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Process Tool Cleanliness for Clean Manufacturing

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Abstract – **Wafer tool cleanliness specifications are generally accepted for various processes and technology nodes. However, the material and component cleanliness required to meet these wafer tool cleanliness is vague. One reason is the lack of uniformly accepted parts cleanliness tests. This paper reviews and recommends test methods for determining the cleanliness of coupons, first articles and production parts.**

I. INTRODUCTION

Process tools are designed primarily for their function. However, tools with applications for small technology nodes must have cleanliness as the driving force over design concept. Material selection based on their cleanliness potential is a prerequisite for manufacturing high throughput tools. The source of process contamination is frequently from the tool components. Therefore, it is important to use pure starting materials and coatings, following contamination-free handling and packaging procedures and precision cleaning of parts.

Acceptable wafer contamination level in a completed tool depends on the process and technology node it was designed for. The purity of materials, such as ceramic, quartz, plastic, aluminum and stainless, is important because bulk impurities can sputter off or outgas onto the wafer surface during processing. Baselining and tracking parts surface cleanliness after precision cleaning can prevent yield losses by reducing the risk of tool contamination excursions.

This paper describes analytical test methods for quantifying bulk materials and part's surface contamination. These tests may be used to establish the build of material (BOM) cleanliness specifications, qualify material suppliers, evaluate precision cleaning houses, and to identify stains and residues on tool parts.

II. BULK MATERIAL CHARACTERIZATION

Bulk material cleanliness is an important criterion as bulk impurities can sputter recoil onto the wafer surface during ion implantation or during reactive ion etching (RIE). Studies have shown Al and other metals impurities from the target wheel of ion implanters have contributed to wafer surface Al detected by VPD ICP-MS [1]. Exposed parts were coated with graphite or plasma sprayed silicon to negate this contamination source [2]. Common bulk analytical methods providing ppb detection limits include:

a) Acid digestion ICP-MS
b) Laser ablation ICP-MS (LA ICP-MS)
c) Glow discharge-MS (GD-MS)

Bulk materials characterization is obviously a destructive test as the total material is consumed during the analysis. No depth distribution information of the impurities is provided. Analytical techniques such as glow discharge optical emission spectroscopy (GD-OES), LA ICP-MS and secondary ion mass spectrometry (SIMS) may be used to provide in-depth distribution of the key contaminants observed.

Laser ablation ICP-MS is becoming the technique of choice for bulk material qualification because the analysis requires no sample preparation [3]. Acid digestion and GD-MS require specialized sample preparation that can result in contamination if care is not taken. An advantage of LA ICP-MS is its capability to also operate in a depth profiling mode. In brief, LA ICP-MS involves the conversion of solid materials into a plume of neutral atoms, ions, and micro particles by focusing a high-powered and pulsed laser beam onto the surface. The plume of materials is transported into an ICP (~10,000 K) for efficient atomization and ionization. The atomic ions formed in the ICP are subsequently analyzed by a mass spectrometer.

Figs. 1 and 2 show LA ICP-MS survey spectra acquired from an aluminum coupon and O-ring. The detected bulk impurities are common metal impurities found on wafers after processing. Quartz, ceramics, polymers, solders, alloys and metals may be analyzed by LA ICP-MS.

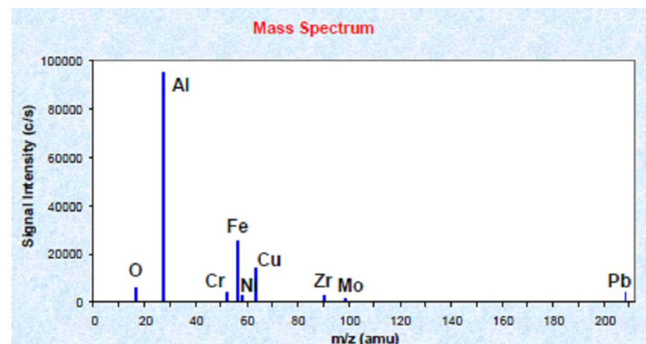

Fig. 1. Laser ablation ICP-MS bulk survey spectrum of an aluminum coupon.

