Development of a Process for the Production of Fluxless Solder Spheres for BGA Electronics Packaging

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Submitted to the Department of Mechanical Engineering  
in Partial Fulfillment of the Requirements for the Degree of  
Mechanical Engineer's Degree

at the

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June 1999

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ABSTRACT

The work presented here focuses on the use of a new process for the production of
fluxless solder spheres for BGA (Ball Grid Array) microelectronics packaging
technology. The Uniform Droplet Spray (UDS) process produces solder spheres by
controlling the breakup of a continuous laminar jet into uniform droplets, which are then
rapidly solidified in a liquid bath or an inert gas. This work discusses the production of
highly size-accurate solder micro spheres (75-1000 μm in diameter) by the UDS process.
To achieve the sphere size distribution requirements an on-line droplet size control
system was developed. Droplet size control is accomplished by performing a real-time
measurement of the droplet size and compensating for the difference between actual and
target sizes by adjusting the break up frequency. The control system is effective in
controlling sphere size, enabling the UDS process to accurately determine and control
solder sphere size within ± 2.5% of target size.

Current BGA assembly processes require that the oxide films, typically up to 100
Å thick, on the surface of both the solder sphere and the substrate, be removed, or
otherwise chemically altered, to permit melting, wetting, and spreading of the solder and
good bonding characteristics. The common technique for cleaning the surfaces while
soldering is to use a liquid solder flux. However, the flux residues have to be cleaned
using chlorofluorocarbon (CFC) solvents. Because of environmental and worker-safety
concerns, CFC will be eliminated in the next few years. Thus the need to develop a
process for producing fluxless solder spheres for BGAs is a critical issue. This work also
presents the coupling of a plasma-assisted treatment and the UDS process for the
production of fluxless solder spheres by transforming the chemical composition of the
spheres' surface. The solder spheres are treated in fluorine-containing plasma to convert
the metallic surfaces into oxy-fluorine layers. These layers protect the spheres from
further oxidation and act as flux during the reflow process. Experimental evaluation of
the solderability performance of the fluxless solder spheres indicates that the plasma-
assisted oxide conversion process is effective in replacing flux, enhancing operator
safety, reducing process time and production cost, and minimizing contamination of the
work environment.

Thesis Supervisor:
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ACKNOWLEDGEMENTS

I would like to thank all who made possible the completion of this work. My most sincere gratitude to:

First and foremost to Grecia, who has always encouraged me to pursue my dreams with unconditional love and support through all the years. And for being there when I most needed it. My parents for all their caring and support, whose values had leading me all my life. Prof. Jung-Hoon Chun, who gave me the opportunity of completing this work, and whose guidance and support were invaluable, and are greatly appreciated. The members of the DBM group, whom I like to consider my friends. Jeanie Cherng whose dedication and motivation were contagious. Dr. Sukyoung Chey whose thoughtful advice was always welcome and to the point. Dr. Ho-Young Kim whose knowledge and sharpness were remarkable. Jiun-Yu Lay who was always ready to lend me a hand and an ear. And last but not least to Wayne Hsiao who made me think there was something else besides work at MIT and whom I would have liked to get to know better. Dr. Nannaji Saka, for his valuable input at the early stages of this project. All of them were the best part of being at MIT thorough out these years.

To you all,

My most sincere gratitude
DEVELOPMENT OF A PROCESS FOR THE PRODUCTION OF FLUXLESS
SOLDER SPHERES FOR BGA ELECTRONICS PACKAGING

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CHAPTER 1 INTRODUCTION

1.1 Introduction

The development of new technologies in the microelectronics industry focuses on smaller, more reliable and cost-efficient electronic components. Ball Grid Array (BGA) packaging is a chip interconnection technology that delivers all those attributes. BGA technology efficiently reduces the size of components while maintaining or improving their performance, by using solder spheres as connection elements between the chip and the circuit module. The demand of BGA has grown exponentially since its introduction to the market in the early 1990s. However, solder sphere production processes have lagged in terms of efficiency and production rate.

The work presented here focuses on the use of a new process technology for the production of solder spheres for BGA microelectronics packaging technology. The Uniform Droplet Spray (UDS) process produces solder spheres by controlling the breakup of a continuous laminar jet into uniform droplets, which are then rapidly solidified in a liquid bath or an inert gas. Although the spheres have a narrow size distribution (± 9% from mean size), still greater size accuracy is required (± 3% from mean size) for industrial deployment. This work discusses the production of highly size-accurate solder micro spheres (75-1000 µm in diameter) by the UDS process. To achieve the sphere size distribution requirements, an on-line droplet size control system was developed. Droplet size control is accomplished by performing a real-time measurement of the droplet size and compensating for the difference between actual and target sizes by adjusting the break up frequency. The control system is effective in controlling sphere size, enabling the UDS process to accurately determine and control solder sphere size within ± 2.5% of a target size.

Current BGA assembly processes require that the oxide films, typically up to 100 Å thick, on the surface of both the solder sphere and the substrate be removed, or otherwise chemically altered, to permit melting, wetting and spreading of the solder, and surface bonding. A liquid solder flux is most commonly used for cleaning the surface oxides. However, the flux residues themselves have to be cleaned using chlorofluorocarbon (CFC) solvents. Because of environmental and worker-safety concerns, CFC will be eliminated in the next few years. Thus the need to develop a process for producing fluxless solder spheres for BGAs is a critical industrial issue.

This work presents a treatment to modify the growth of the surfaces of freshly formed solder spheres produced by the UDS process. The solder spheres are treated in fluorine-containing plasma to convert the oxidized surfaces into oxy-fluorine layers. These layers protect the solder spheres from surface oxidation and act as flux during the reflow process. The plasma treatment can be applied either in a batch process or continuously as the spheres are generated. The treated solder spheres can be stored for long periods of time in air. Because soldering flux is not required, soldered joints and the adjoining areas do not need to be cleaned with organic solvents. This enhances operator
safety, reduces process time and production costs, and minimizes contamination of the work environment.

1.2 Ball Grid Array (BGA) Chip Interconnection Technology

Current interconnection technologies in the microelectronics industry are concerned with providing faster, smaller, and denser (having a greater number of connections per unit area) electronic components. Packaging technology plays an important role in the performance of an electronic system. The on-chip delay in IC (integrated circuits) semiconductor devices has been reduced rapidly in the past few years. However, the signal delay in packaged ICs has not been reduced as much as that of the on-chip ICs [1]. Thus, packages had become a significant performance bottleneck in an electronic system. For this reason chip packaging technology development focuses on size reduction, higher I/O counts, greater density, lower cost, and higher performance.

1.2.1 Chip Interconnection (Packaging) Technologies

Several technologies are used for electronics packaging. The most common are quad flat pack (QFP) and pin grid array (PGA). Figure 1.1 a) shows the schematics of a QFP with "gull-wing" leads. QFPs are characterized by the use of peripherally-attached legs delivering a high surface mount compatibility. When the lead spacing of a QFP is equal to or less than 0.65 mm, it is commonly referred to as a fine pitch surface mount technology (SMT) component. The lead frame material of a QFP is typically copper and the molding material is plastic. The most commonly used QFPs are the 28x28 mm body size with 160, 208, or 256 leads. This low cost package family is usually used for ASIC (application specific IC) applications.

Figure 1.1 b) shows a schematic of a PGA. Ceramic PGAs were introduced in the 1960s to expand spatially-dense flip chip devices. PGAs have had a dominant role in high density packaging for many years. They are commonly used for ASCIs and microprocessors in high pin-count, high power, and high performance electronic systems. PGA technology uses metal pins as the connection element between the chip and the card. PGAs provide an excellent electrical performance (controlled impedance), great interconnection flexibility, and I/O pins spread over the area of substrate, making many I/Os possible.

Both, QFP and PGA packaging technologies have drawbacks. QFPs are space-inefficient due to their peripheral interconnect scheme. PGAs have four main drawbacks. They are costly to produce, using copper pins. Their mounting through-holes on the PCB is not SMT compatible. They lack room for pin pitch size reduction (2.54 mm). They are a process intensive technology (pin handling, bending and insertion).
Figure 1.1  (a) Quad Flat Pack and (b) Pin Grid Array configurations.

BGA is an area array interconnection technology that uses solder spheres as the connection element. There are three types of BGAs packages, each identified by the substrate material used. These types are ceramic (CBGA), which delivers excellent thermal and electric characteristics at a relatively high cost; plastic (PBGA), which has moderate thermal and electric properties at low cost; and tape (TBGA), which has excellent thermal and electric properties at an intermediate cost. Figure 1.2 shows a ceramic BGA package. BGA technology provides higher throughput and yields than QFP components. The defect manufacturing level for BGA is approximately six times lower than that of QFP. BGA technology also produces smaller and denser packages. BGAs have reduced the size of electronics components by more than five times in the last few years. However, size reduction is not the only advantage of using BGA packaging. BGA has proven to be more cost effective and reliable. BGA packages are being considered for high I/O count semiconductor devices and are becoming more popular as technology development advances, improving performance and lowering costs. The market for BGAs is expected to grow rapidly over the next few years, with BGA becoming the dominant packaging technology [2].

Figure 1.2  Ceramic BGA package.
1.2.2 BGA In Comparison to Other Packaging Technologies

BGA is superior to other interconnection technologies in the areas of package pins count, package area, package performance, and package manufacturing yield. BGAs are area array packages in which solder spheres are spaced over the bottom surface of the carrier. For example, a 32x32 mm package can have 184 I/Os using QFP, 300 I/Os using PGA, and up to 600 I/Os using BGA. Figure 1.3 shows a schematic representation of the effect on chip interconnection technology on the package outline area [3]. The performance for the BGAs is better because the inductance for a given package is smaller due to the short runs between chip and card. Reflections and noise level can also be reduced by matching the solder sphere size with the output impedance and by dissipating more heat. BGA assembly is very forgiving due to the self-alignment property of the molten solder during reflow. Thus BGA is the packaging technology with the highest manufacturing yield.

![Figure 1.3](image)

**Figure 1.3** Effect of chip interconnection technology on the package area.

1.3 Role of Solder Spheres in BGA

The performance of a BGA package depends on many parameters and variables. Solder sphere size distribution directly affects the assembly reliability of the package and, indirectly, the yield. Sphere uniformity ensures good coplanarity of the package. Coplanarity for a specific sphere in the array is defined by the distance from the top of that sphere to the horizontal stable sitting-plane of the package. Sphere coplanarity directly determines the characteristics of the formation of the final solder joints. Good solder joints reduce the probability of bridges or opens in the connections. Most current BGA applications show a coplanarity requirement of 6 mils [4] as a standard
specification. A maximum sphere size variation of ±3% from the mean sphere diameter is more than adequate for BGA applications. Consequently, one of the objectives of the current research is to make the UDS process produce solder spheres with a size variation smaller than ± 3% of the mean sphere size while placing the mean sphere size on the target size.

1.4 Role of Flux in BGA Assembly Process

Implementation of BGA technology into the manufacturing environment offers a degree of flexibility that no other high-density interconnection technology can deliver. There are two main assembly technologies currently used for BGAs [1,5]. These are two fundamentally different methods that accommodate many in-place processes and board finishes. For the first of these processes, eutectic solder paste is applied to the metallized pads using printing or dispensing techniques. High melting point solder spheres are then placed into the applied paste by means of a fixture drilled with holes in a pattern matching the desired I/O array. The assembly is reflowed in a furnace at a profile that melts only the eutectic solder paste. Flux-based solder paste (rosin or water-soluble) is used in this process, and notably, the assembly must be cleaned after reflow by CFC solvents or deionized water. Providing a fluxless solder sphere would therefore allow the elimination flux-based solder paste, the cleaning steps of the process and the use of highly dangerous solvents.

The generation of "solder balls" is a common problem when solder paste is reflowed. These solder balls can create shorts and damage adjacent components, or they can introduce voids to the solder spheres. For these reasons, a second BGA assembly process was developed. This process completely eliminates the use of solder paste. Instead, flux is applied either to the metallic pads on the package or the solder spheres. Flux application is a time consuming process. Current methods include: dispensing with a programmable system; transferring using pins in an array matching that of the BGA; dipping the spheres or package; and spraying or brushing. After flux is applied, the assembly is reflowed at the melting temperature of the solder spheres. Cleaning procedures follow, usually involving CFC solvents. For most BGA assembly procedures, flux is only used to allow good reflowing of the solder joints, but for a certain kind of assembly procedure, flux plays a secondary role. Flux must have sufficient retention properties to force the assembly to remain in place through any intermediate processes which precede the reflow of the assembly. For this reason, even if the solder spheres do not need flux to be reflowed, the flux is still needed for its adhesion properties. However, a no-clean flux (lowest active flux, cleaning is not necessary) can be used instead of rosin or water-soluble flux. Fluxless solder spheres enhance the BGA assembly process by eliminating flux dispensing and cleaning procedures, and by making the process environmentally benign.
1.5 Production of Fluxless Solder Spheres for BGA Packaging

Several processes are used for industrial production of solder spheres, all of which have shortcomings in terms of yield and production rates. A widely used process is gas atomization, in which the molten solder is atomized into small droplets that then spheroidize and solidify. The main disadvantage to this process is that the spheres have a very wide size distribution so that intensive classification processes (e.g. sieving) are required. Another commonly used method is stamping, which requires the stamping of disks from a solder foil. The solder disks are then introduced into a variable temperature oil bath, in which the disks melt, spheroidize, and finally solidify. The main shortcoming of the stamping process is its low production rate. The UDS process has proven to be an efficient method of producing mono-sized solder spheres. However, presently the spheres have a rather wide size distribution (± 9% from the mean) and the mean sphere size cannot be reliably placed on the target size. Tighter control of sphere size is necessary to apply the UDS technology to the production of spheres for BGA chip interconnection technology.

