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Electroless nickel bumping of aluminium bondpads. Part 2 - electroless nickel plating

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Electroless Nickel Bumping of Aluminum Bondpads—Part II: Electroless Nickel Plating

David A. Hutt, Changqing Liu, Paul P. Conway, David C. Whalley, and Samjid H. Mannan

Abstract—Electroless nickel has been used for many decades to provide a hard, corrosion resistant surface finish to engineering components. In recent years its application has been extended to the electronics industry for the production of solderable surfaces on printed circuit boards, which utilize a further thin gold coating to prevent oxidation of the nickel surface. The recent interest in the use of flip-chip technology in electronics manufacture has required the development of low cost methods for solder bumping of semiconductor wafers. The electroless nickel process has been considered as a suitable candidate for the deposition of a solderable under bump metallization (UBM) layer onto the Al bondpads. However, the extension of existing electroless nickel plating processes to this new application requires greater understanding of the technique. In particular, the coating of the small isolated bondpads on the wafer surface introduces difficulties that make the use of many commercially available solutions impossible. This paper reports the results of a number of experiments carried out to investigate the electroless nickel bumping of Al bondpads and highlights the issues that need to be considered when selecting materials and techniques.

Index Terms—Electroless nickel, flip-chip, under bump metallization (UBM), wafer bumping, zincate.

I. INTRODUCTION

THE DEMAND for high performance and densely packaged electronic devices is fueling the uptake of flip-chip technology across the electronics industry and has led to the development of a range of interconnection technologies including the use of conducting adhesives and solder [1], [2]. At present, solder based interconnect offers an attractive option for low-cost assembly as the process route merges well with the existing ones for surface mount technology. However, the low cost implementation of this technology into consumer products can only take place if competitively priced solder bumped die are readily available and this demand has led to the development of a range of solder bumping techniques [1], [3]. One of the most popular and lowest cost technologies currently available is electroless nickel bumping followed by solder paste printing. This route has been taken up by a number of companies (e.g., [4], [5]) and has prompted a number of studies related to both the processing

techniques required and the reliability of the final devices [6], [7].

Electroless nickel has been used for many decades as a method for providing a surface finish on engineering components due to its corrosion protection properties and hardness [8], [9]. However, it has also found increasing use in the electronics industry as part of the electroless nickel–gold finish applied to PCBs for solderability preservation. The recent introduction of electroless nickel plating, as part of the wafer bumping process, to provide an under bump metallization on the Al bondpads prior to solder deposition, has led to a number of challenges relating to the activation and plating of the relatively small features on semiconductor devices compared to the larger features of engineering components and PCBs.

This paper reports a number of experimental studies of electroless nickel bumping that have been conducted as part of a wider program of research on low cost flip-chip assembly. The activation of the Al bondpads prior to electroless nickel plating requires careful control and characterization and forms the basis of another publication [10]. The present work only concerns the issues relating to reliable and reproducible electroless nickel bumping.

II. EXPERIMENTAL PROCEDURE

A. Wafers

A number of devices and chip types (both test structures and real devices) have been investigated as part of this work. The majority of the results presented in this work were obtained on wafers composed of daisy chain test structures comprised of AlCu(1%) tracks and bondpads at pitches of 225 and 300 μm (wafer types A and B). These bondpads were 3 μm thick and octagonal in shape with a circular passivation opening of 75 μm diameter. In addition to the flip-chip dimension pads, wafer type B also had a range of larger pads designed for BGA style interconnection and with passivation openings of 580 μm diameter.

B. Bondpad Activation

The aluminum bondpads of semiconductor devices require a number of pre-treatment steps to enable the deposition of electroless nickel. These steps have been described in detail elsewhere [10], but briefly, consist of exposing the sample to a series of chemical baths. To begin with, a 5% sodium hydroxide solution and 50% nitric acid were used to clean the Al surface and thin the oxide layer. Following this, the surface was activated by exposure to an alkaline zincate bath that deposited zinc clusters (a single zincate treatment). Electroless nickel deposition could be activated at this stage, but a smoother surface

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TABLE I
COMPOSITION AND OPERATING CONDITIONS OF ELECTROLESS NICKEL
BATHS USED

	Individual formulation	Commercial solution
Nickel Sulphate Heptahydrate	28.7 g l ⁻¹	Not known
Sodium Hypophosphite Monohydrate	27 g l ⁻¹	" "
Ammonium Acetate	20 g l ⁻¹	" "
Acetic Acid	15.5 cm ³ l ⁻¹	" "
tri - Sodium Citrate Dihydrate	0 - 3.5 g l ⁻¹	" "
di- Sodium Succinate Hexahydrate	2 - 9 g l ⁻¹	" "
Glycine	0 - 3 g l ⁻¹	" "
Thiourea	0 - 0.7 mg l ⁻¹	" "
Lead Acetate Trihydrate	0 - 0.8 mg l ⁻¹	" "
pH	4.6 - 4.8	4.6 - 4.8
Temperature	85°C	85°C
Coating Composition	3 - 5 wt% P	6 - 8 wt% P

finish and stronger adhesion of the deposit could be obtained by using a double zincate treatment. This involved etching away the first zinc deposit layer in nitric acid and then reapplying the zincate treatment which was then thinner and finer grained than the single zincate process.

