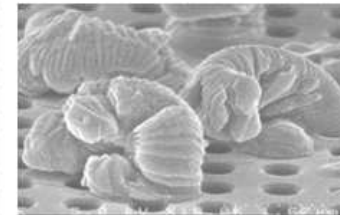
■ Problem indicators

- Cross contamination, process carryover or autodoping of films.
- Metals corroded at bottom of via, Cl induced Al oxidation.
- FOUP material outgassing from reaction with process chemicals.



Analyte	FOUP Background ¹	FOUP Sampling with Wafers	Process Changed
Fluoride (F ⁻)	0.14	348.27	214.65
Acetate (CH ₃ COO ⁻)	0.71	*	3.97
Formate (HCOO ⁻)	0.53	2.46	3.33
Propionate (CH ₃ CH ₂ COO ⁻)	0.00	*	*
Chloride (Cl ⁻)	0.00	9.83	1.52
Nitrite (NO ₂ ⁻)	-0.60	1.26	0.65
Bromide (Br ⁻)	0.00	*	*
Nitrate (NO ₃ ⁻)	0.00	0.21	*
Phosphate (HPO ₄ ⁼)	0.00	*	*
Sulfate (SO ₄ ⁼)	0.00	2.37	0.32
Ammonium (NH ₄ ⁺)	0.17	*	1.23

1. FOUP background requires 2 measurements- 1) FOUP inlet. 2) FOUP with no wafers (can be cleaned or per typical use).



Note: FOUPS do not filter AMC. Purging with N₂ dilutes outgassing.

What levels of AMC are your wafers susceptible to?

- Requirements for FOUP sampling:
 - FOUP with access to interior- hole, nitrogen purge inlet
 - Sample same period of time for each sample.
 - Can see process changes (see table above)
 - Sample time is short due to small volume of FOUP (~30L).

What really matters: Surfaces! Wafer, Mask

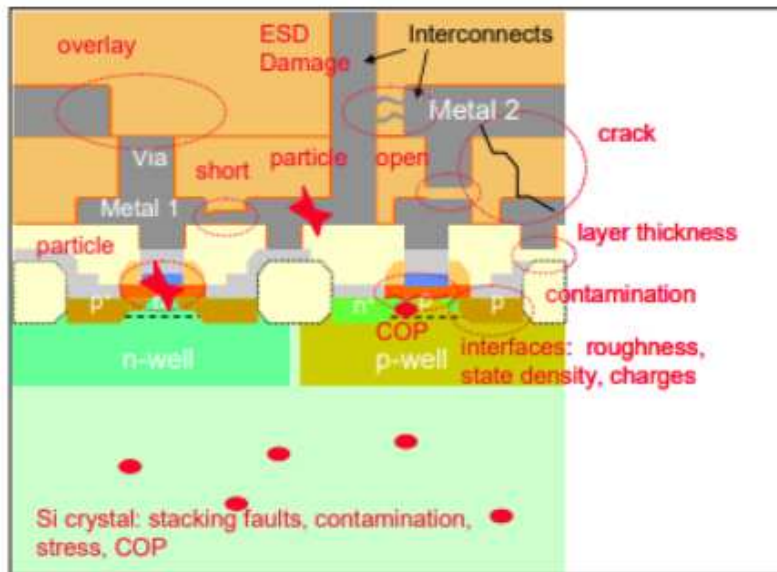


Figure YE3 Yield Enhancement Scope

Hundreds of steps

- Every surface can react w gases, AMCs, water, chemicals
- Each surface can become an interface to next layer
- Many steps not shown, like lithography, ion implantation, inspection, also contamination-sensitive, incl to AMCs



INTERNATIONAL
ROADMAP
FOR
DEVICES AND SYSTEMS

2017 EDITION

YIELD ENHANCEMENT

Other contaminants not covered in SEMI F21

- Ozone, O₃
- Hydrogen peroxide, H₂O₂
- H₂S
- Other guides to AMC classification:
 - ISO 14644-8 includes other categories, bio toxics etc
 - Covers all industries
 - Uses µg/m³ units, not ppbv or pptrv.
 - Can convert if needed, assumes ideal gas, need Molecular weight
 - Considers generation, transport, and sorption of AMCs

- Caveat, Air and CDA have different ISO standards
- CDA = Compressed Dry Air might have particles, oils
- CDA = Clean Dry Air, should be very clean
- CDA = on occasion, “Cruddy, Dirty Air”
- Breathing air can have own standards too

ISO 8573 Compressed Air \neq ISO 14644-1 Air

Compressed Air for particle counts: very different!