Many fluxless soldering technologies are in development, most of which seek to completely avoid or reduce the number of cleaning steps. Work has focused on controlled atmosphere soldering, laser soldering, ultrasonic soldering, and plasma-assisted soldering. None of these new technologies can directly be applied to the production of fluxless solder spheres. Controlled atmosphere and plasma-assisted soldering can be used to reduce or eliminate the oxide layer on the surface of the spheres. However, the assembly must be reflowed in situ, because any exposure to air causes reoxidation. These soldering technologies result in an even more complicated assembly process than the current one. Laser and ultrasonic soldering technologies are extremely localized processes, and by consequence, they are not viable for BGA assembly. A novel fluxless soldering technology that can be very well applied to solder spheres is presented here. This technology transforms the chemical composition of the spheres' surfaces, allowing good reflowing properties without the use of flux.

1.5.1 Control of Solder Sphere Size Distribution

Several steps are taken to achieve control over the size distribution of the solder spheres produced by the UDS process. First, the sphere size variation is reduced. Experimentation determined the sources of sphere size variations. The UDS apparatus was then modified to reduce the size variation from ± 9% to ± 2.5% from the mean size [6-8]. The mean sphere size still has to be placed on the target size. Chapter 2 focuses on the design, development, and implementation of a discrete integral control system for the placement of the mean sphere size on the target. Figure 1.4 shows a block diagram for the implemented control system, showing the major components of the control system: the control input (target sphere size), the plant (UDS apparatus), the control output (actual sphere size), the measurement system, and the controller. The measurement system is based on digital imaging processing. The system acquires an image of the droplets and, based on that image, determines the sphere size. The actual sphere size is compared to the
target size, generating an error signal. This error signal is manipulated by the controller to modify the inputs to the UDS process to bring the actual sphere size in line with the target size.

![Block diagram for the sphere size distribution control system.](image)

**Figure 1.4** Block diagram for the sphere size distribution control system.

### 1.5.2 Production of Fluxless Solder Spheres

A process that transforms the chemical composition of the spheres' surfaces is implemented for the production of fluxless solder spheres. This process converts metal oxide into oxyfluorides. The oxyfluoride layer on the surface of the spheres prevents further oxidation when in contact with air, but breaks when the solder spheres are reflowed, allowing clean solder to flow and form the joint. The oxide conversion process involves the exposure of the solder spheres to a highly active fluorine environment. The oxide conversion process is discussed in detail on Chapter 3. A plasma generator was developed to provide the reactive fluorine environment. The oxide conversion process is optimized to find the best process input parameters. These parameters include: plasma power, plasma pressure, mixture of gases, and treatment time. The development of the plasma generator and process optimization are presented in Chapter 4. Finally, the UDS process and the plasma-assisted oxide conversion process are integrated for the continuous production of fluxless solder spheres. The performance of the fluxless spheres is evaluated in terms of spreading after reflow by comparing the results of the fluxless spheres to the spreading obtained using a highly active flux. Chapter 5 presents process integration and evaluation of the fluxless solder spheres.
CHAPTER 2 ON-LINE DROPLET SIZE CONTROL FOR THE PRODUCTION OF SOLDER SPHERES USING THE UDS PROCESS

2.1 Solder Sphere Generation Using the UDS Process

The UDS apparatus was developed based on Lord Rayleigh’s liquid jet instability concept [9,10]. Rayleigh proposed that the surface tension force of a liquid causes a jet to break into a train of droplets. This phenomenon is known as capillary instability. Rayleigh also suggested that uniform droplets could be produced by subjecting the jet to a periodic oscillation of a wavelength greater than the circumference of the jet diameter [11]. The UDS process generates uniform metal droplets based on the capillary instability theory. Figure 2.1 shows a schematic of the UDS droplet generator. The droplet generator consists of a crucible in which the metal is melted. A pressure difference between the interior and exterior of the crucible forces the metal out through an orifice. Once a laminar jet is established, uniform droplets are generated by the controlled perturbation of the jet. A stack of piezoelectric crystals produces the perturbation. It is transmitted to the melt through a shaft to avoid exposing the crystals to high temperatures. The droplets are charged by electrostatic induction to prevent merging during flight. Then they are either solidified in an inert gas or are quenched in a liquid bath to form the solder spheres.

![Diagram of UDS droplet generator]

**Figure 2.1** The uniform droplet generator.
2.2 Effects of the Process Parameters on Droplet Size Variation

For the droplets generated from the breakup of a liquid jet, the droplet size can be determined by performing a mass conservation analysis at the moment of the break up of the jet into droplets. In this process, a cylindrical metal jet with a diameter \( d_j \) and velocity \( V_j \) breaks into droplets at a rate of \( f \). Assuming that the jet breaks into small cylinders of diameter \( d_j \) and length (wavelength) \( \lambda = V_j/f \), the volume of each cylinder \( V_{cyl} \) is:

\[
V_{cyl} = \frac{\pi d_j^2 V_j}{4 f} \tag{2.1}
\]

Due to surface tension, each cylinder transforms into a spherical droplet of volume:

\[
V_d = \frac{1}{6} \pi d_d^3 \tag{2.2}
\]

where \( d_d \) is the droplet diameter. By mass conservation, the volume in Equation (2.1) is exactly equal to the volume in Equation (2.2). Thus, the droplet diameter is:

\[
d_d = \left( \frac{3 d_j^2 V_j}{2 V_j} \right)^{1/3} \tag{2.3}
\]

where \( d_j \) is the diameter of the cylindrical jet, \( V_j \) is the jet velocity, and \( f \) the break-up frequency as shown in Figure 2.2.

![Diagram of droplet generation](image)

**Figure 2.2** Droplet generation.
The velocity of the jet $V_j$ can be calculated by using Bernoulli's equation between the surface of the metal and the orifice:

$$V_j = C_{ncz} \left( \frac{2(\Delta p + \rho gh)}{\rho} \right)^{1/2} \quad (2.4)$$

in which the velocity of the jet is determined by the pressure difference $\Delta p$, the height of the metal in the crucible $h$, the gravitational acceleration $g$, the discharge coefficient $C_{ncz}$, and the density of the liquid $\rho$. Assuming that the diameter of the jet is the same as that of the orifice, $d_o$ and substituting Equation (2.4) into Equation (2.3), the droplet diameter is calculated by:

$$d_d = \left( \frac{3 C_{ncz} d_o^2}{2 f} \right)^{1/3} \left( 2 \frac{\Delta p + \rho gh}{\rho} \right)^{1/6} \quad (2.5)$$

Equation (2.5) shows that droplet diameter is a function of vibration frequency, pressure difference, density, and orifice diameter. Therefore, variations in these parameters cause variation in droplet size. Based on the previous equations, small variations in the melt temperature do not have a significant effect on material density. For example, to generate a 1% variation in droplet size, a 90% variation in temperature of eutectic solder is required. However, only a 1.03% variation in frequency or an 8% variation in pressure difference is required to produce a 1% variation in droplet size. The orifice size is another important variable that directly affects droplet size. Although the orifice size does not have a significant effect on droplet size variation, the orifice size does define the range of sphere sizes that can be produced. Currently, the UDS process employs a sapphire orifice. The orifice is measured at the beginning and end of each experiment. No changes in size within $\pm 2 \mu m$ have been detected, even though orifices are reused. Therefore, the orifice size is considered to be constant. Since the orifice size does not change, and small variations in temperature can be disregarded, the pressure difference and the breakup frequency are the parameters that can be controlled to determine droplet size distribution.

### 2.3 Solder Sphere Size Control

The first step in the implementation of the new control system involved the development of a system to measure droplet size in flight. This measurement system is based on digital image analysis. That is, a picture of the droplets is captured by a CCD camera, and analyzed, to determine the droplet size. The development of the measurement system began by determining the best position to acquire the image along the stream of droplets. A good understanding of the droplet trajectory is essential to ensure that the acquired image represents actual droplet size. The position and shape of the droplets change as they travel due to drag force, electric charge, surface tension, and gravitational force [12]. Analysis of droplet trajectory and shape oscillations [7,13] showed that the best place for the camera is within 30 mm of the jet break up position for 760 $\mu m$ droplets. Figure 2.3 shows a typical image taken at 20 mm from the jet break up
position. As can be seen from the picture, the droplets are not completely spherical at this point. However, by the time the droplets are completely spherical, they are too dispersed to be captured in focus. Thus, an image of spherical droplets would not represent their actual size.

![Image of Sn-38 wt. %Pb droplets.](image)

**Figure 2.3** Image of Sn-38 wt. %Pb droplets.

The droplet detection process begins by using edge detection to identify the droplet. Edge detection on a digital image consists of the detection of discontinuities, or places with an abrupt change in gray level, that indicate the end of one region and the beginning of another. Figure 2.4 shows the edge detection process; Figure 2.4a shows the gray-level intensity distribution for a cross section of a droplet. The exact position of the edge is where the gradient of the intensity distribution has a maximum absolute value (Figure 2.4b) or where the second derivative has a zero value (Figure 2.4c). For a complete picture, the detection process starts by scanning the image from the center towards the sides until a droplet is detected (Figure 2.5a). Then, the process scans the area in which the droplet was found, first row by row looking for edges within the same row (Figure 2.5b); and then column by column looking for edges within the same column (Figure 2.5c). The position of the edges exactly represents the contour of the droplet. The scanning process determines the exact positions of all horizontal and vertical edges of the droplets in the image (Figure 2.5d).

### 2.3.1 Droplets Size Measurement Method

With regard to droplet size, the accuracy of a given measurement strongly depends on the particular measurement method employed. There are two approaches to obtaining droplet size. One approach consists of determining the droplet size by measuring the diameter of the droplet in the image, while another approach involves correlating either a geometric, or a physical parameter to the droplet size. Since the droplets contained in a typical image are not circular, measuring the diameter from an image reduces the precision and accuracy of the measurement, unless a 3-D image can be obtained. Since a 3-D measurement is difficult to obtain, the second approach was
investigated in this study. Three measurement methods were tried, two using geometrical relations and one based in a physical relation.

Figure 2.4  Edge detection process.

Figure 2.5  Droplet detection process.
The first measured the area of each droplet in the picture to calculate the droplet diameter. The second obtained the droplet diameter from the droplet volume. The volume was obtained by assuming that the shape deformation of the droplets is axis-symmetric, thus, the total volume of a droplet can be calculated by dividing the droplet into discs of thickness one pixel and diameter of the width of the droplet at that row, and then adding all the discs to obtain the total volume. The third method measured the wavelength. The wavelength represents the distance from center-to-center between two droplets. The center is located by determining the geometric center of each droplet. Then, the wavelength is determined by the following equation:

$$\lambda_{1-2} = \frac{V_j}{f} = \sqrt{(x_1 - x_2)^2 + (y_1 - y_2)^2}$$  \hspace{1cm} (2.6)

where $V_j$ is the jet velocity, $f$ is the driving frequency, $(x_1, y_1)$ and $(x_2, y_2)$ are the position coordinates of the centers for droplet 1 and 2, respectively. Then, using Equations (2.4), (2.5), and (2.6) the droplet diameter is:

$$d_d = \left( \frac{3}{2} \frac{d_1^2 \lambda}{d_d} \right)^{1/3}$$  \hspace{1cm} (2.7)

An evaluation procedure was developed to determine which method is best for the measurement of the droplets. This procedure determined the variation that each measurement method added to the process. If the UDS process has a variation of $\pm 2.5\%$ from the mean sphere size, a good measurement method should detect a variation of exactly that magnitude. Table 2.1 shows the results of the evaluation. Based on these results, it can be concluded that measuring wavelength is a very accurate method for determining droplet size.

<table>
<thead>
<tr>
<th>Measurement Method</th>
<th>Variation detected</th>
</tr>
</thead>
<tbody>
<tr>
<td>Area measurement</td>
<td>3.25%</td>
</tr>
<tr>
<td>Volume measurement</td>
<td>4.53%</td>
</tr>
<tr>
<td>Wavelength measurement</td>
<td>2.51%</td>
</tr>
</tbody>
</table>

2.3.2 Calibration Procedure for the Measurement System

High precision, good sensitivity, and adequate resolution are necessary but not sufficient to guarantee a correct measurement. The measurement system first acquires an image of the droplets using a CCD camera. This image is transformed from analog to digital by a frame grabber. The frame grabber divides the image into a 769 (horizontal) x 494 (vertical) pixel array. Thus, all image analysis and droplet detection process use
pixels as the measurement unit. These pixel values have to be converted into length units to produce a measurement in micrometers. The accuracy of the measurement directly depends on the determination of the relationship between the measurement in pixels and the real value of this measurement in units of length (μm). This relation is determined by a camera calibration that accounts for optical distortion and image amplification. The camera calibration procedure starts by positioning a fixture with three circles of known size and wavelength as if they were droplets coming out of the orifice. The camera is then focused and the distance from center to center (wavelength) is measured. As a result, the wavelength is determined in number of pixels and an accurate relationship between the pixels and length units can be determined. This relation is defined as a camera calibration constant:

$$\delta_{\text{camera}} = \frac{\lambda_{\text{real}}}{\lambda_{\text{measured}}} = \frac{[\mu\text{m}]}{[\text{pixel}]}$$

where $\delta_{\text{camera}}$ is the calibration variable, $\lambda_{\text{real}}$ the real wavelength and $\lambda_{\text{measured}}$ the wavelength determined by the measurement system.

### 2.3.3 Droplet Solidification Effects

Once the droplet size has been measured, it is necessary to relate the size of the liquid droplet to the size of the solidified sphere. Volume shrinkage occurs during solidification because of the change in density from the liquid to the solid phase. The sphere diameter is defined as:

$$D_b = \left(\frac{\rho_l}{\rho_s}\right)^{1/3} D_d$$

where $D_b$ is the sphere diameter, $\rho_l$ the density of the liquid metal, $\rho_s$ the density of the solid metal at 25°C, and $D_d$ the liquid droplet diameter. This equation results in the definition of a new variable $\delta_{\text{sol}}$ that accounts for solidification shrinkage:

$$\delta_{\text{sol}} = \left(\frac{\rho_l}{\rho_s}\right)^{1/3}$$

For the production of large Sn-38 wt. % Pb (200-800 μm) spheres using a melting temperature of 235°C, $\delta_{\text{sol}}$ is approximately equal to 0.9848. This means that the diameter of the solder sphere is 1.52 % smaller than the diameter of the droplet.