C. Electroless Nickel Deposition

For the plating of electroless nickel onto the zincate activated bondpads a number of different solutions were evaluated, including a commercially available system used for plating PCB pads and others prepared from individual components that therefore allowed the influence of each part to be investigated. Table I details the compositions of the baths used. In general, acidic hypophosphite baths were used, operating at a pH of 4.6–4.8, producing nickel deposits with phosphorus incorporated into them. Plating was carried out in 3.2l of solution into which samples were suspended vertically. The bath temperature was controlled at 85 °C using a glass coated heating element and was stirred using a magnetic stirrer bar.

III. RESULTS

Good electroless nickel bumps could be obtained using a number of different solutions. Fig. 1 shows SEM images of bondpads on wafer A bumped using both commercial and individually simulated baths. As the electroless nickel process is isotropic, the bumps grew evenly in all directions, and when above the level of the passivation, spread over it, sealing the bondpad. This can be seen clearly in the cross section of Fig. 1(c). The difference in bump height across a 4 in diameter wafer was found to be less than 1 μm for bumps grown to a thickness of 30 μm . The P content of the bumps is listed in Table I together with the bath composition. Generally, the formulations used produced approximately 3–5 wt% P compared to 6–8 wt% P from the commercial bath. In agreement with other groups it was found that a correctly operated process gives smooth, evenly grown bumps, with no extra deposition onto the surface of the passivation material or backface of the die.

A. Identification of Plating Defects

Initially, the commercial plating bath appeared to offer the best process with a well characterized and understood chemistry. However, after operation of the bath for a relatively short

period of time, a number of defects began to occur in the plating process. These defects were not isolated, but occurred across the entire device. Three major types of defects were observed and these are shown in Fig. 2. The major problems consisted of conical bumps, where the spread of the bump appeared to be hindered and similarly, directional bumps where the flow of the stirred solution over the pad surface appeared to be preventing the spread of the bump on the up-stream side. These defects were similar in appearance, and seemed to be related to the action of the electroless nickel bath. The other form of defect involved the influence of the bondpad interconnection. Small pads that were connected to the large bondpads of wafer type B were plated very well, while isolated bondpads had either conical/directional bumps or, in severe cases, only nodular growth of electroless nickel.

The source of these defects was initially unclear and, in particular, they were found to be unaffected by changes in pre-treatments to the surface and bath agitation methods, indicating that surface activation and gas bubble formation were not inducing them. It was noted that using a bath made from individual components (Table I), that these defects did not occur, although the smoothness of the deposit was not as good.

Plating of a similar nature to these defects was observed by van der Putten and de Bakker [11], [12] and very recently by Chen *et al.* [13] during their investigations of the effects of stabilizers in electroless nickel plating. In order to study the factors affecting these defects, measured quantities of stabilizer were added to a standard electroless nickel solution and the changes in the bump quality assessed. Two stabilizers were investigated: thiourea and lead acetate, which are commonly used for electroless nickel plating. The stabilizers are present in electroless nickel baths in mg l⁻¹ concentrations, but have a very important role. First, they prevent the solution decomposing spontaneously, by adsorbing onto the surfaces of particles formed in the bulk of the solution and poisoning the further deposition of electroless nickel on them and, similarly, they prevent the deposition of nickel onto other surfaces which are not to be plated such as the plating tank walls.

B. Lead Acetate Stabilization

Fig. 3 shows the effect on the electroless nickel bump morphology of the addition of lead acetate to the standard bath. It should be noted that the concentrations of lead acetate indicated are only a guide to the total amount added to the solution, as during plating, the Pb ions are incorporated into the deposit in small quantities and subsequently the solution concentration is continually reduced [14]. Fig. 3(a) shows the bumps formed without the presence of stabilizer. As mentioned above, this solution produced NiP bumps without defects, but the surface exhibited a very grainy appearance. In addition, extraneous deposition onto defects in the passivation layer of the die and back and sides of the wafer also occurred. Addition of just 0.44 mg l⁻¹ of lead acetate to the bath produced NiP bumps as displayed in Fig. 3(b). These were much smoother and indicate the advantage of adding stabilizers to the bath. Furthermore, extra deposition of Ni onto the chip passivation was prevented. On addition of more lead acetate to the bath, the conical bumps of Fig. 3(c)