ISO 8573

Table 1 — Compressed air purity classes for particles

Class ^a	Maximum number of particles per cubic metre as a function of particle size, d^b		
	$0,1\ \mu\text{m} < d \leq 0,5\ \mu\text{m}$	$0,5\ \mu\text{m} < d \leq 1,0\ \mu\text{m}$	$1,0\ \mu\text{m} < d \leq 5,0\ \mu\text{m}$
0	As specified by the equipment user or supplier and more stringent than class 1		
1	$\leq 20\ 000$	≤ 400	≤ 10
2	$\leq 400\ 000$	$\leq 6\ 000$	≤ 100
3	Not specified	$\leq 90\ 000$	$\leq 1\ 000$
4	Not specified	Not specified	$\leq 10\ 000$
5	Not specified	Not specified	$\leq 100\ 000$
Class	Mass concentration ^b C_p mg/m ³		
6 ^c	$0 < C_p \leq 5$		
7 ^c	$5 < C_p \leq 10$		
X	$C_p > 10$		

^a To qualify for a class designation, each size range and particle number within a class shall be met.

^b At reference conditions; see Clause 4.

^c See A.3.2.2.

ISO 14644-1, Cleanrooms

Max per cubic meter

Class	> 0.1 μm
ISO 1	< 10
ISO 2	< 100

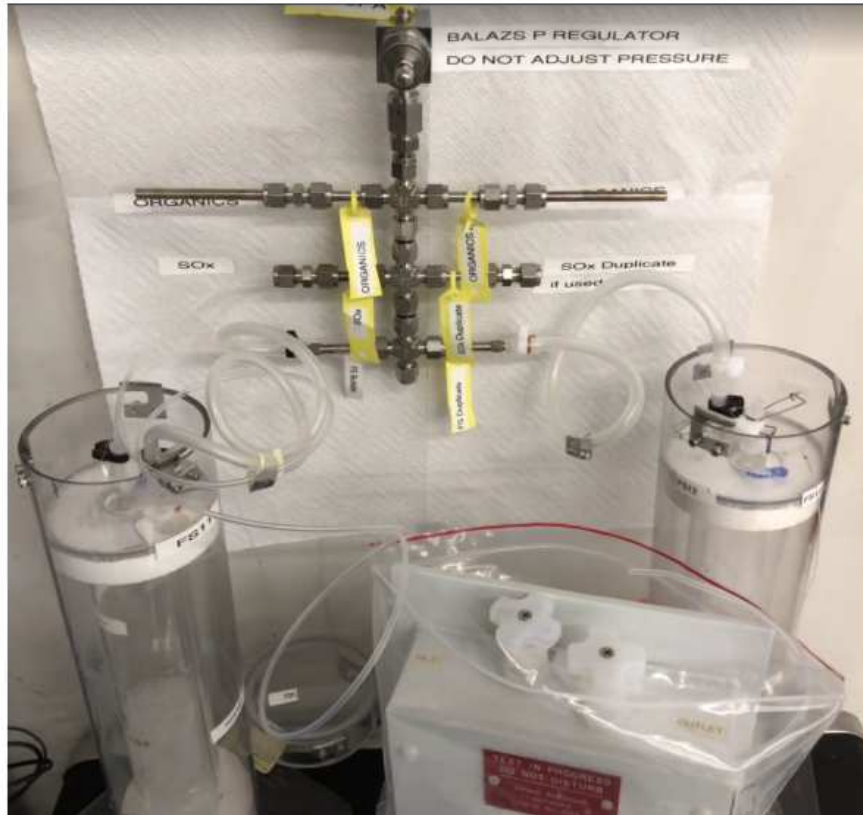
Examples of Analysis Methods

- AMCs, CDA, (XCDA to **10 ppbv**)
- **Pumped sampling** for Air, Minienv, Env chambers, Gloveboxes
- **Manifold sampling** for compressed gases > 20 psig
 - **MA, MB:** Use UPW & bubblers to trap then IC
 - Real-time also possible for some, IMS, CRDS, FTIR
 - **MC:** Use Tenax Sorbent traps for organics, then TD-GC-MS
 - Or witness wafers, **SMOrgs**
Aerospace, glovebox apps also
 - **MD:** IC (PO_4^{3-}), TD-GC-MS, ICP-MS, or witness wafers **SMD**
 - **MM:** Can use bubblers, ICP-MS, or
 - Wafer, VPD-ICP-MS, millionth of monolayer, E9 atoms/cm^2