Using Equations (2.7), (2.8), and (2.10), the final sphere diameter in micrometers is:

$$D_b = \delta_{\text{sol}} \left(\frac{3}{2} d_o \lambda \delta_{\text{camera}}\right)^{1/3}$$

25
2.3.4 Modeling of the Sphere Size Control System

The control of any process starts with the definition of the control input and output. Droplet size is the control output in this study. Equation (2.5) shows that the UDS process has two control input variables: vibration frequency and pressure difference. Vibration frequency is examined here because of its faster response, higher accuracy, and greater sensitivity. The control system was developed based on response requirements. The most important requirement is a response with no steady state error. Also important is a well-damped response, to avoid the sphere size variation caused by an oscillatory response. Because UDS dynamics are nonlinear, as shown in Equation (2.11), the plant model has to be a linearized at some particular point. The linearization of Equation (2.11) is defined by:

\[ D_h(t) = \bar{D}_h + \delta_{sol} \left( \frac{3}{2} d_o^2 \delta_{camera} \right)^{1/3} \left[ \frac{1}{\bar{\lambda}^{2/3}} \lambda(t) - \frac{\bar{\lambda}^{1/3}}{3} \right] = C + C_o \lambda(t) \]  \hspace{1cm} (2.12)

where the linearization is performed around the point:

\[ \bar{D}_h = C + C_o \bar{\lambda} = C + C_o \frac{\bar{V}_i}{f} \]  \hspace{1cm} (2.13)

The wavelength linearization is defined by:

\[ \lambda(t) = \bar{\lambda} + \frac{V_1(t)}{f} - \frac{\bar{V}_i}{f^2} f(t) \]  \hspace{1cm} (2.14)

Equation (2.4) shows that the jet velocity is only a function of the pressure difference and the hydrostatic pressure applied by the weight of molten metal. Because the pressure difference \( \Delta p \) remains constant, the jet velocity is only a function of the height of the metal in the crucible \( h(t) \). By linearizing Equation (2.4) and substituting in equation (2.14), the resulting wavelength is:

\[ \lambda(t) = C_1 + C_2 h(t) + C_3 f(t) \]  \hspace{1cm} (2.15)

Equations (2.12) and (2.15) are the linearized equations that describe the dynamics of the UDS process. After discretizing these equations and taking the z-transform the resulting equations are:

\[ D_h(z) = C_o \lambda(z) \]  \hspace{1cm} (2.16)

\[ \lambda(z) = C_2 h(z) + C_3 f(z) \]  \hspace{1cm} (2.17)

Figure 2.6 shows a block diagram for the linearized dynamic model of the UDS process. The hydrostatic pressure change is modeled as a constant disturbance.
represented by the change in height of the metal in the crucible \( h(z) \). The controller is represented by \( G_c(z) \) having the error signal \( e(z) \) as input and the frequency \( f(z) \) as output.

![Block diagram](image)

**Figure 2.6** Block diagram for the droplet size control of the UDS process.

The model linearization produces an inaccurate gain value that results in a steady state error, even though the model is sufficiently accurate for good feedback control design. The solution is to incorporate an integral controller to the system and then find the optimal integral gain that produces a fast settling time and no steady state error \([14-16]\). Integral control is useful in eliminating steady-state errors due to constant disturbances or reference-input commands, such as the change in hydrostatic pressure. An integral (I) controller was implemented. The I-controller changes the frequency at a rate proportional to the integral of the error signal. Equation (2.18) shows the I-controller with the error, \( e(t) \), as the controller input and the frequency, \( f(t) \), as the controller output:

\[
f(t) = K \int_0^t e(t) \, dt \tag{2.18}
\]

Differentiating Equation (2.18) and using Euler's backward difference equation results in:

\[
f(k) = f(k-1) + K T_{\text{sampling}} \, e(k) \tag{2.19}
\]

where \( K_i = K T_{\text{sampling}} \) is the integral gain, and \( T_{\text{sampling}} \) is the sampling time. The sampling time is currently 0.8 seconds, the time that the system takes to acquire an image, process the image to measure the droplet's size, send the feedback signal to the UDS process, and get ready to acquire another image. Taking the z-transform of Equation (2.19), \( G_c(z) \) is defined by:

\[
G_c(z) = \frac{f(z)}{e(z)} = K_i \frac{z}{z-1} \tag{2.20}
\]
2.3.5 Control System Stability

An important issue in the development of the controller is the definition of the optimal integral gain. The process of defining the optimal integral gain starts by finding the range of gain values at which the system gives a stable response. The range in which the liquid jet can be broken uniformly defines response stability for the UDS process. Rayleigh postulated that if the jet is perturbed at the exit of the orifice, the disturbance grows exponentially with time and sinusoidally with space. Rayleigh derived a growth factor that determined how fast the neck on the surface of the liquid jet contracts. The growth factor is defined by:

\[ q = \frac{\sigma}{\rho d_j^3} \left[ -6(Oh)\gamma^2 + \sqrt{36(Oh^2)\gamma^4 + 4(1-\gamma^2)\gamma^2} \right] \]  

(2.21)

where \( \sigma \) is the surface tension, \( \rho \) is the density of the metal, \( Oh \) is the Ohnesorge number and \( \gamma \) is the dimensionless wave number defined by:

\[ Oh = \frac{\mu}{\sqrt{\rho d_j \sigma}} \]  

(2.22)

\[ \gamma = \frac{\pi d_j}{\lambda} \]  

(2.22)

Figure 2.7 shows the dimensionless growth factor \( q/(\rho d_j^3)^{1/2} \) as a function of the dimensionless wavelength \( \lambda/d_j \). The maximum growth factor is obtained at \( \lambda/d_j = 4.508 \). This value gives the optimal liquid jet breakup parameters, that is, the UDS process response is most stable when the dimensionless wavelength is close to 4.5. The range for response stability was experimentally determined. The UDS process consistently generates a uniform and stable jet breakup when the dimensionless growth factor is equal or greater than 0.8. Figure 2.7 shows that the range for stable breakup is within \( 3.6 \leq \lambda/d_j \leq 7.0 \). At constant jet velocity the limits for stability can be expressed in terms of breakup frequency \([f_l, f_u]\).

The breakup frequency stability limits are used to find the optimal integral gain. To ensure response stability, the controller output has to be contained within the frequency limits defined by:

\[ f_l \leq f(k) = f(k-1) + K_i e(k) \leq f_u \]  

(2.24)

The largest error \( e(k) \) occurs at time \( t=0 \), because after that time the error keeps decreasing due to the integral action of the controller. Consequently, the largest controller response, \( f_o \), occurs at time \( t=0 \). The integral gain is then defined by:
\[ K_I \leq \min \left[ \frac{f_u - f_o}{e_o}, \frac{e_o}{f - f_o}, \frac{e_o}{e_o} \right] \]  \hspace{1cm} (2.25)

where:

\[ e_o = D_o - D_t \]  \hspace{1cm} (2.26)

\[ D_o = \left( \frac{3d_o^2 V_o}{2f_o} \right)^{1/3} \]  \hspace{1cm} (2.27)

where \( f_o \) is the initial breakup frequency and \( V_o \) is the jet initial velocity. As Equation (2.25) shows the optimal integral gain is a function of the target size. The process of defining the optimal integral gain starts by finding the gain values according to Equation (2.25) at which the system gives a stable response. Once this is done, fine-tuning is performed by running the UDS process with the control algorithm.

![Graph](image)

**Figure 2.7** Dimensionless growth factor versus dimensionless wavelength.

### 2.4 Experimental Setup and Procedure

Figure 2.8 shows a schematic representation of the control system. It acquires an image using a CCD camera (Pulnix Co., TM-7X) and a zoom macroscope tube
(Meijilabax, UNIMAC). At the background of the image a stroboscope lamp (Pioneer, DS-303) flashes at 1/10 the frequency at which the droplets are generated. The flashing of the lamp freezes the droplets in space so the system can acquire a clear image. The video signal is sent to the frame grabber card (Data Translation, DT3155). The frame grabber and a function generator (Signametrics Co., SM-1020) that generates the feedback signal are the main components of the control system. These two components are PCI bus cards and are coupled by a computer (Dell, Dimension Pro200n). Customized image processing software processes and measures the droplets from the acquired image. This software includes the control algorithm that coordinates the image processing and the feedback control. The control algorithm incorporates the I-controller as shown in Figure 2.9. The algorithm starts by defining the inputs. These inputs are target sphere diameter ($D_t$), orifice diameter ($d_o$), vibration amplitude ($v$), initial vibration frequency ($f_0$), integral gain ($K_I$), and calibration constants ($\delta_{camera}$ and $\delta_{ud}$). The process then acquires an image. If the acquired image is clear for processing and wavelength measurement, the process calculates the sphere diameter, then the error, and finally the frequency adjustment to alter the size of the droplets. The system remains in the control loop until the process is stopped.

![Schematic of the UDS closed loop control system.](image_url)

**Figure 2.8** Schematic of the UDS closed loop control system.
Figure 2.9  Flow chart of the control system

To evaluate the control system in terms of sensitivity to disturbances and transient and steady state response, the following experimental procedure was applied. The experimental procedure consisted of the controlled production of solder spheres. A target sphere diameter was chosen and spheres were produced using the UDS process and the control system. First the chamber was evacuated to about 0.16 Torr and back-filled three times with nitrogen. Then the solder in the crucible was melted and a positive pressure difference forced the solder through the orifice. Once a laminar jet was obtained, it was subjected to forced vibrations at the initial frequency. The control system was started after the jet broke into uniform droplets. The liquid solder droplets landed in a container filled with silicon oil that allowed the droplets to spheroidize and solidify. Approximately 0.5 Kg of material was collected for each experiment. The spheres were cleaned using baths of acetone and alcohol. A representative sample was measured with an optical microscope. Figure 2.10, 2.11, and 2.12 show scanning electron microscopy (SEM) photographs of 760 μm, 500 μm, and 300 μm diameter solder spheres. Actual sphere size was compared with the measurements made by the control system and with the simulation results.
**Figure 2.10**  SEM picture of 760 µm 63Sn/37Pb spheres.

**Figure 2.11**  SEM picture of 500 µm 63Sn/37Pb spheres.

**Figure 2.12**  SEM picture of 300 µm 63Sn/37Pb spheres.
2.5 Experimental Results and Discussion

2.5.1 Testing the Control System Sensitivity

Sensitivity is defined as the ability of the control system to respond to sudden changes in the input parameters. To test the sensitivity of the control system, the gas pressure was varied within a run, forcing a response from the control system. Table 2.2 shows the input parameters for this experiment. The pressure disturbances were introduced at 19, 39, and 58 seconds. Figures 2.13a and 2.13b show the response of the system to the disturbances and the adjustments in frequency that were made to compensate for the disturbances, respectively. These results show that the control system provides a fast response to the induced perturbations: the settling time for an induced ball-size variation of 3% from the target ball size is only 3.4 seconds.

Table 2.2 Experiment Parameters for Control System Sensitivity Experiment

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material</td>
<td>Sn-38 wt. % Pb</td>
</tr>
<tr>
<td>Target sphere diameter</td>
<td>760 µm</td>
</tr>
<tr>
<td>Orifice diameter</td>
<td>406 µm</td>
</tr>
<tr>
<td>Pressure difference at t = 0 seconds</td>
<td>34.4 kPa</td>
</tr>
<tr>
<td>Pressure difference at t = 19 seconds</td>
<td>39.6 kPa</td>
</tr>
<tr>
<td>Pressure difference at t = 39 seconds</td>
<td>34.4 kPa</td>
</tr>
<tr>
<td>Pressure difference at t = 58 seconds</td>
<td>31.0 kPa</td>
</tr>
<tr>
<td>Initial Vibration frequency</td>
<td>1450 Hz</td>
</tr>
<tr>
<td>K_i</td>
<td>3 (µm s)^{-1}</td>
</tr>
<tr>
<td>T_sampling</td>
<td>0.8 s</td>
</tr>
<tr>
<td>δ_camera</td>
<td>11 µm/pixel</td>
</tr>
<tr>
<td>δ_{sol}</td>
<td>0.9848</td>
</tr>
</tbody>
</table>

2.5.2 Control System Steady State Response

Table 3 shows the experimental parameters for three different target diameters. Figure 2.14 shows a controlled response of the UDS process for target diameters of 760 µm (Figure 2.14a), 500 µm (Figure 2.14b), and 300 µm (Figure 2.14c). The thick line represents the actual response of the system and the thin line, the simulated response using the model previously discussed. The simulated response very closely follows the actual response of the system; thus the numerical simulation represents a reliable method of predicting the response of the system. The integral gains were set to have a settling time of approximately five seconds for all target diameters. The settling time could be reduced even further by increasing the integral gains, however, there is a trade-off between settling time and sphere size variation. When the integral gain is higher than the values used in the experiment, the response of the system increases in the final sphere.
size variation. Figure 2.15 shows the feedback (frequency) control signal corresponding to target diameters of 760 μm (2.15a), 500 μm (2.15b), and 300 μm (2.15c). In this case as well, the prediction of the control feedback signal agrees with the actual control input.