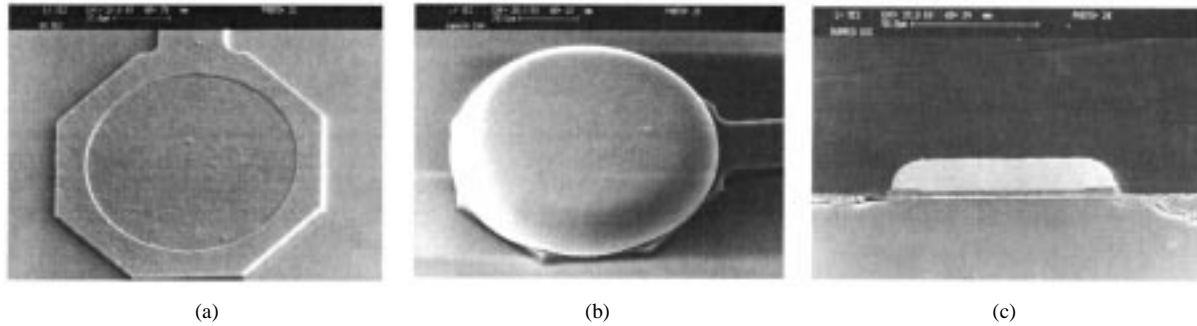


Fig. 1. SEM images of (a) bare Al bondpad before bumping, (b) bondpad after electroless nickel bumping, and (c) cross section of electroless nickel bumped bondpad.

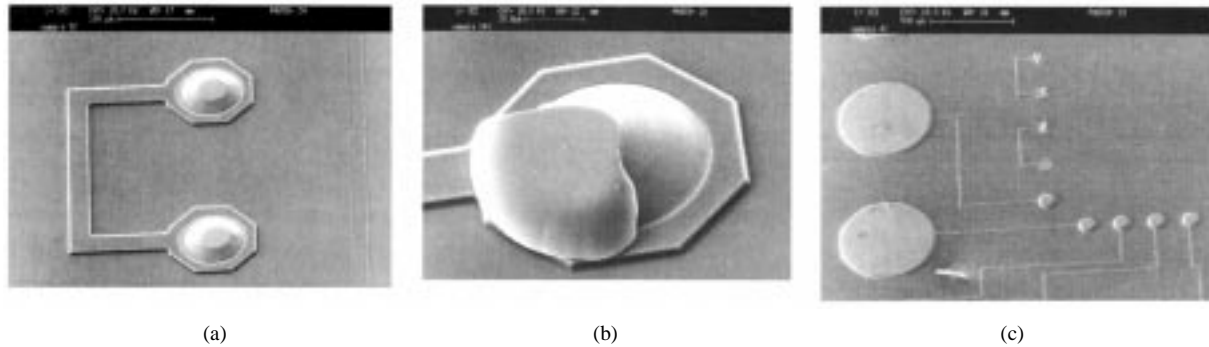


Fig. 2. SEM images of defect structures obtained when bumping wafers using a commercial electroless nickel plating bath: (a) conical bumps, (b) directional bumps, and (c) connection effects.

were obtained and these were very similar to the defects observed with the commercial solution.

Solution movement in the bath during plating caused by stirring or convection currents also introduced directional effects into the bumps, as indicated in the images of Fig. 3(d) and (e). These images show that solution flow over the bump surface and the concentration of stabilizer could cause directional effects. This indicates that the arrival and diffusion of the stabilizer at the surface has a strong effect on the level of stabilizer that the solution can tolerate before defects begin to occur. Finally, Fig. 3(f) shows three bondpads from a die that had both small and large bondpads. The two pads with conical bumps were isolated, while the well plated bondpad was connected to a large bondpad (out of picture). This again displayed strong similarities with the defects observed with the commercial bath.

C. Thiourea Stabilizer

By contrast to the defects observed during the addition of lead acetate to the solution, thiourea had a much reduced impact on the shape of the NiP deposits. In Fig. 4, the effect of adding thiourea to the standard bath is displayed. Addition of small quantities of thiourea clearly improved the quality of the deposit, giving results similar to those for lead acetate [Fig. 4(b)]. However, addition of larger amounts did cause some directional effects when stirred and, more noticeably, introduced pores and pockmarks into the pad surfaces [Fig. 4(c) and (d)].

For both stabilizers, once the concentration had become too high in the bath, all subsequent plating operations produced defective bumps. However, depletion of the stabilizer level could be achieved by plating a large surface area material in the bath,

after which good quality bumps could again be obtained. Similarly, adding more stabilizer resulted in the return of the defects to the plating process.