Wafer Desorption

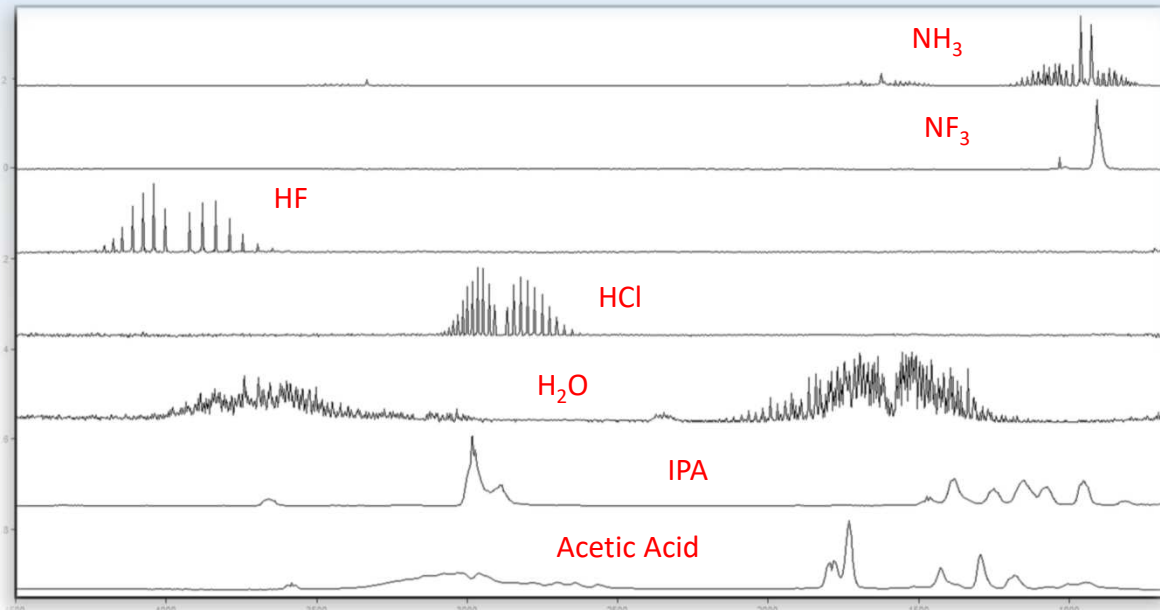
Compressed gas: manifold sampling method From single point



- Pressure regulator
- Tenax GR sorbent tubes
- Fused Silica FS bubblers w UPW, then IC
- SOx bubbler, traps SO₂ using oxidant, makes sulfate, IC

Other methods needed for pptv level DL's

- Real-time monitoring at higher levels for many compounds by FTIR:
 - Originally designed for security applications
 - Balazs was the first to adapt this for cleanroom air monitoring



- ❖ Real-time (1-4 min updates)
 - ❖ Sensitive (<1 to 100 ppbV)
 - ❖ Portable
 - ❖ LN₂ not needed
- Other methods needed
for pptv level DL's