**Figure 2.13** Disturbance response of the UDS control system.
Table 2.3  Experimental Parameters for the Three Different Target Diameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Target diameter</td>
<td>$D_1$</td>
</tr>
<tr>
<td>Orifice diameter</td>
<td>$d_o$</td>
</tr>
<tr>
<td>Pressure difference</td>
<td>$\Delta p$</td>
</tr>
<tr>
<td>Vibration frequency</td>
<td>$f$</td>
</tr>
<tr>
<td>Initial jet velocity</td>
<td>$V_j$</td>
</tr>
<tr>
<td>Melt temperature</td>
<td></td>
</tr>
<tr>
<td>Integral gain</td>
<td>$K_1$</td>
</tr>
<tr>
<td>Sampling time</td>
<td>$T_{\text{sampling}}$</td>
</tr>
<tr>
<td>Camera constant</td>
<td>$\delta_{\text{camera}}$</td>
</tr>
<tr>
<td>Shrinkage constant</td>
<td>$\delta_{\text{sol}}$</td>
</tr>
<tr>
<td>Collection time</td>
<td></td>
</tr>
<tr>
<td>Spheres collected</td>
<td></td>
</tr>
</tbody>
</table>

2.5.3 Sphere Size Distribution Statistics.

The results shown in Figures 2.14 and 2.15 confirm that the control system works properly. However, these results are based on the simulation and control system itself. The most important evaluation parameter is the actual sphere size distribution generated by the control process. Four experiments were conducted for each target size and a random sample of 250 solder spheres was selected for each experiment to estimate the mean and variance of the actual distribution. The mean of the distribution was estimated using the sample average. Since the sample was taken randomly, the Central Limit Theorem applies, which states that the sample average has a normal distribution with mean equal to the population mean and variance equal to the variance of the size divided by the sample size. The criterion used to select the sample size is to construct confidence intervals with reasonably small lengths. The estimates indicate that the value of the mean sphere size is within the intervals shown in Table 2.4 with a confidence of 99%. The spheres were measured using an optical microscope. The parameter measured was the sphere diameter. The diameter was measured at two positions perpendicular to each other; the value presented is the average of the two measurements. Table 4 shows the statistical properties of the sphere size distributions for 760 $\mu$m, 500 $\mu$m, and 300 $\mu$m target diameters. Figure 2.16 shows the normalized sphere size distributions. The mean sphere size is on target for all target sizes. Sphere size variation is within specification: all the spheres are $\pm$ 3% from the target diameter for all three target sizes.
**Figure 2.14** Steady state response of the controlled UDS process for target diameters of a) 760 μm, b) 500 μm, and c) 300 μm.
Figure 2.15  Feedback control signal for target diameters of a) 760 μm, b) 500 μm, and c) 300 μm.
Table 2.4  Statistical Results for the Controlled Production of Solder Spheres

<table>
<thead>
<tr>
<th>Parameter</th>
<th>( D_s=760 ) ( \mu )m</th>
<th>( D_s=500 ) ( \mu )m</th>
<th>( D_s=300 ) ( \mu )m</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean sphere diameter</td>
<td>759.25 ( \mu )m</td>
<td>501.27 ( \mu )m</td>
<td>300.16 ( \mu )m</td>
</tr>
<tr>
<td>Confidence intervals for the mean (( \alpha = 0.01 ))</td>
<td>[758.50, 759.99]</td>
<td>[500.58, 501.95]</td>
<td>[299.78, 300.53]</td>
</tr>
<tr>
<td>Standard deviation</td>
<td>4.61 ( \mu )m</td>
<td>4.19 ( \mu )m</td>
<td>2.33 ( \mu )m</td>
</tr>
<tr>
<td>Production percentage within ( \pm 3 )% of the target size</td>
<td>99.9998%</td>
<td>99.9051%</td>
<td>99.9839%</td>
</tr>
</tbody>
</table>

Figure 2.16  Normalized sphere size distribution.

2.6  Summary

The results of the control system experiments demonstrate that the UDS process can now produce solder spheres for BGA chip interconnection technology. The control system enables the UDS process to produce spheres with a size variation smaller than \( \pm 3 \)\% of the target size. Even more importantly, the control system allows the monitoring and control of the mean sphere size. The mean sphere size now can be exactly placed on the target size. The I-controller is effective in maintaining the sphere size on target with a short settling time and a well-damped response. This control system is responsive to any noise or disturbance on the expelling pressure. The measurement and control systems were developed specifically for the UDS process. However, they can be adapted to any other kind of droplet generation system based on the uniform breakup of a liquid jet.
CHAPTER 3  FLUXLESS SOLDERING TECHNOLOGIES, SOLDER SPHERE OXIDATION, AND PLASMA-ASSISTED OXIDE CONVERSION PROCESS

3.1 Fluxless Soldering Technologies

The Montreal protocol established that CFCs are to be phased out by the year 2000 in developed countries and 10 years later throughout the world. These measures provide incentives to develop alternative manufacturing technologies that are environmentally responsible. This is particularly true for microelectronics soldering. Conventional soldering requires flux to promote wetting of the base of the metal by the solder alloy (Figure 3.1). The flux chemically removes surface oxides, provides a protective layer to the clean surface during soldering, assists heat transfer, removes reaction products, and modifies the interfacial surface tension to enhance spreading of the molten solder. Flux residues are highly corrosive and can cause long term reliability problems, consequently, removal of flux from the assembly is mandatory. Rosin-based flux residues are normally removed with halogenated or CFC solvents. New fluxes, solvents, and cleaning methods are being developed to meet the CFC restriction. Aqueous based cleaning, water-soluble fluxes, and low solids (no-clean) fluxes are showing good results. Alternative fluxless-soldering technologies [17-20] are also being developed to completely reduce or eliminate the number of cleaning steps. The work on developing new soldering technologies has focused on controlled atmosphere soldering, laser soldering, ultrasonic soldering, plasma-assisted soldering, and protective coating processes.

Figure 3.1  Schematic of the oxide removal process using flux.

3.1.1 Controlled Atmosphere Soldering

Controlled atmosphere soldering utilizes low oxygen content or reducing atmospheres to maintain or produce a solderable base surface. The atmospheres usually depend on vacuum, an inert or reducing gas, or an acid vapor-gas mixture. Although the reduction of metal oxides in hydrogen and vacuum is possible, the rate of reaction at typical soldering temperatures (200-300°C) is extremely inefficient and slow. In contrast, diluted acid vapor is much more effective in reducing the surface oxide at soldering temperatures. Acid vapor soldering involves passing a carrier gas and an acid vapor over the surface of the metal to be joined [21]. The acid chemically attacks the surface as the
metal is heated, removes the oxide from the surface, and activates the cleaned surface for wetting by the solder. The acid vapor process can also prevent the solder surface from re-oxidation. This process is usually applied to solder preforms, but it also can be used in wave soldering (a process where assemblies are dragged through a molten solder bath). Acid vapor soldering is not widely accepted because of the use of highly toxic substances. New research on this area is focusing on the use of alternative controlled atmosphere soldering processes that make inert or non-toxic gases more active so they can provide a faster reaction rate.

3.1.2 Laser Soldering

Approaches to fluxless soldering can vary depending on the application. For applications that have low joint count and require very localized soldering, laser soldering is a promising technology [22]. This technology uses a laser beam to excite an otherwise non-reactive gas in the presence of a preheated solder surface, as shown in Figure 3.2. The activated gas components react with the solder to reduce the oxide in the surface. Since the oxide layer is usually thin, the oxide reduction is completed in a few pulses of laser radiation. The complete assembly is reflowed after all solder joints have been exposed to the laser beam. Laser soldering is a remarkably localized process in the sense that the beam has to be applied to each of the solder joints. This along with the fact that the reflow of the assembly has to be performed immediately after the oxide removal are the main shortcomings of laser soldering. However, for assemblies where the joints are few and reasonably accessible laser soldering is a feasible technology.

![Schematic of laser soldering.](image)

**Figure 3.2** Schematic of laser soldering.

3.1.3 Plasma Assisted Soldering

Plasma can be defined as a system of electric neutrality composed of positive and negative charge carriers. The degree of ionization can vary from small, to very high (e.g. nuclear reactions). Within these regions, gases may display a wide variety of physical and chemical properties entirely different from those displayed under normal conditions. Plasma-assisted soldering processes can be divided into three main categories: first,
plasma is used to activate inert or reducing gases at soldering temperatures; second, plasma is used to sputter or etch away the oxide in the surface; and third, plasma transforms the chemical composition of the surface, leaving an active surface that allows fluxless reflowing. In controlled atmosphere soldering, plasma is used to generate ionic or atomic gases, which have the greatest potential for effectively reducing surface oxides. Other fluxless soldering processes use free electrons or ions generated by the plasma to bombard the surface of the metal and sputter the oxide layer. These processes have a huge shortcoming: reflow has to be done in situ, as any exposure of the surfaces to air after the reduction process would result in the re-growth of oxide, even at room temperature. There is, however, another use of plasma that can promote a fluxless soldering. Plasma generates ions, electrons and radicals that when in contact with the metal, promote chemical reactions transforming the surface composition. The chemical conversion can be controlled such that the new composition prevents oxidation or assists reflowing. This technology has the advantage that it prevents reoxidation, even when exposed to air for several days. Thus, transforming the chemical composition of the spheres' surfaces represents the best approach to the generation of fluxless solder spheres.

3.2 The Oxidation of 63Sn/37Pb Spheres

Fluxless soldering technologies can be applied to several different processes depending on the application and reflowing conditions. The oxidation phenomena of the solder spheres should be fully understood to efficiently develop and implement a fluxless soldering technology. BGAs use Sn/Pb alloy solder spheres, the most used alloys are eutectic (63Sn/37Pb) and high melting temperature alloys (10Sn/90Pb). In the BGAs' assembly process, eutectic solder spheres are actually melted to form the solder joints, while the 10Sn/90Pb spheres form the joint using eutectic solder paste. Thus, the reflow requirements (wetting, spreading, balling, etc.) for the eutectic solder spheres are much more demanding than those for the 10Sn/90Pb spheres. Consequently, if the process is developed to produce fluxless eutectic solder spheres, it will guarantee its effectiveness for 10Sn/90Pb spheres. This work focuses on the study of eutectic solder spheres.

3.2.1 Oxidation of Sn-Pb alloys

Research on soldering has focused on the mechanical properties of solders. However, very little is known about the oxidation properties of solder. There is no information available about the solid state of oxidation kinetics of Sn-Pb alloys. But several studies have been done using Auger Electron Spectroscopy (AES) to understand the process of oxidation [23-29]. Particularly, oxidation of the eutectic (63Sn/37Pb) solder has been widely studied using this method. It has been found that the eutectic Sn/Pb alloy oxidizes logarithmically between 22°C and 90°C and parabolically at temperatures greater than 120°C. For both the logarithmic and the parabolic oxidation, oxygen is believed to be the diffusing species. Konetzki [24,25] proposed the following equation to describe the oxidation for eutectic solder at lower temperatures:
\[ X = A + B \ln(t) \quad 22^\circ C \leq T \leq 90^\circ C \]  

(3.1)

where \( X \) is the oxide thickness, \( t \) is the oxidation time in hours, \( A \) and \( B \) are experimentally determined parameters. And for higher temperatures Konetzki defines the oxide thickness as:

\[ X^2 = (X^o)^2 + K^o \exp\left(-\frac{Q}{RT}\right)t \quad 120^\circ C \leq T \]  

(3.2)

\( R \) is gas constant, \( T \) is the temperature, \( Q \) is the activation energy (67.1 kJ/mole for 63Sn/37Pb), \( K^o = 3.13 \times 10^{-10} \text{ cm}^2/\text{s} \) and \( X^o \) is needed to fit the data to the parabolic oxidation equation. If the spheres do not go through any thermal cycle before they are reflowed, it is expected that the eutectic solder spheres produced using the UDS process oxidize logarithmically. The spheres have shown to grow an oxide thickness that converges to approximately 100 Å [32] as Figure 3.3 shows.

![Figure 3.3 Schematic of a cross section of a eutectic solder sphere.](image)

3.2.2 Examination of Oxide Films on the Solder Spheres

An analytical study of the oxide layer on the solder spheres produced by the UDS apparatus was performed for the development of a process to generate fluxless solder spheres. Auger Electron Spectroscopy (AES) was used to analyze the chemical components of the spheres' surface. The AES technique for chemical analysis of surfaces is based on the Auger radiationless process [30]. When a core level of a surface atom is ionized by an impinging electron beam, the atom may decay to a lower energy state through an electronic rearrangement which leaves the atom in a doubly ionized state. The energy difference between these two states is given to the ejected Auger electron, which will have a kinetic energy characteristic of the parent atom. The energy of these Auger electrons can be used to identify the chemical composition of the solid surface. Figure 3.4 shows the results of the differentiation of the energy function dN(E)/dE obtained by AES analysis on the surface of eutectic solder spheres. The figure shows four main peaks at 94 eV, 271 eV, 430 eV, and 503 eV corresponding to lead, carbon, tin and oxygen, respectively [31]. These are the main elements composing the solder sphere's surface. Tin, lead and oxygen were expected to be found. The presence of carbon shows residual traces of the silicon oil used to solidify the spheres.

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Figure 3.4 Auger profile of the surface of a eutectic solder sphere produced by the UDS process.

A depth profile of the solder sphere's surface can also be obtained using AES and a sputtering ion gun. The surface is analyzed, the ion gun is used to sputter away a layer of material, and the surface is analyzed again. The chemical composition is then known at different depths from the initial surface. Figure 3.5 shows the depth profile of a eutectic solder sphere. This figure shows the atomic concentration for the main chemical components within 100 Å from the sphere's surface. Oxygen is the main component of the outermost surface, indicating a high presence of oxides. As the surface is sputtered, the presence of carbon and oxygen decrease until they disappear. The atomic concentration of tin and lead increases until they reach a steady state approximately at 80 Å. The penetration of carbon into the surface is limited within 60 Å. Carbon indicates the presence of residual silicon oil used for solidifying the spheres. It is not clear whether the carbon actually penetrates the surface or migrates from adjacent areas to the analysis area when sputtering until all residual silicon oil is evaporated. The presence of oxygen shows that the oxide layer thickness for a newly generated eutectic solder sphere is approximately 80 Å. Figure 3.6 shows the depth profile for a newly produced sphere and a sphere that has been stored in air for six months at room temperature. The results of the depth profile show that the atomic concentration of oxygen at the surface is around 15% more for the stored sphere. As the surface is sputtered, the oxygen disappears at 80 Å for the newly generated sphere and at 120 Å for the stored sphere [32].