D. Deposit Morphology

As described above, the level of stabilizer in the bath had a significant influence on the morphology of the final NiP deposit. However, a number of other factors were also found to strongly affect the morphology. The first of these was the zincate treatment. As described elsewhere [10], the zincate layer helps to initiate the electroless nickel plating process on Al by exchange of the zinc atoms with the Ni ions in solution, thereby creating a Ni seed layer that can continue the autocatalytic deposition of NiP. The nature of the zincate layer not only affects the adhesion of the deposit, but also influences the morphology by controlling the number of nucleation sites and their distribution. In these studies it was found that a double zincate treatment produced a smoother NiP deposit which was attributed to the thinner, finer grained and more uniform zincate layer produced by the double zincate compared to a single zincate treatment.

In addition to the zincate treatment, the original bondpad structure could also strongly influence the NiP deposit. The effect of probe marks made by testing of the Al bondpads prior to deposition is illustrated in Fig. 5. These results were obtained from a commercially available memory device and show clearly that for the as-received die, there were substantial defects induced in the thin Al bondpad by test probing. Of major concern was the thinning of the pad material beneath these probe marks, such that during pre-treatment of the die prior to electroless nickel deposition the alkaline etches and

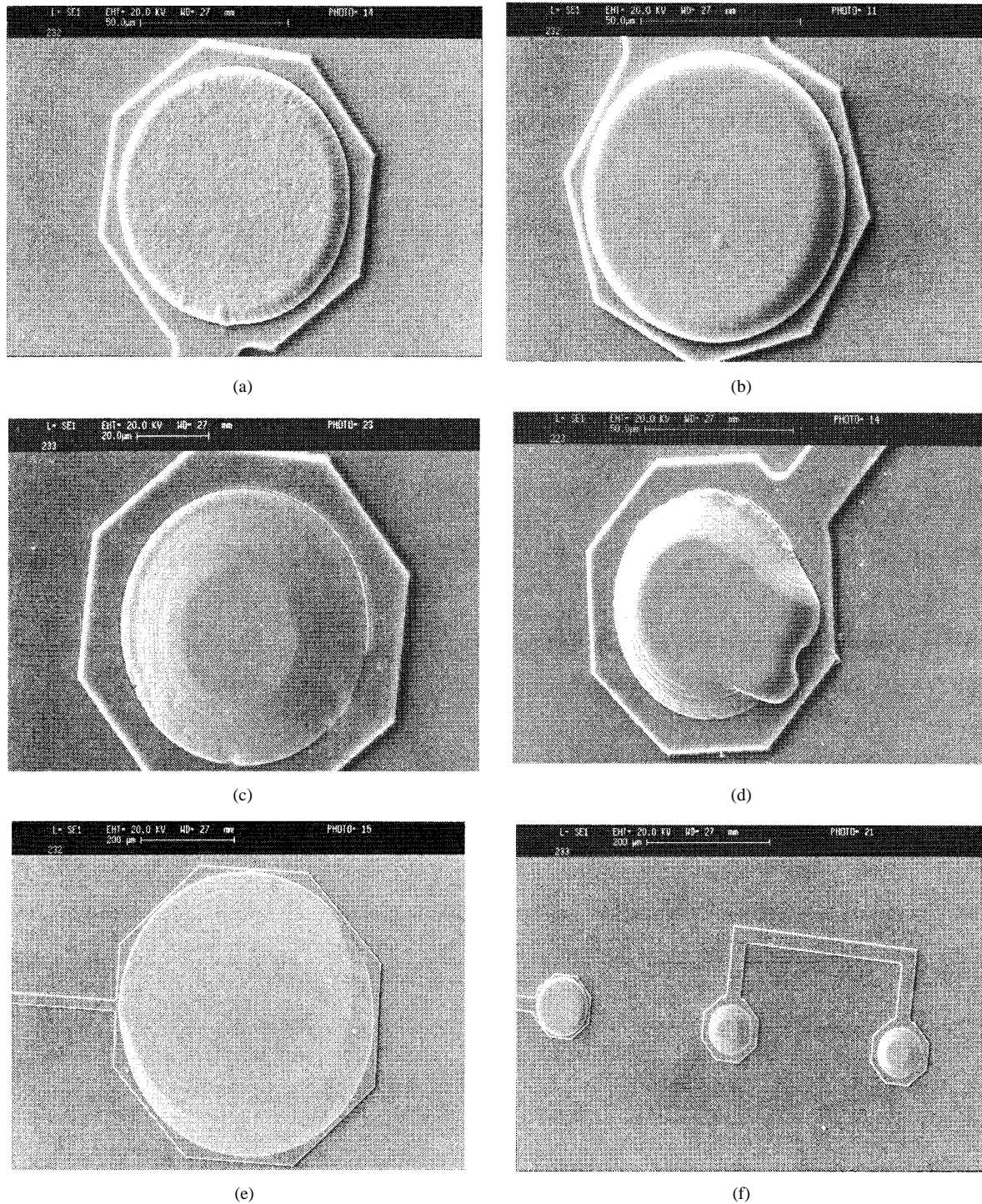


