Cleaning Organic Residue in High-Pb Flip Chip Ball Grid Array Interconnection

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Abstract: This paper discusses the formation of organic residues on high lead (Pb) Flip Chip Ball Grid Array (FCBGA) package. Flux used for Control Collapse Chip Connection (C4) die attachment during assemblies could remain on the die surface as organic residues, thus affecting the Integrated Circuit (IC) performance. Therefore, the effect of implementation flux-cleaning process on the die cleanliness was evaluated in this study. Design of Experiments (DOE) for cleaning chemical parameters using water-based solvents was carried out to investigate the flux-cleaning efficiency. Referring to the IPC-TM-650 test manual of contaminants extraction approach, the test packages were then evaluated in-situ via Fourier Transform Infrared Spectroscopy (FTIR). FTIR results show that there is organic flux residue detected prior the cleaning process. Notably in contrast, there is no residue detected after the cleaning process. This clearly indicates a positive correlation of the cleaning process to the DOE implemented.

Key words: High Pb-flip chip-organic residue-cleaning process-chemical concentration.

INTRODUCTION

Semiconductor IC designed for vigorous industries such as military, avionic, servers as well as automotive are required to have high reliability and excellent long-term performance. High reliability and long lifetime would result in better product performance and low probability of field failures. When UK Restriction of Hazardous Substance (RoHS) Regulation came into effect in 2006, it was mandatory that all the electronic manufacturers make a transition from leaded product into lead-free products. However, some products are exempted from this regulation due to the requirement of high lead solder to form good electrical connections (P. Goodman, 2004). High reliability products that involve flip chip packages also fall into this exemption group. In flip chip packages, high lead solder ball is used with rosin-based flux. Cross-linking reactions would occur, and produce organic carbonaceous by-product (A. Baated, 2010; C. Beddingfield. 1997; D.F. Bernier, 1999; K.S. Hansen, 2009). This organic by-product could be detrimental to the product performance. Thus, post-reflow cleaning is required. The water-based MPC® cleaning technology that is environmental friendly is applied. This paper presents results on flux residue formation due to different flux compositions and the efficiency of non-surfactant cleaning technology in removing the existing contamination. FTIR spectrums that indicate the presence of organic contaminant before and after the cleaning process are also presented.

2. Background:

1. Flip Chip Package:

A well-known advantage of solder bumps in the flip chip technology is the optimal method to increase the packaging density, which simultaneously increase the input/output of the device. A typical FCBGA is shown in Fig. 1. The package consists of active silicon die with an array of solder bumps, which is then flipped onto the substrates to form interconnections. Underfill material is used in between the interconnections to prevent the moisture from attacking the solder bumps interconnections, to strengthen the solder joints as well as to prevent voids near the solder bumps and to protect the bumps from early fatigue (A.F.J. Baggerman, 1996).

The flip chip package is widely used in high performance applications such as servers, high-speed memory units, air system controllers, military systems as well as automotive controllers. Thus, high melting temperature solders are required to ensure reliable joints during C4 attachment, wire bonding to carriers, and discrete components to modules. The RoHS Regulations banned Pb containing solders from the electronics industry. However, there is an exemption for high melting temperature, lead solder types (i.e lead-based alloy containing at least 85% by weight). The high lead solder bumps could endure a high number of thermal fatigue cycles and
could resist electromigration failure (N.C. Lee, 2005). This makes high-Pb solder bumps suitable for good electrical connections.

![Fig. 1: Schematic cross-section of typical FCBGA package.](image)

Traditional eutectic Tin/Lead (Sn/Pb) solder melts at 183 °C. The alternative Pb-free solder bumps melt at temperature 30-40 °C higher than the eutectic solder. The melting temperature for Tin/Copper (Sn/Cu) is at 227 °C, Tin/Silver (Sn/Ag) at 221 °C, and Tin-Silver-Copper (Sn/Ag/Cu) at 217 °C (N.C. Lee. 2005; L.J. Turbini, 2006). High lead solder bumps such as 97Pb/3Sn have even higher melting temperature at 300 °C to 313 °C, 95Pb/5Sn at 308 °C to 312 °C and 90Pb/10Sn melting temperature is 275 °C to 302 °C (N.C. Lee. 2005). Thus, high-Pb solders are still favorable for some electronics applications.