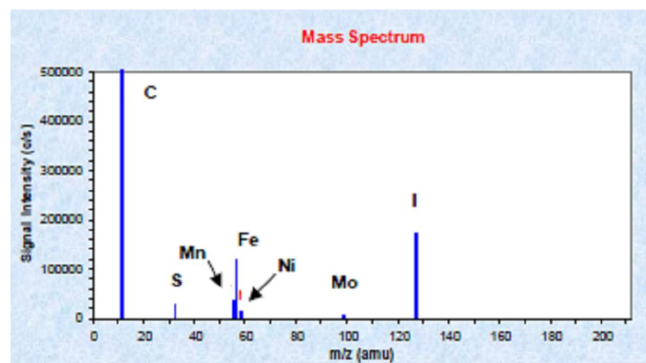

Fig. 2. Laser ablation ICP-MS bulk survey spectrum of an O-ring.

LA ICP-MS can operate in a depth profiling mode to quantitatively provide major composition and impurity distribution information. Fig. 3 shows a depth profile acquired from a ceramic coupon with a total penetration depth of approximately 2 µm.

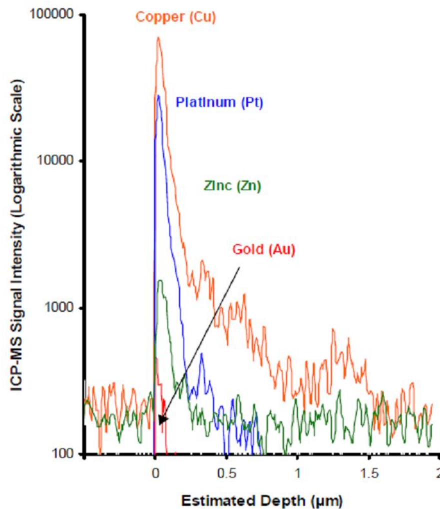

Figure 3: Laser ablation ICP-MS depth profile of ceramic coupon.

Organic airborne molecular contamination (AMC) becomes surface molecular contamination (SMC) with a negative impact on several IC processes [4, 5]. For example, the organic film influences the growth rate of thin gate oxides and the quality of epitaxial films grown at low temperatures. If heated rapidly, the residue hydrocarbon contaminants form silicon carbide (SiC), a compound almost impossible to remove. Lithography and ellipsometery thin film metrology tools are dramatically affected by organic contamination resulting in optic hazing from silicones and plasticizers, for example.

Sources of organics include cable sheaths, isolation pads, lubricants, O-rings (Viton, Kapton), plastics (PEEK, PE, PTFE), epoxy/adhesives, gloves pinched between components during PM, fingerprints, and photoresist on wafers. Even stainless steel can outgas sulfur. Organic compounds can outgas from the bulk and surface of these materials at room temperature and the outgassing process may be accelerated at elevated temperature and in high vacuum in the tool.

The recommended test method for bulk organic is IEST RP-CC-WG031 – "Method for semi-qualitative analysis of outgassed compounds from cleanroom materials and components." This recommended practice (RP) provides a method for preliminary screening of detectable outgassed compounds in materials. It is valuable for incoming materials quality control (QC) and lot-to-lot consistency. Essentially the test is performed using a thermal desorption gas chromatography mass spectrometer (TD GC-MS) with temperature programming of 50 C, 75 C, 100 C, or 150 C for 30 minutes, helium carrier gas flow rate 3 mL/min and mass spectrometer with mass detector range 33-550 amu. The desorption or outgassing temperature selected shall be the temperature that is the next highest to the material's application temperature. So, if the material is to be used at room temperature the material shall be outgassed at 50 C. The detection limit of TD GC-MS is suitable for this test as demonstrated in Fig. 4 that shows a TD GC-MS spectrum of refractory organic compounds including S, P, Si compounds detectable to <100 pptV versus an internal standard of 100 pptV.