3.3 Plasma Assisted Oxide Conversion Process

A process that neither cleans nor reduces the sphere’s surface oxides should be selected to produce fluxless solder spheres. That is, if the process merely cleans or reduces the oxide thickness, the oxide layer will re-grow on the solder spheres as soon as
they are in contact with air. A process that converts the surface oxides to oxyfluorides is used for the production of fluxless solder spheres. The oxyfluoride film has the unique property of breaking up when the solder melts, exposing clean solder and allowing reflow and joining to occur. More importantly, the conversion process does not have to be coupled to the reflow process: the oxyfluoride film prevents re-oxidation even when in contact with an oxidizing atmosphere.

**Figure 3.5** Auger depth profile of a eutectic solder sphere.

**Figure 3.6** Atomic concentration of oxygen for eutectic solder spheres.
3.3.1 Plasma Assisted Soldering Background

Dishon and Bobbio patented a plasma-assisted dry soldering (PADS) process in 1990 [33]. This process uses RF-generated plasma to dissociate a fluorinated gas (i.e. CF₄ or SF₆). The solder surfaces to be treated are placed inside the plasma. The process is performed at room temperature and the plasma pressure can vary from 0.005 Torr to 1 Torr. Fluorinated gases are believed to transform the surface oxide during the plasma process due to the higher electronegativity of fluorine or the instability in the fluorine structure. The activation energy needed to convert the oxides is supplied by the excited fluorne radicals in the plasma which diffuse and hit the oxide on the surface. Various experiments were conducted using a RF-plasma system giving promising results. However, temperature control and sample damage due to occasional sputtering was a problem. In 1993, Koopman, Bobbio, and Nangalia modified the process to solve the problems of high temperature plasma and sputtering [34]. These modifications consisted of using microwave-generated plasma and treating the solder surfaces downstream. The solder components are not placed inside the plasma chamber but rather the products of the plasma are brought to the solder surfaces to be treated. They proved that the treated solder can be successfully refloowed after being stored for one week in air or two weeks in an inert environment [35-38].

3.3.2 Dissociation of Fluorinated Gases Generated by Plasma

The oxide conversion process is based on the dissociation of a fluorine-containing gas through the application of plasma. For the current study, sulfurhexafluoride (SF₆) is used for the oxide conversion process. SF₆ is commonly used as an insulating medium in high voltage power systems because of its high dielectric strength (almost three times greater than air at atmospheric pressure), chemical inertness, and non-toxicity. Despite its high level of chemical stability, SF₆ will undergo some decomposition and oxidation in plasma, particularly when molecular oxygen and water vapor are present [39,40]. The by-products of SF₆ plasma dissociation are fluorinated gases that include SF₄, S₂F₁₀, SOF₂, SO₂F₂, SO₃, HF, and F. Low temperature plasma-induced dissociation of SF₆ is known to cause the effective electron temperature (mean electron kinetic energy) to greatly exceed the gas temperature. This temperature difference allows the rate of gas dissociation to be controlled primarily by the rates of electron-impact induced molecular dissociation processes such as:

\[
e + \text{SF}_6 \rightarrow \text{SF}_{6-x} + xF + e \quad x \leq 6
\]  

Equation 3.3 shows that stable SF₆ plasma allows the formation of highly reactive free radicals and ions by electron collisions that subsequently react within a relatively short time among themselves or with neutral gas species. The oxide conversion takes place when these free radicals come in contact with the solder surface. Thus, the time taken to move the free radicals from the plasma to the solder surface should be minimized to avoid recombination with neutral species.
3.3.3 Oxide Layer Conversion Process

The solder surface reactions that take place during exposure to the SF₆ plasma dissociation products are not fully understood. However, several studies have been performed using electron spectroscopy for chemical analysis (ESCA) and electron conversion Mössbauer spectroscopy (CEMS) [41,42]. Both analysis techniques found that the general reaction takes place when the SF₆ dissociates in the plasma into highly reactive atomic fluorine. The fluorine atoms diffuse to the solder surface at which point the following reaction takes place:

\[ \text{SnO}_x + y\text{F} \rightarrow \text{SnO}_x\text{F}_y \]  \hspace{1cm} (3.5)

Figure 3.7 shows a schematic representation of the plasma-assisted oxide conversion process applied to a solder sphere. SnO and SnO₂ are the main surface oxide components for a eutectic solder alloy. It is believed that the presence of these oxides is necessary for the oxide conversion process. The oxide conversion process gradually reduces the surface oxides to tin-fluorine compounds (i.e., SnF₂ and Sn₃F₈) and oxyfluorides. This converted surface layer is brittle and fractures during reflow. The oxyfluoride layer continues to fracture into smaller pieces until the solder penetrates the oxidation layer and forms a good joint without having to remove the surface oxidation with flux or other mechanical means.

\[ e^- + \text{SF}_6 \rightarrow 4 \text{F}^- + \text{SF}_2 + e^- \]

\[ \text{SnPbO}_x + y\text{F}^- \rightarrow \text{SnPbO}_x\text{F}_y \]

\( e^- \)

\( \text{SF}_6 \)

\( 4 \text{F}^- + \text{SF}_2 + e^- \)

\( \text{SnPbO}_x\text{F}_y \)

\( \text{SnPbO}_x + y\text{F}^- \)

\( \text{Sn/Pb Oxyfluoride} \)

\( \text{Sn/Pb Oxide} \)

Before Treatment

After Treatment

Figure 3.7  Schematic of the plasma-assisted oxide conversion process.
3.3.4 Production of Fluxless Solder Spheres Using Plasma-assisted Oxide Conversion Process

The plasma-assisted oxide conversion process shows great potential for the generation of fluxless solder spheres. The implementation of the oxide conversion process to the UDS process was done in three steps. Next, a microwave plasma generator system was developed. This system generates the plasma environment necessary to dissociate the fluorine containing gas. Second, the process was optimized to produce a maximum oxide conversion rate. Operation parameters such as power, gas mixture and pressure, and treatment time were studied to determine the values that produce a maximum oxide conversion rate. Finally, once the process was fully optimized, the UDS process and the plasma-assisted oxide conversion process were integrated for the continuous production of fluxless solder spheres. The next two chapters discuss these three steps in detail and present the process evaluation.
CHAPTER 4  IMPLEMENTATION AND OPTIMIZATION OF THE PLASMA-ASSISTED OXIDE CONVERSION PROCESS FOR THE PRODUCTION OF FLUXLESS SOLDER SPHERES

4.1 Experimental Plasma Generator

There are two methods for plasma generation. These methods are differentiated by the frequency of the excitation source: RF (radio frequency, usually 13.56 MHz) plasma and microwave (2.45 GHz) plasma. The performance of microwave plasma is much superior than that of RF plasma for the current application. Given the same gas pressure conditions, microwave plasma contains more electrons and ions than RF plasma [43-47]. Consequently, a higher density of fluorine free-radicals can be obtained by microwave plasma. Another important advantage is that stable microwave plasma can be produced using lower power, which translates to stable plasma at low temperatures. A higher dissociation rate and stability at low temperatures make microwave plasma a better source for the current application.

4.1.1 Plasma Generator Apparatus

Plasma volumetric uniformity is not an important issue, because the plasma's only purpose is to dissociate a fluorine-containing gas. However, plasma stability is important in maintaining a constant dissociation rate. Based on these principles, a microwave plasma generator was built using a commercial microwave oven (Sharp, Carousel RC320). A microwave oven is a good source for generating stable plasma, but it is quite difficult to control the volumetric stability of the plasma without changing the configuration of the microwave oven. Figure 4.1 shows the plasma generator and Figure 4.2 shows a schematic of the system. The plasma system consists of a microwave oven, a Pyrex chamber, a vacuum pump, a Pirani vacuum gauge and a couple of flowmeters. The Pyrex chamber is inserted into the microwave oven. An iron oxide polymer (SRC Cables Inc., Polyiron) and aluminum mesh prevent the microwaves from radiating to the outside environment. The flowmeters are used to control the mixture ratio of gases (N₂ and SF₆), and along with the vacuum pump, they control the pressure in the plasma chamber. A Teflon substrate holds the solder samples inside the Pyrex chamber. An array of 300 μm holes with a 200 μm spacing was drilled in the Teflon substrate to allow the sample surface to be covered by the plasma.

4.1.2 Plasma Generator Operation

The Pyrex chamber is sealed and vacuumed. When the pressure in the chamber reaches approximately 0.05 Torr, gas is introduced by opening the valves in the flowmeters. One flowmeter controls the amount of N₂ going in and the other the amount of SF₆.
The vacuum rate is kept constant throughout the process; thus, the individual flow rates determine the pressure in the chamber and the concentration of $\text{SF}_6$ in the mixture. The power used for plasma generation is controlled by adjusting the power level of the microwave oven. A microwave oven controls the power level by switching on and off the power, and not by actually changing the magnetron power. Thus, there is actually only one power level (1000 W) for the plasma apparatus. However, this on-off power control can be used to set the exposure time. For example, solder samples can be treated at a continuous high power level for two minutes, or at 33% power level over six minutes. In both of these settings, the net plasma time exposure is two minutes; however, on the second treatment, the balls are exposed to high energy atomic fluorine during a period.
longer than two minutes. Thus, the time of exposure and the power level cannot be treated as two independent variables. The microwave oven is turned on and plasma starts to be generated once the desired pressure is reached. Stable plasma is characterized by generation of a stable glow (red for pure N₂, and blue for pure SF₆). The flow rate for the gases was changed to determine the range for the generation of stable plasma. Table 4.1 summarizes the operating conditions for the generation of stable plasma.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Power</td>
<td>1000  Watts</td>
</tr>
<tr>
<td>Frequency</td>
<td>2.45  GHz</td>
</tr>
<tr>
<td>Maximum Pressure</td>
<td>5      Torr</td>
</tr>
<tr>
<td>Minimum Pressure</td>
<td>0.15  Torr</td>
</tr>
</tbody>
</table>

4.2 Plasma-assisted Oxide Conversion Analysis

A method to measure the oxide conversion rate should be established before trying to optimize the oxide conversion process. Because the conversion process transforms oxides to metal-fluorine compounds or oxyfluorides, the thickness of the oxide conversion layer can be measured by detecting the presence of fluorine. AES can be used to detect the presence of fluorine on the surface and to obtain a depth profile by sputtering layers of material.

4.2.1 Experimental Procedure for Plasma-assisted Oxide Conversion Treatment

Eutectic (63Sn/37Pb) solder discs of approximately 1 mm in diameter and 0.3 mm in thickness are used for the analysis. Discs were chosen over spheres to guarantee the same sputtering rate in the AES analysis. To guarantee the same sputtering rate between samples, a flat surface is preferred, because the same sputtering angle can be achieved every time. Figure 4.3 shows a picture of the discs used. The eutectic solder discs are placed on the Teflon stage inside the Pyrex chamber. Placing the discs inside the plasma produces higher oxide conversion rates than performing a downstream treatment. This is the standard procedure for all optimization experiments. Once the samples are in the chamber, it is closed and vacuumed. When the pressure and gas mixture reach the desired level, the microwave power and timer are set and the oven is turned on. After the oven shuts off, the chamber is flushed with N₂ to evacuate any reaction products left. Then, the chamber is opened and the discs are taken out to be analyzed by AES.
Figure 4.3  Eutectic solder discs used for the process optimization experiments.

4.2.2 Oxide Conversion Layer Thickness Analysis

All samples in this study are analyzed using the following parameters for AES. The parameters for the scanning electron beam and the sputtering ion beam are shown in Table 4.2. These parameters remain constant for the analysis of every sample. The ion beam current directly determines the sputtering rate: a current of 12 nA produces a sputtering rate of approximately 50 Å minute of sputtering for eutectic solder. Two surface scans and a depth profile is performed for each sample. The first scanning is done over the surface of the disc. Figure 4.4 shows the results of the surface scanning for a treated disc using the parameters shown in Table 4.3. Figure 4.4 shows the presence of the same elements as the solder sphere O, C, Sn and Pb because the discs were generated by compressing solder spheres between a two polished steel plates. The energy curve of the treated disc shows a peak at 647 eV which indicates the presence of fluorine. The AES is performed over an area of 10 μm². 20 random points are scanned within this area. After the surface scanning is finished, a depth profile is performed. Figure 4.5 shows the result of the depth profile for the same sample. This figure shows the atomic concentration of each element as a function of depth. It can be clearly seen that the profile of fluorine follows the trend for the profile of oxygen very closely; indicating the presence of oxyfluorides. Oxygen disappears at around 100 Å, while fluorine can still be detected at 150 Å. This behavior can be explained by the presence of metal-fluorine compounds such as SnF₂ and Sn₅F₉. After the depth profile is finished, another AES scanning is performed over the sputtered surface. Figure 4.6 shows the results of this scanning. It detects the presence of only two elements: Sn and Pb. The plasma-assisted oxide conversion process generates a conversion layer of around 150 Å using the parameters shown in Table 4.2. These exact scanning parameters and procedures are to be used for process optimization.
Table 4.2  Parameters used for AES.