II. Solder Flux:

Solder interconnection is invented to perform three major functions: electrical, mechanical and thermal (D. Shangguan, 2005). In flip chip assemblies, solder bumps are used as the interconnection between the silicon die and the substrate. In order to have an excellent joining process, the metal pad surfaces must be clean from contamination and oxidation. This chore is performed by the solder flux (A.F.J. Baggerman, 1996). In addition to cleaning metal pad surfaces, flux also provides a good metallurgical wetting to the solder. When solder melts and interacts with the copper metal during the reflow process, Sn forms an intermetallic compound with Cu (L.J. Matienzo, 1991). If the metal oxide remains, the solder would not be able to properly interact with the base metal and a good solder joints would not be produced. Solder flux is also helping to improve the thermal function during the soldering process to ensure a complete wetting. Flux reduces the interfacial surface tension between the metal pad and the solder. This allows the molten solder to smoothly flow over and properly wet the soldered surfaces (N.C. Lee, 2005).

High-Pb solder interconnection requires high melting point and for that, rosin based flux is usually used. Rosin-based flux is a type of soldering flux that is composed of an organic compound namely colophony (a pine trees rosin). Colophony basically consists of a mixture of resin acids, which are predominantly carboxylic acids such as abietic acid and neoabietic acid, dehydroabietic acid and also di-hydroabietic acid (N.C. Lee, 2002). Rosin flux also consists of 90% resin acids and the other 10% is non-acidic materials, including resin acid esters, fatty acid esters, di-terpenealdehydes, alcohol and etc. (D.F. Bernier, 1999). This 10% of ingredients add to the complexity of the flux chemical.

III. Flux Residue:

The flux is activated when heated. After chip alignment, the assemblies are subjected to a reflow process in furnace with nitrogen-forming gas (5% H₂ + 95% N₂) and oxygen-free ambient. During this time, fluxing reactions such as acid-base reactions and oxidation-reduction reactions would occur. Even though rosin is thermally stable, it would undergo isomeric transformation (N.C. Lee, 2002).

At higher temperature, the abietic acid would undergo simultaneous oxidation and reduction resulting in a mixture of de-hydroabietic acid, di- and tetra-hydroabietic acid (D.F. Bernier, 1999). The oxidized rosin is less soluble in solvent and may irregularly distribute on the surfaces as a layer of white film (K.G. Sachdev, 1999). This organic acid would also react with solders (Pb/Sn) to produce unwanted lead and/or tin abietates (A. Baated, 2010). This unwanted metal is bound to the organic acid and redeposit on the device surface, forming white residue.

Other than organic acid, flux activator also has the potential to be a catalyst for electromigration and dendrite. Flux activator is hygroscopic and acidic in nature. In the presence of conductive electrolysis and electrical field, it would lead to electrochemical migration as well as dendrite growth (L.J. Turbini, 2000). In addition, flux residue could induce creeping current and result in electrical open circuit and/or interrupt RF signal integrity (K.S. Hansen, 2009). The hygroscopic characteristic of flux could also lead to corrosion in the solder. Further problem that could be present is tin whiskers. It is suspected that the source of whiskering is related to the corrosion of solders (K.G. Sachdev, 1999). The following are the few potential surfaces that could be affected by the flux residues during C4 attachment (K.G. Sachdev, 1999):
1- Lead-tin solder interconnection,  
2- Bonding metallurgy on the substrate,  
3- Ceramic substrate, polyimide passivation layer on device chip.  

