Contamination Troubleshooting for Microelectronics Packaging

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Abstract
A goal of advanced packaging design performance is to reduce power and to achieve better control of heat and electromagnetic interference. Materials to achieve efficient packaging include the use of gold (Au) wire, copper (Cu) alloy, gold/silver (Au/Ag) plating, solder, low-k epoxy and dry-film polymers, silicones and polyimides. Material purity verification and contamination control during the production process is a prerequisite to ensure high yields in packaging because getting this wrong means throwing away multiple chips. This paper describes an Analytical Decision Tree to guide methodology selection, reviews contamination troubleshooting methodologies, and case studies to resolve process issues.

Key words
Analytical techniques, cleanroom, contamination, impurity, microelectronic package, troubleshooting

I. Introduction

Contamination control is important throughout the packaging process. When contamination results in quality failure, the defect type requires identification, after which an effort to find the source of the defects and to fix the root source and cause of the problem is required [1].

There are many possible types and sources of contamination [2] that include (i) environmental induced defects from cleanrooms, cleanroom suits and high efficiency particulate air (HEPA) filters, (ii) particle defects from process chemicals, ultrapure water, packaging and consumables, (iii) handling errors from automation and static electricity control, and (iv) equipment-induced defects from the process equipment.

Root cause resolution of the contamination source can improve yield in the long-term [3]. The purpose of contamination troubleshooting is to identify and eliminate major yield limiters. This requires the use of a variety of analytical techniques. Most important, it requires an understanding of analytical techniques and results, and the principle approach to contamination troubleshooting.

We will review key analytical techniques to characterize and quantify contamination in clean microelectronic packaging operations. We will discuss an approach to determine the root source and possible cause of the contamination.

II. Analytical Decision Tree

Defects are events that lead to or can lead to product failures. Their detection requires extensive use of off-line and in-line inspection tools, in situ sensors, and electrical testing. While defect characterization requires sophisticated failure analysis equipment, the selection of which failure analysis technique to use depends on the nature of the sample and the desired information.

The determination of a defect root cause is usually the most time consuming and costly component of yield improvement efforts [4]. Defect types like particles are typically not too difficult to diagnose because they are often still present during in-line inspection. The particle composition can be determined with a screening technique, such as Scanning Electron Microscopy with Energy Dispersive Spectroscopy (SEM-EDS). SEM provides the visual “answer” while EDS provides the elemental “answer.” In contrast, some defect types are difficult to diagnose because they occur sometime during a process cycle and takes on some distorted shape or appearance that can further complicate diagnosis.

Contamination troubleshooting is a multidisciplinary process and includes visual inspections, metallographic, environmental and chemical analysis, and simulation tests. An analytical test plan can utilize a number of analytical techniques ranging from traditional wet chemical analysis to