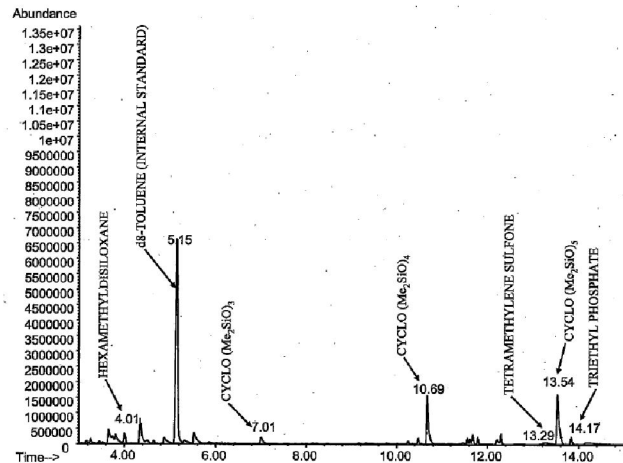

Figure 4: TD GC-MS spectrum of refractory organic compounds.

III. SURFACE COMPONENT/PART CHARACTERIZATION

Parts cleaning is a highly complex process that is required for advanced process tools used throughout the entire manufacturing process, FEOL and BEOL [6]. For >250 nm technologies parts cleaning only required the parts to be cleaned following a recipe. However, for <90 nm technologies experimental cleanliness verification is required to ensure the tool meets the ITRS roadmap for enhanced performance. This is particularly important with the introduction of new materials and coatings, particularly at 45 nm and below.

Parts cleanliness verification dictates non-destructive surface analytical methods must be used as the part is to be returned and installed in the process tool. Ultra-pure water, chemicals and solvents are ideal extraction solutions to remove surface contaminants because; a) their chemistry is well known and documented; b) they are effective in removing surface contamination with minimal substrate material loss; c) their use is not dictated by the size of the part, and; d) they result in negligible damage to the surface enabling the part to be returned to the field after testing.

Table 1 summarizes the most frequently used non-destructive test methods. The extraction solution recipes may be customized for a specific material and coating or target a specific contaminant. Key points of Table 1 are summarized below.

1. Metal: typically whole surface extraction; some cosmetic stain may result
2. Metal: typically whole surface extraction; collection efficiency is less than using acid
3. Metal: localized surface scan using acid
4. Organic: solvent extraction for organic residue; UPW can be used for TOC measurement
5. Organic: solvent extraction for weight (NVR) and organic identification with FTIR
6. Ionic: typically whole surface extraction
7. Particle: extraction sometimes using sonification, typically whole surface extraction and particle composition identification with EDS

TABLE I
NON-DESTRUCTIVE SURFACE ANALYSIS TECHNIQUES

Metal	Acid extraction & ICP-MS	1
	UPW extraction & ICP-MS	2
	Drop scan etch & ICP-MS	3
Organic	Solvent extraction & GC-MS	4
	Solvent extraction & NVR/Raman	5
Ionic	UPW extraction & Ion Chromatography	6
Particle	UPW extraction & LPC (EDS)	7

It is important to note the experimental extraction or leaching condition must be identical for lab-to-lab comparison. This is because the extraction efficiency depends on the experimental condition used. For example, the extraction may be performed at ambient temperature for 1 h to 1 day, at elevated extraction temperature for 1-2 h, or at 85 C for 7 days as in SEMI F-57.