IV. Flux Residue Cleaning:  
Before halogenated solvent was recognized to cause several environmental and disposal issues, chlorinated solvent such as tetrachloroethylene and aromatic hydrocarbons were used for flux cleaning (K.G. Sachdev, 2004). However, xylene, such as cleaner requires lots of considerations including the regulatory compliance for air emissions in the case of Volatile Organic Compound (VOC) and Hazardous Air Pollutants (HAPS) (W.D. Spall, 1994). Therefore, the Micro Phase Cleaning (MPC) technology, which is water-based, is proposed as an alternative to the organic solvent cleaning method.  
MPC has the advantages of simultaneous aqueous and solvent-based cleaning, with water mixed with highly active cleaning agents. This technology works by removing the contaminants from the substrate and transferring to the surrounding aqueous medium. Subsequently, the contaminations filtration system in the cleaning machine filters off the released contaminants (IPC., 2004).  
DI water is known to have high surface tension over 70 dynes/cm. This characteristic poses a limitation to the DI water to effectively penetrate underneath the low standoff component such as flip chip. Surface treatment could affect the wetting and surface tension properties by reducing the liquid drop size, thus, enabling the cleaning fluid to move easily under the low gap spaces (S. Stach, 2006). The surface tension could be reduced to 30 dynes/cm and below when chemical is added to the cleaning fluids, such as DI water (H. Wack, 2011). Thus, in the experiment, MPC solvent will be used as an added chemical to pure DI water.

3. Experimental Procedure:  
I. Test Vehicle:  
Test vehicle is built from a FCCBGA package with a 12 x 15 mm² active silicon die, which is considered a large die in size. The product is manufactured for high speed and high power microprocessor devices in automotive applications. The die was developed with CMOS-90 nm technology, internally engineered low-k dielectric and polyimide (PI) passivation. The bump is made of high-Pb (Pb90/Sn10) with 65 μm maximum height and a pitch size of 150 μm. The die was later placed onto the glass slide to replace the real ceramic substrate for the purpose of visual inspection. Washable flux was used for die attachment before the solder reflow process. The package was then put in the reflow oven and preceded with the cleaning process.  
II. Cleaning:  
For the chemical parameters DOE study, the full factorial analysis evaluated the variables of wash temperature, cleaning agent technology and cleaning agent concentration during the cleaning process. The conveyor belt speed was set at 0.3 m/min. Three sets of MPC solvent concentration that are 3%, 5% and 7% were used to observe the cleaning efficiency at different concentration while the temperature was varied at 65 °C, 75 °C and 85 °C. The variation of temperature is expected to influence the characteristic of MPC solvents throughout the cleaning process. State-of-the-art inline equipment was used to perform all the experiments. Visual inspection was performed. The results are categorized according to the cleanliness levels.  
III. FTIR Sample Preparation:  
Sample preparation for this method is based on IPC-TM-650 Test Method Manual 2.3.28A (N.C. Lee, 2002). Using this approach, contaminations on the samples were extracted by dissolving the die/samples into the KAPAK™ polyester extraction bag, which was filled with an extraction solution, consisting of 75/25 v/v IPA/H₂O. The use of IPA helps to wet the board and dissolve the contaminations from the flux residue. The solution filled bags was then heated in a water bath at 80°C for one-hour of extraction. The solution was then tested with the FTIR equipment.  

RESULTS AND DISCUSSION  
I. Visual Cleanliness:  
The experiments were first conducted using DI Water as a benchmark. Fig. 2 shows the visual cleanliness results using DI water.
The visual cleanliness result in Fig. 2 shows that there were large amounts of flux residues left after the cleaning process. It proves that DI water alone is unable to remove the flux residues that exist in between the low gaps of the samples. In comparison to the organic solvent, pure water is a good solvent for cleaning certain materials such as polar and ionizable materials. However, pure water is a poor solvent for non-polar materials such as resin and rosin (S. Stach, 2004). The visual cleanliness results via MPC solvents at concentrations of 3%, 5% and 7% are shown in Fig. 3, Fig. 4 and Figure 5 respectively.

Fig. 3 shows that 3% MPC solvent has been added to the DI water, almost all of the entrapped flux residues were removed from the test die at 75 °C. However, as the washing temperature increased to 85 °C, even more flux residues remain underneath the samples. This results shows that high temperature is not suitable for this cleaning product. At high temperature, the cleaning solution could undergo “surfactant separation” segregate, where individual raw materials can precipitate and hence affect the cleaning consistency (S. Stach, 2008). At 85 °C, it become soluble and loses its cleaning properties. There are also possibilities that at high temperature, the fluid loses its viscosity and the cleaning efficiency reduced.