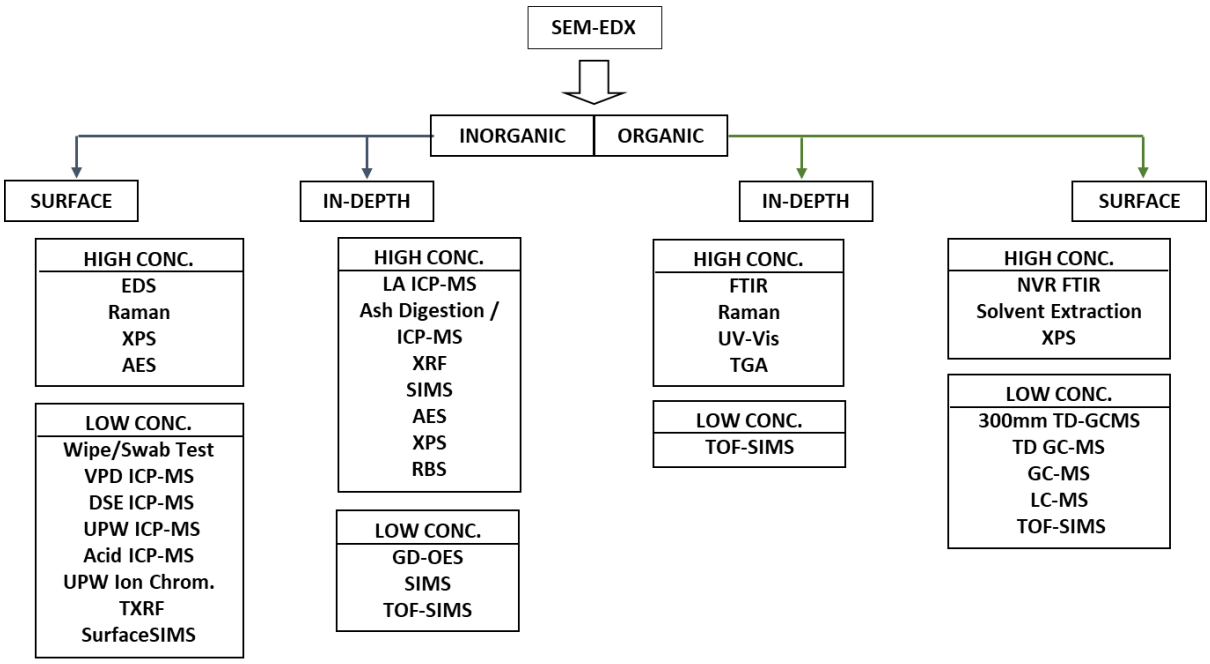

Figure 1: An analytical decision tree for contamination troubleshooting and failure analysis

microanalytical techniques. Each technique has advantages and limitations for a specific application. The specific requirements of individual situations will often dictate the choice of the technique. In general, no single analytical technique can provide the answer to every contamination problem, especially if the goal is to identify and determine a root cause fix of the defect source.

Figure 1 shows an analytical decision tree to guide the selection of an analytical technique. SEM-EDS is often a recommended "first look" technique because it provides elemental information to determine if the defect is inorganic or organic in nature. The decision tree then points the way to the selection of an appropriate inorganic and organic technique for surface and in-depth analyses with a requirement for low or high concentration detection limits.

Notice some techniques in Fig. 1 are represented in two categories. For example, Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS) and X-ray Photoelectron Spectroscopy (XPS) are versatile techniques that can operate in a surface and in-depth analysis mode. In addition, these two techniques can provide elemental and organic information. In contrast, techniques usually provide high or low detection limit unless they have a large dynamic range like SIMS.

III. Chemical Analytical Methods

Analytical techniques in this category are capable of detecting trace metal impurities in liquids (aqueous and non-aqueous) and include Inductively Coupled Plasma Mass Spectrometry (ICP-MS) [5], Inductively Coupled Plasma Atomic Absorption Spectroscopy (ICP-AAS), Graphite Furnace Atomic Absorption Spectroscopy (GFAAS), Ion Chromatography (IC), and Gas Chromatography Mass Spectrometry (GC-MS).

ICP-MS, ICP-OES [6], and GFAAS are in many laboratories together because they complement each other. The principle advantage of GFAAS is its ability to analyze very small volume (µL) of sample easily. Its detection limit for most elements is significantly lower than ICP-OES. GFAAS is often used when very small volumes are available and low detection limits are required for just a few elements. The main advantage of ICP-OES over the GFAAS is its multi-element capabilities and excellent linear dynamic range. In addition, besides the refractory compound-forming elements, elements such as Iodine, Phosphorus, and Sulfur are detected with more sensitivity by ICP-OES.

For applications requiring multi-element analyses of samples in a complicated matrix or if a high sample throughput rate with moderate sensitivity is required, then ICP-OES may be the best choice. ICP-MS has the sensitivity and detection limits typical of GFAAS, combined with the multi-element capability of ICP-OES. This makes ICP-MS ideal for

applications requiring very low detection limits and multi-element analyses. Capabilities exist for 60+ elements via ICP-MS.