Fig. 3. Effect on the electroless nickel bump shape following the addition of lead acetate stabilizer to the electroless nickel bath: (a) no stabilizer, (b) 0.44 mg l^{-1} , no stirring, (c) wafer type B, 0.74 mg l^{-1} , no stirring, (d) wafer type B, 0.74 mg l^{-1} effect of convection in unstirred solution, (e) large bondpad on wafer type B, 0.74 mg l^{-1} , solution stirred, and (f) wafer type B, 0.74 mg l^{-1} , no stirring, connection effect.

zincate treatments could cause the bottom of the probe mark to be etched through to the underlying metallization [Fig. 5(c)]. Where this happened it led to uneven NiP bumping of the bondpad [Fig. 5(b)].

E. Solderability of Deposits

In general an immersion gold coating, or “gold flash” around 50 to 100 nm thick is applied over the electroless nickel surface

immediately after plating to preserve the solderability of the nickel surface. In these trials, a gold flash was not used, as solder bumping using solder paste printing was carried out within a few days of the application of the UBM. It was found that with the solder paste used, the flux activity was sufficient to enable complete reflow of the solder onto the UBM whenever reflow was conducted in a nitrogen atmosphere. This has a number of advantages for the overall process route: first, the immersion gold process is one of the most expensive steps of the bumping

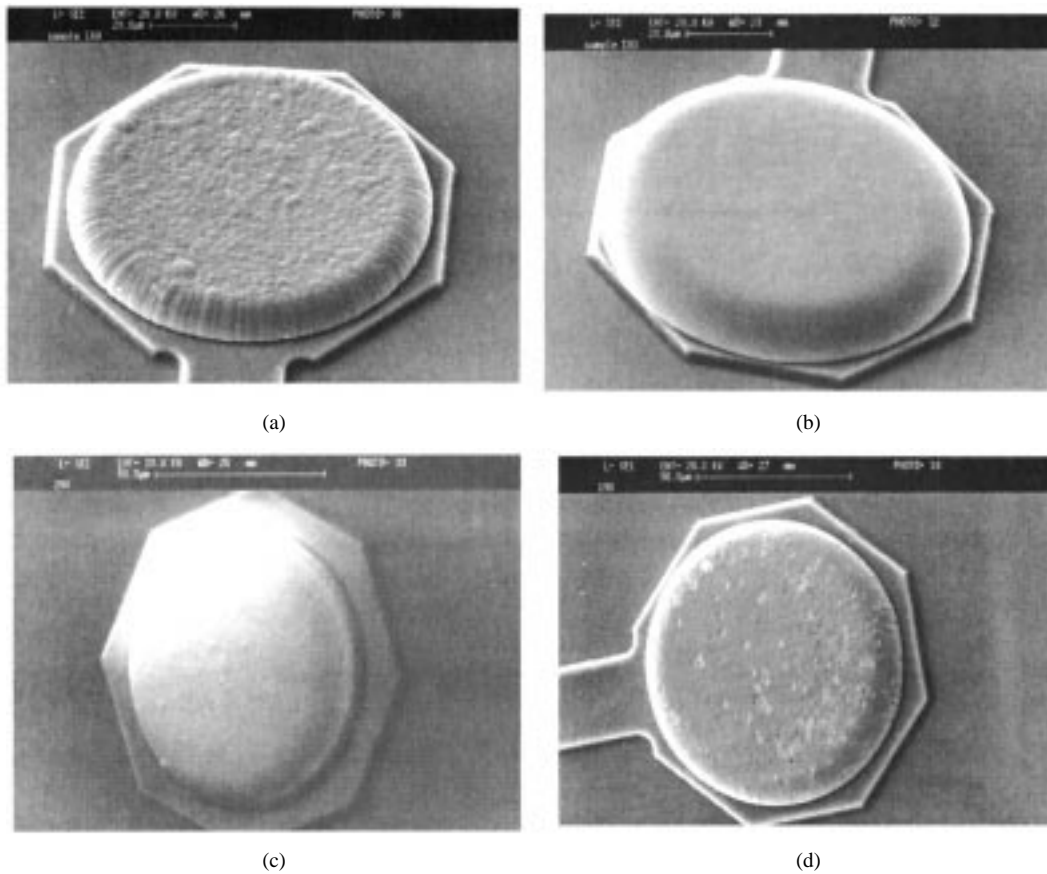


Fig. 4. Effect of thiourea stabilizer on the morphology of the electroless nickel bumps as a function of concentration in the electroless nickel bath: (a) 0.16 mg l^{-1} , (b) 0.64 mg l^{-1} , (c) $> 0.7 \text{ mg l}^{-1}$, stirred, and (d) pockmarked sample.