Table II shows UPW extraction results acquired at ambient temperature for 7 days of three piping materials, namely polyvinylidene fluoride (PVDF), polypropylene (PP) and polyvinyl chloride (PVC). It is evident a pre-clean procedure

TABLE II
ULTTRA-PURE WATER EXTRACTION OF PIPING MATERIALS

Element	DL (Detection Limit) ppb	High Purity PVDF	High Purity PP	High Purity PVC
TOC	5	90	94	50
Fluoride	2	77	*	*
Chloride	0.25	1.0	0.66	49.54
Aluminum	0.05	2.3	0.68	1.16
Barium	0.01	0.24	0.09	0.05
Calcium	3	*	12	15
Magnesium	0.02	0.66	1.0	2.17
Sodium	0.06	0.51	0.18	23.22
Tin	0.02	*	*	1.19
Zinc	0.06	0.47	0.96	1.19

is required for PVDF and other fluorinated polymer materials to reduce the fluoride contribution before use. For certain applications it is best to avoid using this material such as tubing used in air impinger samplers to monitor airborne molecular fluoride, a molecular acid (MA). Even after extensive leaching of PVDF tubing some level of fluoride is detected by the air sampler that is attributable to the tubing.

Fig. 5 illustrates an irrigation method to selectively extract surface contaminants from a particular area. This protocol can be performed without cutting or damaging the part and may be used on small parts as well as large solar panels. Ultra-pure water, chemical or solvent may be used depending on the type of contaminant – metal, ionic, or organic. The extract is then analyzed by the techniques shown in Table I.

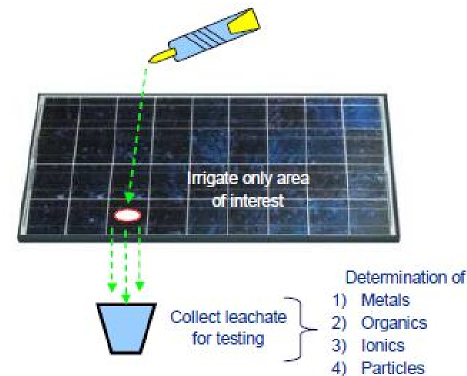

Fig. 5. Irrigation extraction method to collect localized surface contaminants

XPS (x-ray photoelectron spectroscopy) is a surface analytical technique commonly used to determine the cleanliness of coupons. Parts cannot be analyzed by XPS unless it is cut to fit into the XPS sample chamber; a typical sample size requirement is 2" x 2" x ¼". The detection sensitivity of XPS is about 0.1 atomic%. Despite this, XPS is used as a Pass/No Pass test to verify the efficacy of cleaning recipes because it is the only technique that provides chemical state information. Fig. 6 shows XPS spectra of two ceramic coupons cleaned by two cleaning vendors. It is evident from the spectra different cleaning recipes were used resulting in different surface states after cleaning.

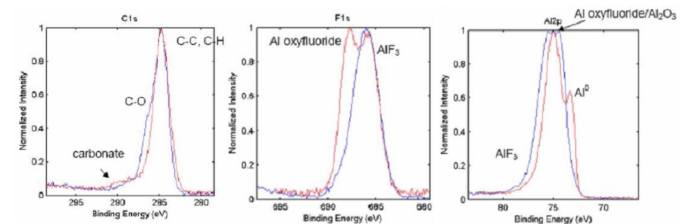

Fig. 6. XPS spectra of two ceramic coupons cleaned by two cleaning vendors.

Other surface techniques used include UV (black) light for visual inspection for residue polymer on the surface and

profilometry for surface roughness measurement after cleaning, as cleaning smoothes the surface through atomic layer removal. For example, ceramic etch domes require some roughness to be effective for photoresist adherence and therefore surface roughness is one component of its cleanliness specification.

IV. CASE STUDY

After a performing a preventive maintenance (PM), the tool base pressure was higher than usual and Cl was detected by TXRF on a witness wafer at $5x10^{13}$ at/cm^2 instead of $5x10^{10}$ at/cm^2 that is normally observed and meets specification. The metal concentrations measured by TXRF were at or below the wafer metal specification of $5x10^{10}$ at/cm^2.

This has never happened before and was a complete surprise. The first step to determining the root cause is to characterize the Cl – – Is it inorganic or organic in nature? Just knowing it is Cl is not helpful in determining the root cause. If the Cl is inorganic and present as Cl^- then the root cause may be insufficient rinsing of tool parts during PM as HCl was a constituent of a cleaning solution. This may easily be remedied by rinsing all the parts again that were cleaned. If the Cl was organic then the root cause may be an excess of lubricant or a pinched glove in a gasket seal, for example.