Ion Chromatography (IC) [7] detects both cations and anions, and can separate interferences making this technique attractive for analyzing trace level contamination in ultrapure water (UPW), solvents, cleaning solutions, plating baths and other chemicals, as well as for residue analysis collected from UPW extraction. Common UPW extractable anions and cations include F^-, Cl^-, NO_2^-, Br^-, NO_3^-, SO_4^{2-}, PO_4^{3-}, Li^+, Na^+, NH_4^+, K^+, Mg^{2+}, and Ca^{2+}. Ion chromatography can analyze cleanroom air for cations and anions.

The combination of Gas Chromatography (GC) for separation and Mass Spectrometry (MS) for detection and identification into a single GC-MS system makes it a very capable analytical tool [8]. Organics in a cleanroom environment can be problematic as it can change surface hydrophobicity, lower breakdown voltage, form silicon carbide, affect oxide growth and quality, cause unintentional doping, and contribute to haze formation. GC-MS can monitor major sources of organic contamination such as cleanroom construction materials and polymers [9], cleanroom air, and organics in high-purity water [10] and chemicals. A secondary source of organic contamination is from cleanroom accessories such as garments, gloves, wipers, and from wafer and packaging containers.

Witness silicon wafers strategically placed in the packaging facility can determine the effectiveness of chemical filters installed in the air supply system. Measurement of surface organics on test wafers is best performed using a wafer desorption unit and an Automated Thermal Desorption system ATD GC-MS [11, 12]. The wafer desorption unit heats the whole wafer to the required temperature. Volatile organic compounds are desorbed from the wafer surface and are trapped in a thermal desorption tube that is consequently placed in an ATD GC-MS for organic analysis.

An important time to perform organic outgassing tests is when a newly built cleanroom is completed. The cleanroom bay designed to house equipment for processing must be qualified to exhibit acceptable organic compound outgassing to the environment before it enters into service. In essence, this is the ultimate test to verify the choice of components used in the construction of the cleanroom was appropriate. The cleanroom air is sampled using an air sampler fitted with a glass thermal desorption tube, filled with an appropriate adsorbent.

The volatile organic compounds (VOC) trapped by the adsorbent is desorbed with an ATD and analyzed by GC-MS. Quantification is achieved by using the response factor of an external standard, such as n-decane. The method detection limit is about 0.1 µg/g (ppmw).

IV. Microanalytical Techniques

There are many analytical techniques capable of characterizing contaminants in a solid material, such as packaging materials, plastics, silicon wafers, films on the wafer, particles, metal targets, equipment components such as apertures and lenses, and flat panels [13]. Many of these techniques involve a primary beam of electrons, photons, or ions to probe the sample. The primary beam acts as an excitation source and interacts with the material in some way.

Depth profiling is the characterization of a material as a function of depth. Most techniques can vary the depth probed by varying the analytical condition to remove the surface, layer by layer, while collecting data. This sputtering process takes place in a vacuum chamber to ensure the exposed surface remains free of contamination during the analysis period. Analytical techniques that use this principle include Secondary Ion Mass Spectrometry (SIMS), Time-of-Flight SIMS (TOF-SIMS), AES, and XPS. A subtle difference between SIMS and the other techniques is that in SIMS the primary beam and the sputtering beam are the same. In the case of the other techniques, an argon beam removes the material and a primary beam then interacts with the newly exposed surface.

Fourier Transform Infrared (FTIR) spectroscopy and Transmission Electron Microscopy (TEM) monitors the changes induced in the primary beam (energy, intensity, and angular distribution) after the interaction with the sample. In the other techniques, the analysis information comes from electrons, photons, or ions that ejected from the sample during primary beam bombardment. In most cases, several related quantum processes are occurring more or less simultaneously and the analytical technique focuses on one particular aspect.

X-ray Photoelectron Spectroscopy (XPS) analyzes the photoelectrons that emitted from the sample after excitation by x-rays. In Auger Electron Spectroscopy (AES), the incident electron ejects a K-shell electron, for example. In accordance with quantum theory, an electron of higher energy, such as an L-shell electron fills the K-shell vacancy. Energy loss by this L-shell electron during this process results in the ejection of another L-shell electron known as an Auger electron that is ultimately measured.