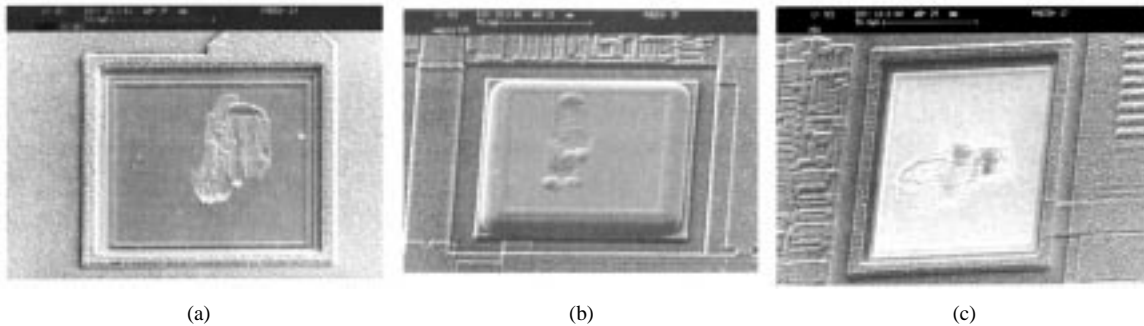


Fig. 5. Effect of bondpad probe marks on the morphology of the electroless nickel bumps: (a) as received bondpad, (b) after electroless nickel bumping, and (c) after zincate treatment.

process and typically involves solutions based on cyanide with their associated health risks and disposal issues (although a recent study [15] has demonstrated the use of cyanide free plating baths). Secondly, the absence of the gold coating from the electroless nickel bumps may help to enhance the reliability of the final flip-chip joints due to the absence of any brittle AuSn_4 intermetallics. Generally, it is accepted that a concentration of gold in the final solder joint of less than 3 wt% will not lead to the formation of significant AuSn intermetallics. While the amount of gold deposited in the flash is very small, the limited amount of solder in the flip-chip joint could make this quantity significant. However, of more concern are recent publications [16]–[19] investigating the reliability of BGA devices attached to electroless nickel/gold pads. Some of these have shown that even a small

amount of gold can segregate near the Ni pad, creating high local concentrations of AuSn_4 intermetallics potentially resulting in premature device failure. Considering the lower pad surface area to volume ratio of solder present in a BGA joint compared to a flip-chip joint this issue should not be disregarded.

IV. DISCUSSION

The results reported above for the action of stabilizers on the electroless nickel bump formation support the work of van der Putten and de Bakker [11], [12] who found that lead acetate produced more shape defects than thiourea. However, in the present study it was found that a much smaller level of stabilizer was required to induce the same level of defects in the bump shape. In