<table>
<thead>
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<th>Source</th>
<th>Parameter</th>
<th>Value</th>
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</thead>
<tbody>
<tr>
<td>Electron Gun (Analysis)</td>
<td>Beam Voltage</td>
<td>2 KV</td>
</tr>
<tr>
<td></td>
<td>Filament Current</td>
<td>1.6 A</td>
</tr>
<tr>
<td>Ion Gun (Sputtering)</td>
<td>Beam Current</td>
<td>12 nA</td>
</tr>
<tr>
<td></td>
<td>Raster Size</td>
<td>0.5 mmx0.5 mm</td>
</tr>
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</table>

Table 4.3  Parameters for plasma-assisted oxide conversion process.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Power (Level)</td>
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</tr>
<tr>
<td>Pressure</td>
<td>3 Torr</td>
</tr>
<tr>
<td>SF₆ Flowrate</td>
<td>1300 cm³/min</td>
</tr>
<tr>
<td>N₂ Flowrate</td>
<td>0 cm³/min</td>
</tr>
<tr>
<td>Time</td>
<td>150 seconds</td>
</tr>
</tbody>
</table>

Figure 4.4  AES profile of the surface of a eutectic solder disc treated using plasma-assisted oxide conversion process.
Figure 4.5  AES depth profile of a eutectic solder disc treated using plasma-assisted oxide conversion process.

Figure 4.6  AES analysis at 200 Å from the surface of a eutectic solder disc treated using plasma-assisted oxide conversion process.
4.3 Plasma-assisted Oxide Conversion Process Optimization

The plasma-assisted oxide conversion process was optimized to maximize the oxide conversion rate in eutectic solder. The optimization of the process was performed by using the treatment procedure and analysis described in the previous section. Eutectic solder discs are treated and measured for oxide conversion layer thickness as described in the previous section. The optimization goal is to define process-input parameters that maximize the oxide conversion rate. Design of experiments (DOE) is used to evaluate how the input parameters affect the oxide conversion rate. The input parameters for the process are plasma power, gas mixture ratio, pressure and flow rate, and treatment time. The effect of each of these parameters over the oxide conversion layer thickness is presented below.

4.3.1 Plasma Power and Frequency

The power and frequency are fixed parameters for the experimental plasma generator. These parameters are set by the microwave oven specifications: 1000 watts and 2.45 GHz. SF$_6$ plasmas have been widely studied [48-53], and specifically those generated using a 2.45 GHz source. In the case of SF$_6$ plasmas, ion densities such as SF$_3^+$, SF$_2^+$, and SF$_5^+$ have been measured. However, for the current study, the parameter of interest is the concentration of atomic fluorine in the plasma, because it is directly related to the oxide conversion rate. The F atom concentration can be measured using optical emission actinometry. Figure 4.7 shows the plot of the F atom density as a function of microwave power input at 2.45 GHz with SF$_6$ as the gas source. The concentration of fluorine increases with the pressure, with clear saturation above 600 watts of microwave power. The saturation in the fluorine level indicates that the dissociation of SF$_6$ is practically complete at this power level. An additional argument which favors the complete dissociation of SF$_6$ is that the saturation level is proportional to the SF$_6$ pressure. A power of 1000 W at 2.45 GHz provides an optimum power level for the maximization of atomic fluorine generation. In terms of power and frequency, the microwave oven provides an optimum environment for the oxide conversion process.

4.3.2 Gas Mixture for Plasma-assisted Oxide Conversion

A mixture of nitrogen (N$_2$) and sulfur hexafluorine (SF$_6$) is used for the generation of atomic fluorine. N$_2$ is commonly used to increase the production of oxyfluorides (SOF$_2$, SO$_2$F$_2$, SOF$_4$) in SF$_6$ plasma. The presence of N$_2$ accelerates the decomposition of SF$_6$ at a given power and total pressure. However, the oxide conversion rate is the parameter to maximize in this study. Several experiments were conducted to measure the oxide conversion rate in eutectic solder as a function of gas mixture. The experiments were performed by treating 63Sn/37Pb solder discs using different SF$_6$ concentrations at different pressures. The oxide conversion layer was then analyzed using AES. Figures 4.8, 4.9, 4.10 and 4.11 show the results of these experiments. The atomic concentration of fluorine at the surface, 25 Å, 50 Å, 75 Å and 100 Å deep, is shown as a
function of concentration of SF$_6$. Adjusting the flowmeters for each gas sets the concentration ratio of SF$_6$ and N$_2$. The same experiments were conducted at four different pressures: 0.8 Torr, 1 Torr, 1.5 Torr and 3 Torr. The discs were treated for 30 seconds in all of the experiments.

![Graph showing Atomic Concentration of Fluorine (%)](image)

**Figure 4.7** Variation of the relative concentration of F atoms in SF$_6$ plasmas as a function of 2.45 GHz microwave input power at different pressures.

Figure 4.8 shows that the atomic concentration of fluorine is barely detectable beyond 50 Å at plasma pressure of 0.8 Torr. The surface of the treated disc shows a small upward trend in atomic concentration of fluorine as the concentration of SF$_6$ increases. Figure 4.9 shows the results obtained at a plasma pressure of 1 Torr. Here the upward trend is very noticeable at the surface of the solder disc. Figures 4.10 and 4.11 show the concentration of fluorine obtained at plasma pressures of 1.5 Torr and 3 Torr, respectively. The effect of a rich SF$_6$ gas mixture on the concentration of fluorine in the solder surface is even more evident with higher plasma pressures. The largest atomic concentration of fluorine in the solder at any depth is consistently found on the discs treated with a 100% SF$_6$ gas mixture. Although the presence of N$_2$ promotes the dissociation of SF$_6$, based on the experimental results, the presence of N$_2$ minimizes the atomic concentration of fluorine on the treated surface. N$_2$ is commonly used to perform ion-sputtering; however, it is very prone to ionize when subjected to high frequencies at low pressure. N$_2$ in the gas mixture may be sputtering away the oxyfluoride layer during treatment. Using pure SF$_6$ as the gas source to generate the plasma maximizes the oxide conversion rate in eutectic solder. All the subsequent experiments for process optimization are performed using only SF$_6$ as the gas source for the plasma-assisted oxide conversion process.
Figure 4.8  Atomic concentration of fluorine in solder surface as a function of gas mixture at 0.8 Torr.

Figure 4.9  Atomic concentration of fluorine in solder surface as a function of gas mixture at 1 Torr.
Figure 4.10  Atomic concentration of fluorine in solder surface as a function of gas mixture at 1.5 Torr.

Figure 4.11  Atomic concentration of fluorine in the solder surface as a function of gas mixture at 3 Torr.
4.3.3 Plasma Pressure and Gas Flow Rate

Plasma pressure and gas flow rate are not independent. In fact, the plasma pressure is controlled by the amount of gas flowing into the chamber. Eutectic solder discs were treated and analyzed to determine the pressure that maximizes the oxide conversion layer thickness. The discs were treated at four different pressures: 0.8 Torr, 1 Torr, 1.5 Torr, and 3 Torr using three different gas mixtures with SF$_6$ levels of 50 %, 90 %, and 100%. The effective flow rates for each gas are shown in Table 4.4. Figures 4.12, 4.13, and 4.14 show the atomic concentration of fluorine in the surface of the discs as a function of plasma pressure. As the plasma pressure increases, the concentration of fluorine within 75 Å from the disc's surface increases, as demonstrated in Figures 4.12 and 4.13. Using 100% SF$_6$ for the plasma causes the atomic concentration of fluorine to saturate at pressures higher than 1.5 Torr, as shown in Figure 4.14. The saturation pattern remains throughout the depth profile, with saturation values of fluorine atomic concentration in the solder of 35%, 33%, 20%, 15% and 10% at the surface, 25 Å, 50 Å, and 75 Å, respectively. The constant saturation pattern suggests that the treatment is uniformly applied over time. Pure SF$_6$ at 1.5 Torr or higher pressure produces the largest and most uniform oxide conversion layer for eutectic solder. Thus, all subsequent experiments are performed using pure SF$_6$ at 1.5 Torr of pressure.

Table 4.4 Effective flow rates for plasma-assisted oxide conversion process.

<table>
<thead>
<tr>
<th>Plasma Pressure (Torr)</th>
<th>Gas Mixture (SF$_6$ %)</th>
<th>SF$_6$ Flow Rate (cm$^3$/min)</th>
<th>N$_2$ Flow Rate (cm$^3$/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.8</td>
<td>50</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>50</td>
<td>9</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>100</td>
<td>0</td>
</tr>
<tr>
<td>1</td>
<td>50</td>
<td>17</td>
<td>17</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>120</td>
<td>14</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>476</td>
<td>0</td>
</tr>
<tr>
<td>1.5</td>
<td>50</td>
<td>35</td>
<td>35</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>316</td>
<td>26</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>985</td>
<td>0</td>
</tr>
<tr>
<td>3</td>
<td>50</td>
<td>115</td>
<td>115</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>992</td>
<td>84</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>1302</td>
<td>0</td>
</tr>
</tbody>
</table>
Figure 4.12  Atomic concentration of fluorine in solder surface as a function of plasma pressure using a gas mixture of 50% SF₆ and 50% N₂.

Figure 4.13  Atomic concentration of fluorine in solder surface as a function of plasma pressure using a gas mixture of 90% SF₆ and 10% N₂.
Figure 4.14 Atomic concentration of fluorine in solder surface as a function of plasma pressure using 100% SF$_6$.

4.3.4 Treatment Time Effects on Oxide Conversion Layer Thickness

Now that the optimum power, gas mixture and pressure are known, there is yet another process parameter to be studied: treatment time. Eutectic Sn/Pb solder discs are treated using different amounts of time to determine the effect of time on the oxide conversion layer process. Table 4.5 shows the process parameters used for this study. The total treatment times were 50 min, 25 min, 5 min and 2.5 min, using a power level of 20%. The effective plasma generation times were 10 min, 5 min, 1 min, and 0.5 min, accordingly. The solder discs are analyzed with the standard AES procedure.

Table 4.5 Process parameters for plasma-assisted oxide conversion process

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Power (Level)</td>
<td>1000 W (20%)</td>
</tr>
<tr>
<td>Frequency</td>
<td>2.45 GHz</td>
</tr>
<tr>
<td>Gas mixture</td>
<td>100 % SF$_6$</td>
</tr>
<tr>
<td>Plasma pressure</td>
<td>1.5 Torr</td>
</tr>
</tbody>
</table>

Figure 4.15 shows the AES depth profiles for the treated solder discs with the atomic concentration of fluorine in solder as a function of depth and the treatment time. There are two time treatment effects to be noticed: first, the average atomic concentration
of fluorine increases for all depths as treatment time increases; second, the maximum atomic concentration of fluorine is found farther from the surface as treatment time increases. The first effect can be easily noticed in Figure 4.15. The average concentration of fluorine is 21.2%, 19.8%, 9.16% and 8.58% for treatment times of 10, 5, 1, and 0.5 minutes respectively. The maximum concentration of fluorine is at the surface for a treatment time of 30 seconds, but as the treatment time lengthens, the maximum concentration of fluorine moves deeper into the solder. The maximum concentration of fluorine is at 15 Å, 50 Å, and 90 Å deep for treatment times of 1 minute, 5 minutes, and 10 minutes respectively. It is important to notice that for long treatment times, the concentration of fluorine at the surface is actually much smaller than the fluorine concentration in the inner layers. This suggests the migration of fluorine from the surface to the inner layers. Marshall found similar treatment time effects and developed a chemical model [41]. Figure 4.16 shows a schematic representation of Marshall’s model. SnO compounds can be found in the surface of the untreated solder, Figure 4.16 (a). Once the treatment is applied, the fluorine atoms react with tin atoms to form tin oxyfluorides. Figure 4.16 (b). As the treatment goes on, bridging tin-fluorine bonds develop, Figure 4.16 (c); and finally, the fluorine migrates into the interior of the solder. This chemical model seems to fit the plasma-assisted oxide conversion process. Using the AES results for the solder discs, the entire chemical model can be observed after 5 minutes of treatment and within 200 Å from the surface.

Figure 4.15 AES depth profiles showing the atomic concentration of fluorine in eutectic solder using different treatment times.
Figure 4.16  Schematic of chemical model for the Plasma-Assisted Oxide Conversion Process as a function of treatment time.

4.4 Preliminary Performance Evaluation for the Plasma-assisted Oxide Conversion Process

A qualitative evaluation was made to test the solderability performance of the plasma-assisted oxide conversion treatment. The evaluation consisted of reflowing treated and untreated eutectic solder discs. The solderability was measured qualitatively by comparing balling and joining of treated and untreated solder discs. All solder discs were treated using the optimum process parameters shown in Table 4.6.
Table 4.6  Process parameters for qualitative solderability study.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Power (Level)</td>
<td>1000 W (30%)</td>
</tr>
<tr>
<td>Frequency</td>
<td>2.45 GHz</td>
</tr>
<tr>
<td>Gas mixture</td>
<td>100 % SF₆</td>
</tr>
<tr>
<td>Plasma pressure</td>
<td>1.5 Torr</td>
</tr>
<tr>
<td>Total treatment time</td>
<td>6 Minutes</td>
</tr>
<tr>
<td>Effective treatment time</td>
<td>1.8 Minutes</td>
</tr>
</tbody>
</table>

4.4.1 Qualitative Solder Balling Tests

Solder balling consists of melting a flat disc of solder to see if it will reflow into a spherical ball. The surface tension of clean liquid solder is sufficient to overcome gravity and to force the liquid into the lowest free energy configuration, a sphere. Solder balling can measure the efficiency of the plasma-assisted oxide conversion process as a fluxless soldering process and can measure its ability to break up surface oxides. Figure 4.17 and 4.18 show optical pictures of a solder disc prior to reflow. The treated and untreated discs were reflowed in air. Reflow in air is performed by placing the discs on a pre-heated glass substrate at 200 °C. The time to melt is very short, so the discs can spheroidize before air re-oxidizes the surface. Figure 4.19 shows the untreated disc after reflow. The oxide layer prevents the disc from spheroidizing. Figure 4.20 shows the treated disc after reflow; they transformed into perfect spheres. This preliminary balling study shows that the plasma-assisted oxide conversion process consistently improves the reflowing characteristics of eutectic solder discs, resulting in a notable difference between treated and untreated solder.

Figure 4.17  Solder disc top view.
Figure 4.18  Solder disc lateral view.

Figure 4.19  Rapid reflow in air of an untreated solder disc.

Figure 4.20  Rapid reflow in air of a treated solder disc.