Detection sensitivities in microanalytical techniques vary

from atom% to ppb concentration levels. Spatial resolution is an important parameter to consider as defect sizes of interest gets smaller. Field Emission-AES can analyze particles as small as 10 nm in diameter [14]. This is a significant improvement from 0.2 µm analysis area with conventional AES operating in an imaging mode. Another example of an improved spatial resolution technique is micro-XPS instruments that can operate with 10 µm lateral resolution compared with 600 µm to 2 mm by conventional XPS instruments. However, if organic or chemical state information is required from a small area, less than 100 nm, the recommended analytical techniques are XPS and TOF-SIMS despite their limitation to sub-micron area.

V. Contamination Troubleshooting Examples

Table I summarizes several contamination failure analyses of microelectronic packaging.

A. Delamination

Pad peeling or interlayer dielectric (ILD) delamination and cracking [15] is a major packaging challenge. It may result from the reduced strength of the ILD films, with respect to their adhesive and cohesive strength, from stresses introduced by packaging materials with significantly different thermal expansion coefficients, or from organics and phthalate plasticizers residue on surfaces. The delamination failure mode is difficult to detect because the separation caused by the delamination can be only 50 microns or less.

Appropriate tests for organic silicone characterization includes solvent extraction and organic characterization using Non-Volatile Residue (NVR) analysis, Fourier-Transform Infrared spectroscopy (FTIR), Nuclear Magnetic Resonance (NMR), Raman spectroscopy, Gas Chromatography (GC), Gas Chromatography-Mass Spectrometry (GC-MS), Thermal Desorption Spectroscopy (TDS), and High-Performance Liquid Chromatography (HPLC).

Organic contribution may be from airborne molecular contamination (AMC) and from surface molecular contamination (SCM) in the cleanroom. AMC in the cleanroom environment is monitored using air impingers and witness wafers. Surface molecular contamination (SCM) may be measured using surface analysis tests like critical wipes for metals and NH_4^+, NVR, and solvent extraction. [16]. SMC is important to measure on a regular basis because the results will illuminate potential cross-contamination pathways during the packaging process and help to determine the frequency and locations of wipe down.

B. Corrosion

Materials, design, packaging type, and the environment can influence corrosion in microelectronics. Metal exposed to moisture is highly sensitive to corrosion due to the chemical interaction of aluminum with water. Surface corrosion and oxidation may result from ionic Chloride, Fluoride, and brominated fire retardants such as polybrominated biphenyls (PBB) and polybrominated diphenyl ethers (PBDE).

Common sources and mechanisms of corrosion in microelectronics includes anodic, cathodic, and electrolytic reactions resulting in uniform corrosion, galvanic corrosion, pitting corrosion, creep corrosion, dendrite growth, stress-corrosion cracking, and whisker growth. Troubleshooting tests of surfaces may include inorganic characterization with ultrapure water extraction, IC, SEM-EDS, X-ray Fluorescence (XRF), and chemical state analysis and inorganic testing using XPS and FTIR.

C. *Solder impurities*

Bonding of the wire to the surface of a semiconductor chip is a difficult task. The wire must make a good electrical connection that will not degrade with time. The process generally involves tiny balls of solder that in essence solder the wire to the chip. The control of bond parameters such as bond pad thickness and cleanliness, wire softness, age, purity, forming gas flow, spark control and all bonding parameters are required in high volume production.

Unfortunately, there are many potential wear-out mechanisms such as brittle fracture or galvanic corrosion. Solder impurities such as metallic and non-metallic impurities can effect wetting properties of the solder and the amount of reworking of defective joints.

Troubleshooting techniques include bulk analyses such as Laser Ablation (LA) ICP-MS and acid digestion ICP-MS, to detect impurities introduced into the solder from soldered parts, from holding fixtures, and from the solder pot itself. Table II shows comparable results obtained by LA ICP-MS and two other independent methods. The wet chemistry method was acid digestion followed by ICP-OES analysis.