Another interesting data was, a static wafer left in the chamber for 1h showed no Cl by TXRF. In contrast a dynamic wafer cycling test, with a wafer cycled through the tool from the FOUP and back multiple times, showed surface Cl at $5x10^{13}$ at/cm^2 that was reproducible. This experimental observation will reveal its significance once we identify the contamination source.

The recommended test for inorganic Cl on a wafer is UPW extraction followed by ion chromatography (IC) with detection sensitivity in the ppb range – see Table I [7]. Full Wafer TD GC-MS is ideal for determining if the Cl is organic using SEMI MF 1982-1103 Method-B. These two tests were performed on individual wafers. The IC results revealed no Cl^- was present and Full Wafer TD GC-MS showed the Cl to be a form of organo-chloride compound. The conclusion is that no amount of parts rinsing would have resolved the Cl contamination escalation.

The organo-chloride compounds were identified as common flame retardant constituents. Flame retardants are often used on foam cushions, sofas and beds to prevent them from catching on fire. After reviewing the build of material (BOM), the root source of the organo-chloride compound was suspected to be from the vibration isolation pads. The BOM specified black pads but the tool was installed with blue pads. These two types of pads were analyzed by TD GC-MS and the results are shown in Figs. 7 and 8. The outgassed organic compounds from the blue isolation pad (Fig. 8) matched the wafer organic outgassed spectrum.

The static and dynamic wafer test results by TXRF now become clear. No water leaks if you hold a wet sponge. However, if you squeeze the sponge lightly or even shake the sponge with your hand some water will leak out. This is the case with the vibration isolation pads. When the wafer handler is static the isolation pads are not active and do not outgas. In contrast, when the wafer handler is moving and transporting wafers the pads are adsorbing vibrations produced in the process and they react by compressing and outgassing. So, even though the design specification for vibration insulation was met with the blue pad its bulk properties were not characterized and resulted in a contamination escalation.

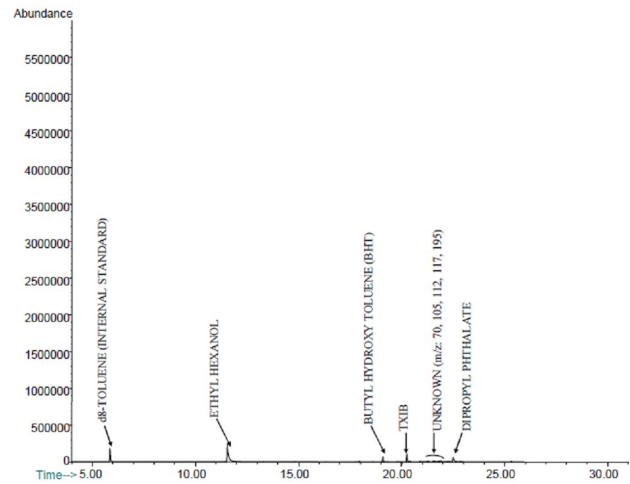

Fig. 7. TD GC-MS of a black vibration isolation pad.

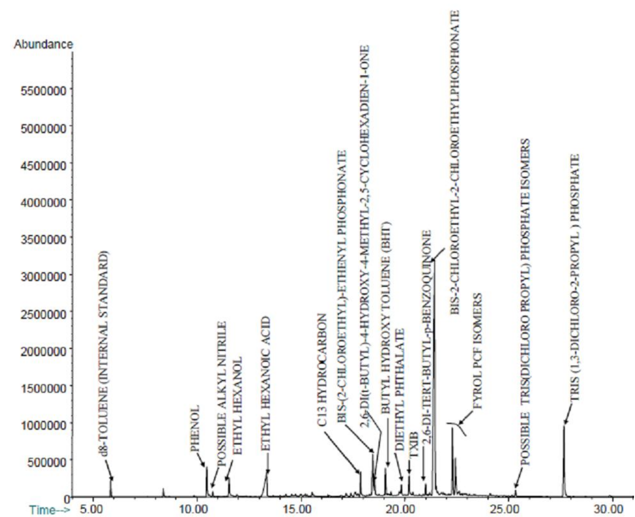

Fig. 8. TD GC-MS of a blue vibration isolation pad.