4.4.2 Qualitative Joining Tests of Solder Discs

Another qualitative evaluation of the oxide conversion process can be made by stacking two solder discs, then reflowing them to determine if joining occurs. Figure 4.21 shows the eutectic solder discs prior to reflow. These discs were also rapidly reflowed in air. Figure 4.22 shows the untreated discs after reflow. This picture demonstrates that joining does not occur due to surface oxidation. Figure 4.23 displays the treated discs after reflow. As Figure 4.23 indicates, there is complete joining between the two discs, resulting in the creation of just a single element. However, oxide formation during reflow
did not allow the joined discs to completely spheroidize. Nevertheless, a clear difference between the treated and untreated discs can be observed.

**Figure 4.21** Eutectic solder discs.

**Figure 4.22** Untreated solder discs after rapid reflow in air.

**Figure 4.23** Treated solder discs after rapid reflow in air.
CHAPTER 5  CONTINUOUS PRODUCTION OF FLUXLESS SOLDER SPHERES

5.1  Introduction

A plasma-assisted oxide conversion process for the production of fluxless solder spheres was developed using a microwave plasma generator. The goal of this research is the continuous production of fluxless solder spheres. The approach taken for the continuous production of solder spheres consists of the integration of the UDS process and the plasma-assisted oxide conversion process. Since this involves the integration of the droplet generator and the plasma generator, the solder droplets are treated as they are generated. This chapter discusses in detail the integration of these two processes for the generation of fluxless solder spheres. The chapter also presents an evaluation of the fluxless spheres.

5.2  The UDS Process and Plasma-assisted Oxide Conversion Process Integration For the Continuous Production of Fluxless Solder Spheres

5.2.1  Experimental Setup

There were several issues that had to be resolved to facilitate the integration of the droplet generator and the plasma generator. The integration of the oxide conversion process with the UDS process can be performed in two configurations: either the plasma is generated in the chamber where the solder droplets are produced, or the plasma is generated independently and the dissociation products are brought into the droplet generation chamber (downstream plasma treatment). The downstream plasma treatment presents a better option for process integration, since the UDS apparatus does not have to be modified for microwave shielding. Figure 5.1 shows a schematic representation of the new apparatus. The plasma generator depicted on the schematic is exactly the same as the one used for the optimization of the plasma-assisted oxide conversion process. The plasma generator is connected to the top of the UDS apparatus to allow the flow of the SF₆ dissociation products from the microwave plasma to the droplet generation chamber. The droplets can be generated using the procedure previously described in Chapter 2. A container filled with oil is placed at the bottom of the droplet generation chamber to solidify the droplets into spheres.
Figure 5.1 Schematic of the apparatus for the continuous production of fluxless solder spheres.

5.2.2 Experimental Procedure for the Continuous Production of Fluxless Solder Spheres

To produce eutectic, fluxless solder spheres, the droplet generator, containing approximately 0.5 Kg of 63Sn/37Pb solder, is mounted on the glass chamber, as shown in Figure 5.1. The plasma generator is connected to the UDS chamber through the top. The whole system is evacuated to 0.15 Torr and then it is filled with N₂ to a pressure of 13 kPa. The evacuation-refilling procedure is repeated once to make sure that a minimal amount of oxygen is left in the system. Then the system is evacuated again, but this time a constant flow of SF₆ is introduced to the system setting the pressure to 1.5 Torr. Once the pressure stabilizes at 1.5 Torr, the heater is set to 235°C and the solder is melted. In this process the droplets are generated in a vacuum (1.5 Torr). To force the molten solder through the orifice, the connection between the chamber and the crucible is shut off and the pressure in the crucible is increased until a stable stream of molten metal is produced using a vacuum regulator (Power Aire Inc., V800-50). Uniform droplets are generated by applying a constant disturbance to the jet of liquid metal as described in Chapter 2. Once uniform breakup is achieved, the microwave oven is turned on using a 100% power...
level, and the solder sphere size control system is started. Thirty seconds after the onset of plasma generation, the solder droplets are collected in a container filled with silicon oil. This 30 second period allows the plasma products to saturate the UDS chamber with an environment rich in atomic fluorine. When the desired amount of fluxless solder spheres is collected, the process is stopped by connecting the crucible to the chamber, stopping the control system, and turning off the heater and microwave oven. The solder spheres are drained and cleaned in baths of acetone and alcohol. A solder sphere target diameter of 300 µm is used for this study. Table 5.1 shows the process parameters for the continuous production of 300 µm fluxless solder spheres. Figure 5.2 shows a picture of the 300 µm fluxless solder spheres.

**Table 5.1** Process parameters for the continuous production of fluxless solder spheres.

<table>
<thead>
<tr>
<th>Process</th>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plasma-assisted Oxide Conversion Process</td>
<td>Power (level)</td>
<td>1000W(100 %)</td>
</tr>
<tr>
<td></td>
<td>Frequency</td>
<td>2.45 GHz</td>
</tr>
<tr>
<td></td>
<td>Gas Mixture</td>
<td>100 % SF₆</td>
</tr>
<tr>
<td>Plasma Oxide Conversion &amp; UDS Process</td>
<td>Plasma &amp; UDS Chamber Pressure</td>
<td>1.5 Torr</td>
</tr>
<tr>
<td>UDS Process</td>
<td>Orifice diameter d₀</td>
<td>178 µm</td>
</tr>
<tr>
<td></td>
<td>Vibration frequency f</td>
<td>4200 Hz</td>
</tr>
<tr>
<td></td>
<td>Initial jet velocity Vᵢ</td>
<td>4.6 m/s</td>
</tr>
<tr>
<td></td>
<td>Melt temperature</td>
<td>235 °C</td>
</tr>
<tr>
<td>UDS &amp; Control System</td>
<td>Target diameter D₁</td>
<td>300 µm</td>
</tr>
<tr>
<td>Closed-Loop Sphere Size Control System</td>
<td>Integral gain Kᵢ</td>
<td>15 (µm s)⁻¹</td>
</tr>
<tr>
<td></td>
<td>Sampling time Tₛampling</td>
<td>0.8 s</td>
</tr>
<tr>
<td></td>
<td>Camera constant δₑ camera</td>
<td>5 µm/pixel</td>
</tr>
<tr>
<td></td>
<td>Shrinkage constant δₛol</td>
<td>0.9848</td>
</tr>
<tr>
<td>Other</td>
<td>Treatment time (Approx.)</td>
<td>225 ms</td>
</tr>
<tr>
<td></td>
<td>Collection time</td>
<td>5 min.</td>
</tr>
<tr>
<td></td>
<td>Spheres collected (Approx.)</td>
<td>1,260,000</td>
</tr>
</tbody>
</table>

### 5.3 AES Evaluation of the Fluxless Solder Spheres

The surface of the 63Sn/37Pb fluxless spheres was analyzed using AES to determine the effects of the plasma-assisted oxide conversion treatment on the solder spheres. The AES parameters for the analysis are in Table 5.2. The sputtering rate of the ion beam is approximately 50 Å/minute of sputtering for eutectic solder. For each sphere, two surface scans and a depth profile are performed. The first scan is done over the surface of the eutectic solder sphere. Figure 5.3 shows the results of the surface scanning for a fluxless solder sphere produced using the procedure described in section 5.2.2. The AES results show the presence of the same elements in the treated solder discs: O, C, Sn, Pb, and F. The magnitude of the energy for Fluorine at 647 eV is smaller than that observed on the treated discs, indicating less fluorine.
Figure 5.2 300 μm 63Sn/37Pb fluxless solder spheres.

Table 5.2 Parameters used in AES for the analysis of fluxless solder spheres.

<table>
<thead>
<tr>
<th>Source</th>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Electron Gun</td>
<td>Beam Voltage</td>
<td>2 KV</td>
</tr>
<tr>
<td>(Analysis)</td>
<td>Filament Current</td>
<td>1.6 A</td>
</tr>
<tr>
<td>Ion Gun</td>
<td>Beam Current</td>
<td>12 nA</td>
</tr>
<tr>
<td>(Sputtering)</td>
<td>Raster Size</td>
<td>0.5 mm X 0.5 mm</td>
</tr>
</tbody>
</table>

Figure 5.3 Auger profile of the surface of a fluxless solder sphere.
A depth profile is performed after the surface scanning. Figure 5.4 shows the result of the depth profile for the same fluxless sphere. This figure shows the atomic concentration of each element (C, O, F, Sn, and Pb) as a function of depth. The small presence of fluorine is attributed to the short treatment time of only 250 milliseconds and to the loss of atomic fluorine species from the plasma generator to the UDS chamber. Even though the concentration of fluorine is small, suggesting an oxide conversion layer thickness of only 35 Å, the oxide conversion treatment has quite a significant effect on the oxide layer. Oxygen is present until a depth of 45 Å. This suggests that the oxygen is only present when fluorine is also present, indicating the formation of oxyfluorides. The oxyfluoride layer is very thin. Pure eutectic solder can be found at just 50 Å from the surface as Figure 5.5 shows. This figure shows an AES analysis of a fluxless solder sphere at 60 Å from the surface.

A thin oxyfluoride layer breaks faster and allows clean solder to reflow faster. However, it is believed that once the oxyfluoride layer breaks, it keeps preventing oxidation as the solder flows. Thus, a thicker oxyfluoride layer would cover a larger area, allowing slower reflow rates. Another important characteristic of the fluxless solder spheres is the thickness of the oxide layer. Figure 5.6 shows an AES depth profile for a fluxless solder sphere and a newly produced sphere without oxide conversion treatment. The plasma-assisted oxide conversion process produces solder spheres with an oxide layer thinner than that found in a newly produced regular solder sphere.

![Figure 5.4](image)

Figure 5.4 Auger depth profile of a 63Sn/37Pb fluxless solder sphere.
**Figure 5.5**  AES analysis of a fluxless solder sphere 60 Å from the surface.

**Figure 5.6**  Atomic concentration of oxygen for eutectic solder spheres and eutectic fluxless spheres.
5.4 Solderability Analysis of Fluxless Solder Spheres

Solderability is defined as the ability to achieve a clean, metallic surface on substrates during the dynamic heating process, so that good wetting of the molten solder on the substrate can be formed. The most important requirement of any attempt to measure the effectiveness of the oxide conversion process on the solder spheres is a quantitative solderability test that would allow comparison to the performance achieved using flux. Many tests are available to do this, most of them based upon some aspect of soldering processes [54-55]. Some of these tests include: the area of spread test, the edge dip test, the capillary test, the globule test, the rotary dip test and the surface tension balance test. This section discusses the results obtained using area of spread tests to evaluate the performance of the fluxless solder spheres.

5.4.1 Experimental Test Procedure

All experiments performed to evaluate the solderability performance of the fluxless solder spheres were done using the area of spread test. The area of spread test is a quantitative test that involves spread, when melted, of a solder sphere of known size. The solder sphere is placed in a substrate of a wetting material. The substrate can be preheated to perform rapid reflow or it can be heated after the solder sphere is placed on the substrate. The test is allowed to continue until the spreading action has stopped, indicating the combination of the maximum substrate-solder interaction with the minimum solder-atmosphere surface tension. Then, the area of spread is determined by measuring the area of the smallest circle that encloses the reflowed solder.

In this case, 300 μm eutectic solder spheres were produced with and without applying the plasma-assisted oxide conversion treatment. The non-treated solder spheres were produced by the exact same procedure described in Chapter 2. The fluxless solder spheres were produced using the same parameter and procedure defined in this chapter. Both types of solder spheres were produced at the same time to assure exposure to the same conditions. The substrates used for the area of spread test are copper plates 2.5mm thick, coated with a 0.5 μm gold layer. The gold coating was applied through electron beam deposition to assure uniformity. Two kinds of experiments were conducted according to the reflow rate and environment. The first method is rapid reflow in air, here, the substrate is preheated to 200 °C on a heating plate, then the solder sphere is placed on the substrate. In this process the solder sphere melts and spreads instantly, so the test is conducted in air. After the solder stops reflowing, the substrate is removed from the heating plate. The solder is let to solidify in air, and the spreading area is measured. The second method is slow reflow in N₂. Here, the solder sphere is placed on the substrate at room temperature. Then the substrate is placed inside a glass chamber. The glass chamber is sealed and vacuumed. After the vacuum level has reached approximately 0.05 Torr, the chamber is refilled with N₂ at 34 KPa. This procedure is repeated one time to assure the minimum content of oxygen in the chamber. Then, the substrate starts to be heated at a rate of approximately 20 °C/minute up to 200 °C. Once the solder stops spreading, the heater is turned off and the solder solidifies in N₂. After
the substrate has cooled down, it is taken out of the chamber and the area of spread is measured. These experiments were performed using non-treated solder spheres, fluxless spheres, non-treated spheres and flux, and fluxless spheres and flux respectively. The results of all experiments are discussed in detail in the following sections.

5.4.2 Area of Spread Test Using Flux

The evaluation of the solderability performance of the fluxless solder spheres is made relative to the performance of a regular solder sphere using flux. There are three types of fluxes commonly used in the microelectronics industry: rosin-based flux, water-soluble flux and no-clean flux. The rosin-based fluxes are regarded as the most effective and reliable in the microelectronics industry. Rosin is a natural product derived from the resinous secretions of trees, it is a glassy mix of abietic acid (COOH). Traditionally the rosin-based fluxes have been classified according to their level of activation. Type R is the weakest, containing only rosin chemistry without the presence of activators. Type RA is mildly activated and type RM is fully activated. For many years, rosin-based fluxes have been used successfully in conjunction with CFC solvents as the post-soldering cleaning agent. With the elimination of CFC solvents, two types of flux chemistries, water-soluble and no-clean, are being adopted and are gaining acceptance. Water-soluble fluxes are defined as the fluxes which soldering residues can be removed using water. The water-soluble fluxes possess the stability and reliability of an RMA flux. Water-soluble fluxes have been used in wave soldering and structural (nanoelectronics) soldering for years, yet their use in fine pitch surface mount technology (BGAs) is relatively new. Another type of flux is the no-clean flux, which does not need cleaning after soldering, a characteristic which makes this type of flux the least active of the three.