Table II: Microanalytical analysis of solder bumps

Analysis Method	Tin (Sn)	Lead (Pb)
LA ICP-MS	5.78%	94.20%
Wet Chemistry	5.45%	94.50%
X-Ray Fluorescence	3.48%	96.50%

Table I: Summary of contamination failure analysis of microelectronic packaging

Failure	Causes	Analytical Testing
Pad peeling or ILD delamination	Organics and phthalate plasticizers residue on surfaces	Surface leach and extractable, environmental AMC monitoring, organic silicone characterization - **SEM-EDS, Solvent Extraction, NVR, FTIR, Raman, NMR, GC, GC-MS, HPLC, TDS**
Corrosion and oxidation	Ionic Cl, F and brominated fire retardants such as polybrominated biphenyls (PBB) and polybrominated diphenyl ethers (PBDE) on surfaces	Surface leach and extractable tests, enviromental AMC monitoring, and inorganic characterization - **Ultrapure water extraction IC, SEM- EDS, XRF** - **Chemical state analysis and inorganic testing such as XPS and FTIR**
Solder impurity	Metallic and non-metallic impurities can effect wetting properties of the solder and the amount of reworking of defective joints	Bulk analyses to determine impurities introduced into the solder from parts being soldered, from holding fixtures and from the solder pot Itself - **LA ICP-MS and acid digestion ICP-MS**
Polymer dielectric breakdown	Inner layer dielectric (ILD) and underfill in the chips are fragile and prone to cracking, necessitating careful management of stresses during assembly	Thermal analysis - **TGA** for polymer degradation, thermal stability as a function of time and inorganic fill materials - **DSC** for polymer curing temperature and glass transition Trace element analysis - **ICP-MS, GF-AAS, ICP-OES**

D. Polymer dielectric breakdown

Polymer materials such as low-κ back-end of line (BEOL) dielectrics are common in IC fabrication because polymer films show high breakdown strength. Polymer dielectric breakdown can result from the fragile inner layer dielectric (ILD) and underfill in the chips that are prone to cracking [17]. In addition, polymers are highly susceptible to slow chemical degradation due to environmental moisture ingress from hydrolytic deterioration, which degrades changes in their electrical and mechanical properties of the material [18].

Such issues necessitate careful management of stresses during assembly and thermal analysis to characterize the materials used. For example, Thermo Gravimetric Analysis (TGA) for accessing polymer degradation and thermal stability as a function of time and Differential Scanning Calorimetry (DSC) for determining polymer curing temperature and glass transition are valuable techniques

VI. Conclusion

Advanced packaging processes [19] involve the use of front-end-of-line (FEOL) technologies in back-end applications. There are a broad range of materials for advanced packaging applications, wafer-level packaging, and 3D integration to enable miniaturization. Materials used include lead-free solders, die attach, flip chip underfills, encapsulant, epoxy molding compounds, metals, organic polymers, and conductive adhesives. Material selection with low impurities is important to achieve reliable wafer bonding for wafer-to-wafer or chip-to-wafer.

Contamination introduced during the manufacturing process has serious repercussion because an impurity in one chip, or in the materials used to bond those chips, can influence the multi-chip package, resulting in reduced yield and higher costs. Impurities can be introduced from anywhere in the materials supply chain and can for example be nanoparticles in ultrapure water and chemicals, or AMC and SMC from the cleanroom environment. Cleanroom cleanliness monitoring and Clean Manufacturing protocols should be practiced to control AMC and SMC in the manufacturing environment and on critical surfaces.

Contamination control, vigilant inspection, and metrology capability at critical steps are required to ensure proficient system level integration, board level performance, increased reliability, and high yield manufacturing at a low cost. Conventional optical inspection techniques may find it challenging to characterize organic residues left after etching or to investigate metal grain features, for example. Fortunately, there are sophisticated analytical techniques to analyze the effects of chemical contamination and environment exposure to advanced microelectronic packaging.

An Analytical Decision Tree can guide the selection of an appropriate analytical technique to characterize defects, analyze contamination with a high degree of specificity and detection sensitivity, and to troubleshoot their root cause and

source. Chemical and microanalytical techniques complement each other. It is common for more than one technique to be required along with the expert data interpretation to complete a contamination investigation.