When 5% and 7% of MPC solvent was added to the DI water, the cleanliness levels were degraded as shown in Fig. 4 and Fig. 5. There were flux residues left at the sample edge after the cleaning process for 65 °C, 75 °C and 85 °C. Researchers from Zestron (L. Azar, 2009) recognized that solution density and viscosity would also affect the end results in addition to surface tension. The concentration of solution correlates with the solution density. By adding more solute to a solution, it increases the density of the solution. By increasing the
concentration, the viscosity increases. However when increased to a certain high concentration, the solute (MPC) might exceed its solubility and begin to precipitate, reducing the cleaning efficiency. Therefore, an optimal concentration of cleaning chemistry that can provide optimum-cleaning results is to be determined. In this work, 3% concentration is found to yield the highest cleaning efficiency.

As a conclusion to this study, the flux cleaning process could be optimized by using 3% concentration of MPC® cleaning at 75 °C wash temperature. These parameters combination could provide excellent cleaning results (almost 100% full cleanliness) under the flip chip, in comparison to the pure DI-water inline cleaning process. The FTIR spectrum of the test samples with 3% MPC and subjected to 75 °C wash temperature were subsequently obtained to investigate the presence of the flux residue before and after the cleaning process.

II. FTIR Analysis:

Fig. 6 shows the comparison between the FTIR reference spectrum for the flux used (red line) and non-cleaned sample (black line). Non-cleaned sample is the sample taken prior the cleaning process. The spectrums show that the peaks for both samples are almost the same. This indicates the presence of flux residue in non-cleaned sample. The main ingredients for flux are the rosin/resin component (50-60% by weight) and also benzyl alcohol (30-40% by weight).

![Fig. 6: The comparison of the FTIR spectrum of flux used with non-cleaned sample spectrum.](image)

The spectrum in Fig. 6 shows that there are strong absorptions detected due to the rosin composition molecules together with the benzyl alcohol molecules. The band at 1750 cm⁻¹ is due to the carbonyl C=O double bond stretch from the abietic acids in the rosin. The broad band in the range of 2700-3700 cm⁻¹ is due to the presence of OH band, overlapping with CH stretch, which falls at 2830 cm⁻¹. The CO single bond from the acid structure is reflected in the spectrum range of 1100- 1300 cm⁻¹.

Benzyl alcohol molecules vibrations were also reflected in the spectrum. Benzyl alcohol is an organic compound with the formula C₆H₅CH₂OH. Alcohol gives a strong and broad IR absorption beyond 3000 cm⁻¹ as shown by the OH stretch in the Fig.6. Other than the C-H stretch at about 2850 cm⁻¹ and C=C at 1460 cm⁻¹, there are two important regions of aromatics spectra, which indicate the presence of benzyl alcohol in the compositions:

1- 2000 - 1665 cm⁻¹ (overtone).
2- 900 - 675 cm⁻¹ (out of plane bands).
Fig. 7: FTIR spectrum for cleaned sample and extractor solution.

Fig. 7 shows spectrum for cleaned sample. When the cleaned sample spectrum is compared to the extraction solution spectrum, the peaks for both spectrums are very similar. This point out that there is no flux residue extracted from the sample that undergoes the cleaning process. The same results are obtained for all cleaned samples. The extractor solution consists of IPA and DI water. It is shown that the composition for this spectrum is Isopropanol Alcohol (IPA). The presence of IPA and DI water functional groups is shown by a strong and broad OH stretch at about 3000-3625 cm⁻¹. There are also CO peaks at 1300 and 1130 cm⁻¹ that show the presence of CO single bond in the alcohol composition.

Conclusion:
The results obtained from this study have proven that the chemistry-assisted cleaning method outperformed the pristine DI-water cleaning. The optimum result obtained with 3% MPC concentration at 75 °C wash temperature. As proven by the FTIR results, the water-based cleaner is able to completely wash off the flux residues under the low pitch flip chip test die.

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