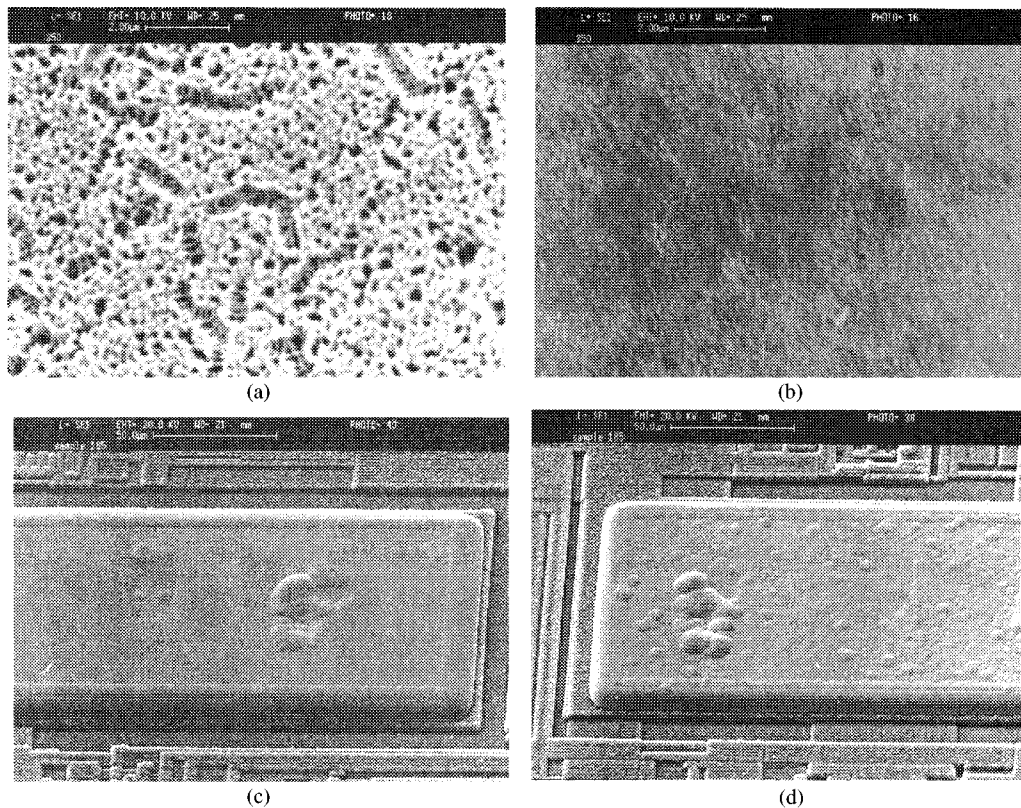


Fig. 6. Effect of internal connections on the activation and bumping process. a and c) normal bondpad following: (a) zincate treatment and (c) electroless nickel bumping, (b) and (d) ground bondpad connected to Si substrate after (b) zincate treatment and (d) electroless nickel bumping.

their work, they describe the action of the stabilizer as poisoning the plating process by altering the electrochemical potential of the surface after adsorption, thereby restricting the reduction of Ni ions to Ni metal at the surface. The small isolated bondpads are most affected by the stabilizer level, as these will be poisoned more due to the increased concentration of stabilizer arriving at the surface from nonlinear diffusion. Non-linear diffusion results in more rapid diffusion of species to the edge of the bondpads and in unstabilized solutions results in the thicker build up of electroless nickel around the edge of the pad than in the center [20] [see Figs. 3(a) and Fig. 4(a)]. Addition of small quantities of stabilizer results in the poisoning of the edges of the bondpads in preference to the center and evens out the plating across the pad. However, addition of too much stabilizer combined with nonlinear diffusion to the edges of the pads results in excessive poisoning of the plating process and the growth is terminated at the edges leading to the formation of conical bumps. Stirring the solution clearly brings the stabilizer to the surface preferentially in one direction and results in the distorted growth of bumps in a direction parallel to the flow of solution across the surface. Large bondpads are not affected as strongly by nonlinear diffusion and therefore plate more evenly, although still exhibiting some edge effects. However, because the majority of the surface is not poisoned, the electrochemical potential of the pad is maintained at that established by the electroless nickel process and so, therefore, is any small bondpad connected to it. This results in small bondpads connected to large ones plating well, while isolated bondpads produce conical bumps, as observed here.

In a similar set of experiments, Zhang *et al.* [14] studied the electroless nickel plating of flip-chip size features on PCBs. However, their paper challenges the electrochemical potential theory of van der Putten and de Bakker. While they agree that nonlinear diffusion of the stabilizers to the pad surfaces is the cause of the irregular growth, they argue that it is simply the concentration of the stabilizer at the surface that poisons the deposition rather than an electrochemical effect. In their work they observed only “skip” plating or good plating—no conical features. They argue that this indicates only two mechanisms which, once triggered, cannot be changed. The first mechanism involves the almost immediate poisoning of the growth surface by the stabilizer, leading to a skip. This occurs when the rate of arrival of the stabilizer is greater than its incorporation into the coating. This results in a steady increase in stabilizer at the surface, reducing the rate of NiP growth and, in turn, reducing the rate of incorporation. This leads to a faster build up of stabilizer and termination of the plating. The second mechanism involves the opposite situation, with the rate of incorporation of the lead ions into the NiP coating being more rapid than the rate of adsorption of lead ions. This encourages rapid growth of the deposit due to limited poisoning and leads to the formation of good deposits.