Acknowledgment

The author wishes to thank the Balazs Fremont laboratory scientists for their contribution to this paper.

References

[1] J. N. Sweet, D. W. Peterson, A. H. Hsia, M. Tuck, High Reliability Plastic Packaging for Microelectronics, SAND97-1721, Printed July 1997
[2] E. Sperling, What Goes Wrong In Advanced Packages, S*emiconductor Engineering*, March 24, 2021
[3] P. R. Gangidi, *Application of Six Sigma in Semiconductor Manufacturing: A Case Study in Yield Improvement*, *Applications of Design for Manufacturing and Assembly*, Ancuţa Păcurar, IntechOpen, DOI: 10.5772/intechopen.81058. Available: https://www.intechopen.com/books/applications-of-design-for-manufacturing-and-assembly/application-of-six-sigma-in-semiconductor-manufacturing-a-case-study-in-yield-improvement
[4] R. P. Donovan, *Contamination-Free Manufacturing for Semiconductors and Other Precision Products*, 1st Edition, CRC Press, March 14, 2001
[5] J.W. Robinson, *Atomic Spectroscopy*, Monticcello, NY, Marcel Dekker, 1996
[6] P.W.J.M. Boumans, Editor, *Inductively Coupled Plasma Emission Spectroscopy, Part 2: Applications and Fundamentals*, ISBN: 978-0-471-85378-7, June 1987, John Wiley & Sons
[7] J. Weiss, *Ion Chromatography*, 2nd Edition ISBN: 978-3-527-61562-9, July 2008
[8] M.C. McMaster and C. McMaster, *GC-MS: A Practical User's Guide*, New York, NY, Wiley-VCH Publishers, 1998
[9] S. Lin and S. Graves, *Comparing the Molecular Contamination Contribution of Clean Packaging Films*, Micro, October, 1998
[10] A. Kumar, M.J. Camenzind, and C.J. Chargin Jr., *Identifying Organic Contaminants in Ultrapure Water at Sub-Parts-per-Billion Levels*, Micro, January, pp. 51-59, 2000
[11] P. Sun, M. Adam, and T. Bridges, *Monitoring Organics on Wafer Surfaces Using Thermal Desorption GC-MS*, Micro, March, pp. 59-71, 2000
[12] M. Saito, *Evaluation and Control Method of Organic Contamination on Wafer Surfaces*, International Symposium on Semiconductor Manufacturing, pp. 171-174, 1995
[13] C. Evans, R. Brundle, and W. Wilson, *Encyclopedia of Materials Characterization: Surfaces, Interfaces, Thin Films (Materials Characterization Series)*, Butterworth-Heinemann, September 1, 1992
[14] K.D. Childs, D.G. Watson, D.F. Paul, and S.P. Clough, *Analysis of Submicron Defects Using an SEM-Auger Defect Review Tool*, Characterization and Metrology for ULSI Technology, International Conference, AIP Conference Proceedings 449, pp. 810-814, 1998
[15] L. Wei-Sheng Lei and A. Kumar, *Delamination and Reliability Issues in Packaged Devices*, in book: Adhesion in Microelectronics (pp.267-305), Edition: 1st, Chapter: 7, Publisher: Wiley, Editors: K. L. Mittal, Tanweer Ahsan
[16] M.J. Camenzind, H. Kiang, J. Fusko, and M.K. Balazs, "*How Clean is you Cleanroom Air*, MICRO, October, 1995
[17] S. Lee, L. Yang, W. Liu, and W. Wang, *Dielectric Breakdown of Polymer Nanocomposites: From Fundamentals to Devices and Applications*, In book: Chapter 9, Tailoring of Nanocomposite Dielectrics (pp.243-280), October 2016
[18] Y. Li, M. Cordovez, and V. M. Karbhari, *Dielectric and mechanical characterization of processing and moisture uptake effects in Eglass/epoxy composites*, Compos. Part B Eng., vol. 34, no. 4, pp. 383-390, 2003
[19] D. Lu, Daniel and C.P. Wong (Eds.), *Materials for Advanced Packaging*, Springer US publisher, 2009

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