All of the three types of fluxes described above are actively used in the microelectronics industry. Thus, they represent good benchmarks against which to evaluate the performance of the fluxless solder spheres. The objective is to perform the same area of spread test using a regular solder sphere, and to compare the results of the test to those obtained using the fluxless solder spheres. Three types of fluxes were used to perform the area of spread experiments: rosin-based flux, water-soluble flux, and no-clean flux. Table 5.3 shows the specifications for each type of flux. Slow reflow in nitrogen of regular solder spheres was performed using the procedure previously described. The flux was applied by dipping the substrate in flux, while making sure the spheres were completely wetted by the flux. Figure 5.7 shows a photograph of a reflowed sphere; in this case flux was not applied. A very small amount of spreading occurred due to the reducing atmosphere that the nitrogen provides. Figures 5.8, 5.9, and 5.10 show photographs of reflowed regular spheres using rosin-based flux, water-soluble, and no-clean fluxes respectively. From the photographs, it is difficult to derive a conclusion as to which kind of flux provides the largest area of spread. However, distinctive differences were found after measuring the area of spread, as presented in Figure 5.11.
Table 5.3 Description of fluxes used in area of spread test.

<table>
<thead>
<tr>
<th>Flux Type</th>
<th>ANSI/J-STD-004 Flux Type Symbol</th>
<th>Solids Content</th>
<th>Flux Activity Level</th>
<th>Halide %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rosin-based Flux</td>
<td>F</td>
<td>25 %</td>
<td>High</td>
<td>&gt;2 %</td>
</tr>
<tr>
<td>(AIM Inc., R.A.G.E.-25)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Water-soluble Flux</td>
<td>Q</td>
<td>24 %</td>
<td>Moderate</td>
<td>0.5-2 %</td>
</tr>
<tr>
<td>(AIM Inc., WS-715)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>No-clean Flux</td>
<td>M</td>
<td>3.54 %</td>
<td>Low</td>
<td>0</td>
</tr>
<tr>
<td>(AIM Inc., NC 264-5)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Figure 5.7 Slow reflowed solder sphere without flux.

Figure 5.8 Slow reflowed sphere using rosin-based flux.

Figure 5.9 Slow reflowed solder sphere using water-soluble flux.

Figure 5.10 Slow reflowed solder sphere using no-clean flux.

Three sets of experiments were conducted for each kind of flux to obtain the solderability performance. For each experiment, five reflowed solder spheres were measured, the spreading area was determined by completely enclosing the reflowed
solder with the smallest possible circle. The area of spreading is equal to the area of that circle. Figure 5.11 shows the normalized solderability performance as a function of flux type. The solderability performance represents the area of spread after reflow. The solderability performance was normalized by dividing the area of spread for each condition by the largest area obtained. In this case, the best performance is obtained using rosin-based flux, thus the normalized performance for rosin-based flux is one. The other types of fluxes, as well as the fluxless solder spheres, were evaluated in terms of the performance obtained using rosin-based flux. Figure 5.11 is consistent by flux type. The best solderability performance was from the most activated flux (rosin-based), followed by water-soluble flux with a 0.85 performance, and no-clean flux with 0.68 normalized solderability. These results were used to evaluate the performance of the fluxless solder spheres and to compare the plasma-assisted oxide conversion treatment with a rosin-based flux, a water-soluble flux, or a no-clean flux.

5.4.3 Area of Spread Test for the Fluxless Solder Spheres

Now that the reference for solderability performance has been established, the fluxless solder spheres can be evaluated. The objective of the production of fluxless solder spheres is to eliminate the use of flux in BGAs. Comparing the performance of the fluxless solder spheres to the performance of the most common fluxes used in microelectronics soldering is the best way to prove the ability of the fluxless solder spheres to replace flux.

![Graph showing normalized solderability performance for different flux types]

**Figure 5.11** Normalized solderability performance for eutectic solder spheres using different types of fluxes.
The evaluation of fluxless solder spheres is performed using the two types of reflow conditions previously described. Figure 5.12 shows a picture of a rapid reflowed fluxless solder sphere. In this case, the fluxless sphere stops spreading within 5 seconds after reaching a considerably larger area than that achieved in slow reflow in nitrogen, depicted in Figure 5.13. The sphere reflowed in nitrogen shows a cleaner surface. This is due to the oxidation that occurs at high temperatures in air.

![Figure 5.12 Rapid reflow in air of a fluxless solder sphere.](image1)

![Figure 5.13 Slow reflow in N₂ of a fluxless solder sphere.](image2)

Figure 5.14 shows the normalized solderability performance for the fluxless solder sphere rapidly reflowed in air as a function of time after treatment. The solderability performance for the fluxless solder sphere is better than the performance using rosin-based flux for the first three days after treatment. Nevertheless, there is a continuous, slowly declining trend in performance for the first 6 days after treatment. From the 7th day after treatment the slope of the declining trend changes drastically and the solderability performance declines rapidly. This declining trend in performance indicates the presence of a degradation mechanism [56]. The thickness of the oxyfluoride layer decreases as a function of time. The experimental data shown in Figure 5.14 suggests that the thickness of the oxide conversion layer decreases uniformly until the 6th day, while the change in slope on the 7th day indicates that re-oxidation is occurring. Even though the oxyfluoride layer degrades with time, for the first 6 days after reflow the fluxless solder spheres can provide a solderability performance equally effective to that obtained using rosin-based flux.
Figure 5.14  Normalized solderability performance for fluxless solder spheres using the rapid reflow procedure.

Figure 5.15 shows the normalized solderability performance for a fluxless solder sphere, slowly reflowed in nitrogen, as a function of time after production. The performance level for the fluxless solder spheres is comparable to that obtained using water-soluble flux for the first four days after treatment. In this case, the change in slope occurs on the 5th day. The solderability performance results for slow reflow also suggest degradation of the conversion layer thickness. The change in slope for both reflow processes occurs around the 5th or 6th day, indicating that the degradation process is occurring at the same rate. A reasonable conclusion is that the degradation process occurs while the fluxless solder spheres are stored in air. The solderability performance level measured using slow reflow in nitrogen is not as good as that obtained using rapid reflow. The rate of heating is a key variable for all soldering operations, especially for the fluxless processes. When the conversion layer breaks up, exposing clean solder, there is nothing to prevent re-oxidation. Thus, the difference in performance between slow and rapid reflow is not the degradation of the conversion layer, but the reflow rate. For the rapid reflow, the fluxless solder sphere spreads in less than 5 seconds. Thus re-oxidation does not play an important role in spread size. But for the slow reflow, the fluxless sphere takes approximately 10 minutes to start spreading. Even though the process is performed in a nitrogen environment, oxidation occurs decreasing the solderability performance level. For rapid reflow as well as for slow reflow, the best solderability performance is obtained for the first 6 days after treatment with the fluxless solder spheres stored in air.
Essentially, the fluxless solder spheres have a solderability performance level as good as rosin-based flux for rapid reflow in air, or as good as water-soluble flux for slow reflow in nitrogen.

![Normalized solderability performance for fluxless solder spheres using the slow reflow procedure.](image)

**Figure 5.15** Normalized solderability performance for fluxless solder spheres using the slow reflow procedure.

### 5.4.4 Area of Spread Test for the Fluxless Solder Spheres Using Flux

There are two major objectives for the production of fluxless solder spheres, one is to reduce the number of process steps for the BGA assembly by eliminating the use of flux, and the other is to eliminate the use of rosin-based fluxes. For some BGA assembly processes flux holds the solder spheres in place until they are reflowed. In this case, the elimination of flux is not possible without making drastic changes to the assembly process. However, the fluxless solder spheres can enhance the solderability performance of environmentally responsible fluxes like water-soluble or no-clean fluxes, and eliminate the use of rosin-based fluxes in any assembly process. Figures 5.16 and 5.17 show the normalized solderability performance for the fluxless solder spheres as a function of time after treatment using water-soluble flux and no-clean flux respectively. The solderability performance for the fluxless solder spheres using water-soluble flux was better than the
performance obtained, using rosin-based flux for the first 7 days after treatment. After the 7th day, a change in the performance slope occurred, giving place to a decrease in the performance until the conversion layer had no effect on the performance at the 10th day. The change in slope observed around the 7th day is consistent with the conversion layer degradation theory stated in previous experiments.

Figure 5.16  Normalized solderability performance for fluxless solder spheres using water-soluble flux in the slow reflow procedure.

Figure 5.17 shows the normalized solderability performance as a function of treatment time for the fluxless solder spheres using no-clean flux. In this case, the oxide conversion process enhances the solderability performance using no-clean flux to the performance of using water-soluble flux for the 7 days following production. Here again, a change in slope occurs after the 7th day when the degradation causes the performance to decrease, until the no-clean flux performance level is reached. Using fluxless solder spheres enhances the solderability performance for the water-soluble flux and the no-clean flux to that of the rosin-based flux and water-soluble flux respectively.
Figure 5.17  Normalized solderability performance for fluxless solder spheres using no-clean flux in the slow reflow procedure.

5.5 Results and Discussion

The solderability performance for the fluxless solder spheres is evaluated relative to the solderability performance using rosin-based flux. Figure 5.18 shows the average solderability performance for the fluxless solder spheres. The upper performance limit is defined by the performance using rapid reflow in air where re-oxidation does not occur during spreading. The lower performance limit is determined by slow reflow in nitrogen where oxidation occurs when spreading. The solderability performance for the fluxless solder spheres is as effective as using rosin-based or water-soluble flux for the first 3 days after treatment with spheres stored in air. Fluxless solder spheres are as effective as the performance of water-soluble and no-clean flux from the 4th day to the 6th. After the 6th day, a drastic change in the solderability performance occurs due to the degradation of the conversion layer. The change in slope may indicate the point at which the conversion layer does not cover the whole surface and the spheres start to re-oxidize. The degradation in the conversion layer occurs at the same rate for both types of reflow, as the slopes for the upper and lower solderability limit are very similar. This suggests that the degradation process occurs before the fluxless spheres are reflowed. The conversion
layer degrades while the spheres are stored in air. The fluxless solder spheres can also be used to enhance the performance of low activated fluxes such as water-soluble or no-clean fluxes. The solderability performance analysis showed that using fluxless solder spheres can increase the performance level obtained using water-soluble flux to that obtained using rosin-based flux, and can increase performance level obtained using no-clean flux to that obtained using water-soluble flux.

![Graph showing solderability performance over time](image)

**Figure 5.18** Normalized solderability average performance range for fluxless solder spheres.

The experimental evaluation of the solderability performance of the fluxless solder spheres indicates that the plasma-assisted oxide conversion process is effective in producing fluxless solder spheres. Furthermore, these fluxless spheres can eliminate the use of any type of flux, depending on the storage time after production. Thus, a process that consists in the integration of the UDS process and a plasma generator can be effectively used for the production of highly precise fluxless solder spheres for BGA chip interconnection technology. The goal of a faster, more reliable, and environmentally responsible BGA assembly process has been met.
CHAPTER 6  SUMMARY

A process that allows the production of fluxless solder spheres for BGA electronics packaging was developed, implemented and evaluated. This process involved the implementation of an on-line droplet size control system to the UDS process, and the development of a plasma-assisted oxide conversion treatment that can be applied to the solder spheres produced by the UDS process. The droplet size control system is based on a digital image analysis: an image of a droplet is used to determine the final size of the solder sphere. The break up frequency is used to place the mean sphere size on target. Experimental results of the droplet size control system demonstrated that the UDS process can now produce spheres for BGA chip interconnection technology. The control system enables the UDS process to produce spheres with a size variation smaller than ± 2.5% of the target size. Even more importantly, the control system allows the monitoring and control of the mean sphere size. The mean sphere size is exactly placed on the target size using an integral controller, which allows no steady state error, a short settling time and a well-damped response. This control system is sensitive to any noise or disturbance on the expelling pressure. The control system was developed specifically for the UDS process. However, it can be adapted to any other kind of droplet generation system based on the uniform breakup of a liquid jet.

Furthermore, a plasma-assisted oxide conversion process was developed to produce fluxless solder spheres. This process converts solder oxides into oxyfluorides by exposing the solder to a highly activated atomic fluorine, generated by the decomposition of SF₆ in plasma. The oxyfluoride layer on the solder spheres breaks when the spheres are reflowed, exposing clean solder, which has good wetting and joining properties. The plasma-assisted oxide conversion process was integrated with the UDS process to allow the continuous production of fluxless solder spheres. The solderability performance for the fluxless solder spheres was evaluated relative to the solderability performance using rosin-based flux. The solderability performance for the fluxless solder spheres proved to be as effective as using rosin-based flux or water-soluble flux for the first 3 days after treatment having stored the spheres in air, and as effective as the performance of water soluble and no-clean flux from the 4th to the 6th day. After the 6th day, a drastic change in the solderability performance occurs due to the degradation of the oxyfluoride layer. The fluxless solder spheres can also be used to enhance the performance of low activated fluxes such as water-soluble or no-clean fluxes. The solderability performance analysis showed that using fluxless solder spheres can increase the performance level obtained using water soluble flux to that obtained using rosin-based flux, and the performance level obtained using no-clean flux to that obtained using water soluble flux.

The experimental evaluation of the solderability performance of the fluxless solder spheres indicates that the plasma-assisted oxide conversion process is effective in producing fluxless spheres. These fluxless spheres can eliminate the use of any type of flux, depending on the storage time after production. Thus, a process that consists in the integration of the UDS process with the on-line droplet size control system, and the plasma-assisted oxide conversion process, can be effectively used for the production of
highly precise fluxless solder spheres for BGA chip interconnection technology. This is significant because it ultimately allows for a faster, more reliable and environmentally responsible BGA assembly process.
BIBLIOGRAPHY