The observation of conical defects and directional bumps in this work, does not fit with the two-state mechanism proposed by Zhang *et al.*, but appears to be more closely explained by the theory of van der Putten and de Bakker. The similarity between the effects induced by the addition of lead acetate to a standard bath and the defects observed with the commercial so-

lution leads to the conclusion that too high a level of stabilizer (probably lead acetate) in the commercial solution was the cause of the defective bumping. This highlights the major difference between a solution optimized for plating of relatively large area features compared to the small and isolated bondpads of a silicon chip. In the case of the commercial solution, this will be heavily stabilized to ensure the extended life of the bath with limited amounts of unwanted deposition onto tank walls. In this study it was observed that, on occasion, the commercial solution could operate correctly for a short time before defects started to occur. In particular, good plating took place when the bath was fresh and a smaller volume of solution was used. This leads to the suggestion that the bath became over-stabilized during operation i.e., the lead acetate concentration increased with time in relation to the other components, due to not enough of it being incorporated into the NiP deposit. The commercial solution is designed to operate with a high workload of 0.5 to 2.5 dm² of surface area to be plated for every liter of solution. However, a typical 4 in wafer used in this study had a total bondpad area of only 0.01 dm², but the solution volume was 3.2 l. This is clearly a situation where over-stabilization may occur and is supported by the observation above, that for formulations prepared from individual components, increasing the bath workload temporarily produced well formed bumps from a solution that had previously resulted in defects. A possible method for the prevention of defects with a commercial bath could be implemented by plating a large object together with the wafers in order to maintain the bath workload, but with an inevitable waste of other chemicals due to the unrequired coating produced.

While the conical bumps formed by an incorrectly stabilized bath are undesirable in a standard electroless nickel bumping process, it may represent a useful way of overcoming the problems presented by the isotropic growth of the bump on fine pitch devices. Semiconductor devices designed for wire-bonding applications typically use large pads at relatively fine pitch to enable the maximum number of connections to be made around the periphery of the die. For flip-chip assembly, this arrangement is undesirable as the close proximity of the edge of one bondpad to the next, means that the isotropic growth of the electroless nickel could produce a short between neighboring pads. If the stabilizer level within the electroless nickel bath can be controlled, then conical bumps could be grown preventing the outward spread of the NiP thereby avoiding this problem. While this might not be of use for solder bumped devices due to the spread of the solder ball, this might find application for anisotropic conducting adhesive interconnect.

The electroless nickel bumping of bondpads that had been test probed, resulted in the formation of deposits with uneven surfaces. While this level of unevenness is unlikely to cause problems for subsequent solder bumping, it could present difficulties for processes such as anisotropic conductive adhesive attachment. However, of more concern is the effect that the etching of the underlying pad may have on the long-term reliability of the device. The exposed material (adhesion promoting metal layers or Si/SiO₂) underneath the Al may not be active to electroless nickel deposition and might only be covered by overgrowth of the NiP deposit from surrounding areas. This would result in a weakening of the adhesion between the NiP

and the bondpad and possibly the trapping of corrosive plating chemicals within the structure. While the study of these effects did not form part of this work, Tan *et al.* [5] have investigated the merits of probing wafers before and after electroless nickel bumping to determine these effects. In their work they found that large probe marks did not significantly affect the reliability of the Ni/Au bumped assemblies, but suggested that probing after Ni/Au bumping was a preferred option providing the overdrive of the probing system was not so high as to damage the coating.

Finally, a comment should be made regarding the back face coating of the wafers prior to plating. This has been highlighted by Ostmann *et al.* [21] as an important issue for devices that contain bondpads with direct electrical contact with the Si substrate. In this situation a galvanic cell can be created in the activation and plating solutions through contact with exposed areas of Si. This results in a varied rate of etching/deposition, leading to poor activation, damage to the bondpad, or an uneven NiP plating thickness. In these experiments no back face coating was applied to the test wafers employed, as all the bondpads were separated from the Si substrate by a thermal oxide layer and the back faces of the wafers had a defect free oxide layer. However, for the memory devices shown in Fig. 5 above, one ground pad of the device was directly attached to the substrate. This showed significant variations in zincate activation and subsequent electroless nickel plating as a result of this galvanic cell effect. Fig. 6 shows pictures comparing the surfaces of the ground bondpad with other pads after zincate treatment and after electroless nickel plating. It is clear that very little zincate deposition occurred on the ground pad during the zincate treatment, in comparison with the ordinary pads and this subsequently affected the morphology of the electroless nickel deposit. Unfortunately, no suitable resists were available during these trials with which to protect the rear of the die in order to confirm that these effects could be removed by isolating the Si from the solution.

V. CONCLUSION

The electroless nickel bumping of Al bondpads is a convenient, maskless method for the deposition of an under-bump metallization that is suitable for solder deposition and subsequent flip-chip interconnect. However, the work presented here and by others has demonstrated that to reproducibly bump wafers, carefully controlled bath formulations are required that, in particular, offer close control over the stabilizer level. Furthermore, the treatment of wafers at other steps in the manufacturing process needs to be considered in order to ensure a uniform deposit. Nevertheless, the electroless nickel bumping process is now being carried out commercially by a number of organizations, indicating that it is rapidly becoming a mainstream technology for low cost flip-chip assembly.

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