V. COMPLETED PROCESS TOOL CLEANLINESS

Tool cleanliness specifications, determined by witness wafers for particles, metals and organics, are becoming more stringent with decreasing technology nodes. It is important to ensure parts cleanliness specifications are in place so cleaning vendors and supplier chain vendors understand what is

required of them. It is possible to achieve the required wafer specifications shown in Tables 3-5 with a stringent parts cleanliness program.

TABLE III
PROCESS TOOL PARTICLE SPECIFICATION

Technology Node	Analytical Test	Particle Specification		
		Wafer Front Side	Wafer Back Side	Tool Surface
>90 nm	Full pipeline test, 6 wafers, 150 cycles, KLA SP2	0.2 @ 90 nm (0.0002/cm² pwp)	**Full Contact** <1500 @ 90 nm (2.8/cm² pwp) **Low Contact** <500 @ 90 nm **Edge Contact** <20 @ 90 nm	**Non-Critical Surface** ≤10/in² @ 0.3 µm **Critical Surface** ≤1/in² @ 0.3 µm
90 nm				
65 nm				
<45 nm				

TABLE IV
PROCESS TOOL ORGANIC SPECIFICATION

Technology Node	Analytical Test	Organic Specification
		Organic component Sum ≥C7 tested in Dynamic Mode
>90 nm	1) Load Lock Partition test: 1x2 wafers, 200 cycles (100 cycles each), ~ 30 mins exposure/wafer. 2) Organic Pipeline test: 2x2 wafers, 120 cycles (30 cycles each), ~20 mins exposure/wafer. 3) Full wafer outgassing by TD-GC-MS	≤20 ng/cm²
90 nm		≤20 ng/cm²
65 nm		≤15 ng/cm²
<45 nm		≤10 ng/cm²

TABLE V
PROCESS TOOL METAL SPECIFICATION

Technology Node	Analytical Test	Metal Specification
	Full Pipeline test, 6 wafers by 100 cycles	
>90 nm	VPD-ICP-MS	≤5E11 at/cm² per metal
90 nm	VPD-ICP-MS	≤1E10 at/cm² per metal
65 nm	VPD-ICP-MS	≤1E10 at/cm² per metal
<45 nm	VPD-ICP-MS	≤5E9 at/cm² per metal

VI. CONCLUSION

All tool components and parts must be designed using materials that are compatible to both its function and cleanliness. This means individual tool parts in the completed tool must have cleanliness specifications for its technology node. The smaller the technology node the cleaner the tool components and assemblies must be.

Tool cleanliness is a prerequisite for increased production ramp up, reduced tool down-time and higher production yields. Process tools are qualified to be clean by monitoring witness wafers for particles, metals, ionics and organics after processing. Any materials used in the tool build of material (BOM) can contribute to the wafer contamination level. It is therefore important to verify the parts and assemblies are clean, both in the bulk of its material and on its surface after cleaning, packaging and handling.

Bulk analysis is destructive due to the nature of the test and is usually performed on coupons and first article products. Laser ablation ICP-MS for elemental analysis and TD-GC-MS for organic analysis are recommended bulk techniques. In contrast, surface analysis using aqueous, chemical or solvents is recommended for certifying production parts as surface extraction techniques are non-destructive allowing the parts to be re-used after cleanliness verification.

The case study illustrates the consequence of not having a part cleanliness specification and relying only on a part number. OEM, Fabs and supply chain management operating with parts cleanliness specifications in place will show an increased profit margin through increased tool up-time and productivity.

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