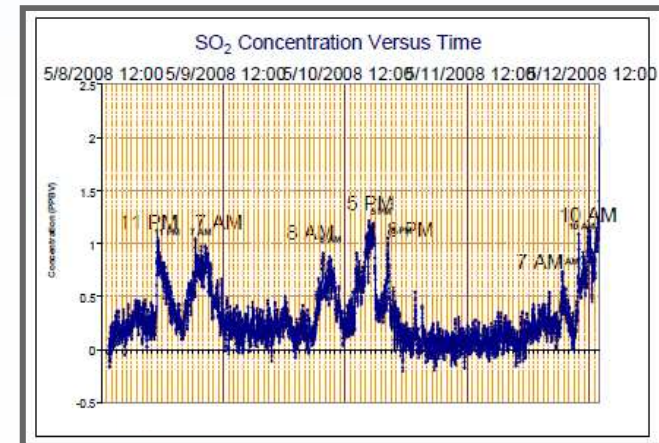
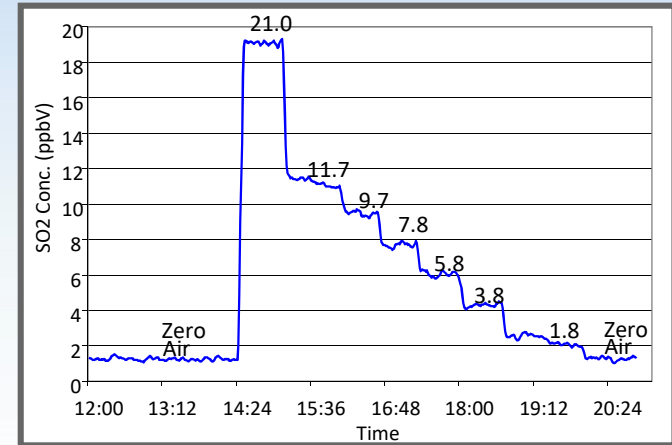
SO₂ and total-sulfur analyzer



SO₂ and total-sulfur analyzer

- Reporting limit is 200 pptV for SO₂ and/or total sulfur
- Instrument challenge of SO₂ to determine its sensitivity (top right)
- SO₂ concentration in cleanroom make-up air (MUA) over a period of time (lower right)

Below 200 pptrv, grab sampling methods needed, and can go to 5-10 pptrv using bubblers, oxidant, ion chromatography



- Similar concepts apply to manufacturing disk drives, lasers, optics, detectors, microfluidics, DNA chips, MEMS, telecom, Nanotech, Nano photonics, LIDAR, VCSELs, Data Centers
- Other environments need similar tests:
 - Gloveboxes, Environmental test chamber, AMC, SMC:
 - Particles residues collected by swabs, wipes, filters, adhesive stubs, witness wafer for sampling, then analysis
- Pharma also care about particles, esp viables, contamination analysis in isolators
- Filter autopsies at end of life useful to assess filter challenge for particles, AMCs, ammonium salts, oils: very sensitive since online for months to years, vs days for test filter

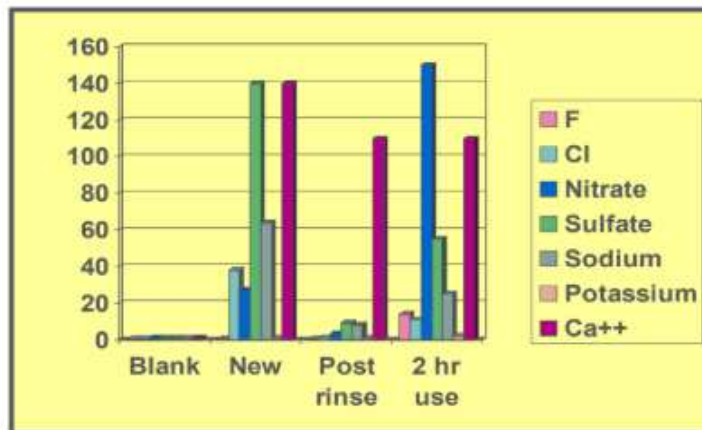
- Application of concepts for similar tests of T, RH test chambers w hot, humid air: must be sure no contamination from wafer, air, CDA, room, outgassing, cross contamination of parts under test:
- Medical devices
- Consumer electronics, cell phones, laptops, smart watches
- Military, Aerospace, satellites, cameras, IR detectors, LEDs
- Hot humid air sampled via ice chilled coils to collect AMCs incl acids, ammonia into bubblers, then ion chromatography to ppbv levels
- Can sample organics, metals onto witness wafers



- Overall must balance overall contamination from air vs other sources, people, used gloves, used garments, water, chems

Clean gloves are important

- Can transfer organics (lubricant), particles, ionics and metals on contact
- Can shed particles onto products from excessive abrasion
- Can transfer glove contaminants through a wetted wipe onto the product surface



Many ISO 14644 standards for cleanrooms

Document Number	Title of Part	Date for next action	Action
ISO 14644-1:2015 (Ed. 2)	Classification of air cleanliness by particle concentration	-	Standard published
ISO 14644-2:2015 (Ed. 2)	Monitoring to provide evidence of cleanroom performance related to air cleanliness by particle concentration	-	Standard published
ISO 14644-3:2005 (Ed. 1)	Test methods	-	Standard published; Ed. 2 in progress
ISO/DIS 14644-3 (Ed. 2)	Test methods	6/8/2019 (IS)	FDIS
ISO 14644-4:2001 (Ed. 1)	Design, construction and start-up	-	Standard published; Ed. 2 in progress
ISO/AWI 14644-4 (Ed. 2)	Design, construction and start-up	-	CD
ISO 14644-5:2004	Operations	-	Standard published
ISO 14644-7:2004	Separative devices (clean air hoods, gloveboxes, isolators and mini-environments)	3/4/2019	Systematic Review
ISO 14644-8:2013 (Ed. 2)	Classification of air cleanliness by chemical concentration	-	Standard published
ISO/PWI 14644-8 (Ed. 3)	Classification of air cleanliness by chemical concentration	-	NWIP
ISO 14644-9:2012 (Ed. 1)	Classification of surface cleanliness by particle concentration	-	Standard published
ISO/PWI 14644-9 (Ed. 2)	Classification of surface cleanliness by particle concentration	-	NWIP
ISO 14644-10:2013 (Ed. 1)	Classification of surface cleanliness by chemical concentration	-	Standard published
ISO/PWI 14644-10 (Ed. 2)	Classification of surface cleanliness by particle concentration	-	NWIP
ISO 14644-12:2018	Specifications for monitoring air cleanliness by nanoscale particle concentration	-	Standard published
ISO 14644-13:2017	Cleaning of surfaces to achieve defined levels of cleanliness in terms of particle and chemical classifications	-	Standard published
ISO 14644-14:2016	Assessment of suitability for use of equipment by airborne particle concentration	-	Standard published
ISO 14644-15:2017	Assessment of suitability for use of equipment and materials by airborne chemical concentration	-	Standard published
ISO/DIS 14644-16	Code of practice for improving energy efficiency in cleanrooms and clean air devices	12/9/2019 (IS)	FDIS
ISO/AWI 14644-17	Particle deposition rate application	-	CD
ISO/PWI 14644-18	Assessment of cleanliness suitability of consumables	-	NWIP
ISO 14698-1:2003	Biocontamination control -- Part 1: General principles and methods	10/15/2019	Systematic Review
ISO 14698-2:2003 and Cor 1:2004	Biocontamination control -- Part 2: Evaluation and interpretation of biocontamination data, and Technical Corrigendum 1	10/15/2019	Systematic Review

Table 1—Projects under the ISO/TC 209 title “Cleanrooms and associated controlled environments”

Other Standards Organizations

- ASHRAE.org, esp. for corrosion of electronics using coupons
- SEMI.org, esp. for semiconductors, gases, cleanrooms
- IDEMA.org: disk drive industry standards incl outgassing
- IEST.org, Standards and ESTECH conferences, design, outgassing, AMCs, NVR (Non Volatile residue), IEST 1246E
- ASTM.org, outgassing
- JACA: Japan Air Cleaning Association (<http://www.jaca-1963.or.jp/en/>)
- Controlled Environments magazine (www.cemag.us)
- SPCC: The Surface Preparation and Cleaning Conference
 - <https://www.linx-consulting.com/spcc/>

ACKNOWLEDGMENTS

- Victor Chia, PhD of Balazs NanoAnalysis for sharing selected slides
 - Balazs Staff worldwide
 - Selected clients who chose to remain anonymous
 - Phil Spate of Environmental Protocol Inc for sharing data center Zn whisker example
 - IEST.org for use of selected figures from Balazs NanoAnalysis presentations at previous ESTECH conferences, 2006, 2012, 2014...
-
- There is a lot to do on contamination control, beyond particle counts!
 - Any questions, comments, concerns, examples to share?
-
- Contact Hugh.Gotts@AirLiquide.com
 - or Mark.Camenzind@AirLiquide.com
 - Main 510-624-4000