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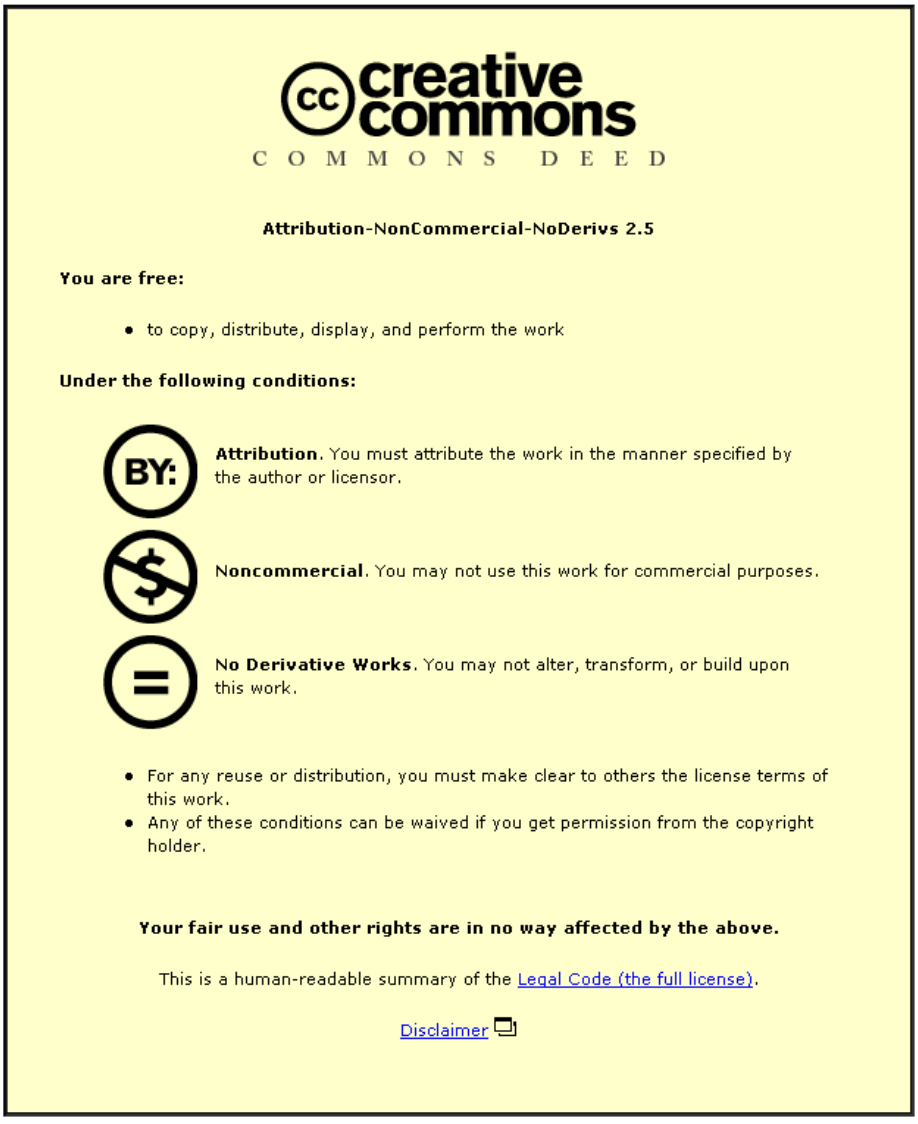

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Electroless Nickel Bumping of Aluminum Bondpads—Part I: Surface Pretreatment and Activation

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

***Abstract*—Electroless nickel bumping of aluminum (Al) bondpads followed by solder paste printing is seen as one of the lowest cost routes for the bumping of wafers prior to flip-chip assembly. However, the electroless nickel bumping of Al bondpads is not straightforward and a number of activation steps are necessary to enable the nickel deposit to form a strong, electrically conductive bond with the Al. For the electroless nickel coating of mechanical components made of aluminum, a zincate activation process has been used for many years, however extension of these techniques to semiconductor wafers requires careful control over these pretreatments to avoid damage to the very thin bondpads. This paper reports a number of experiments designed to characterize the activation of Al bondpads to electroless nickel plating, focusing on the effects of solution exposure time and bondpad composition. In addition, the results are discussed in the context of other studies presented in the literature to provide an understanding of the mechanism of the zincate activation process applied to Al bondpads.**

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

I. Introduction

FLIP-CHIP technology is being taken up rapidly as the demand for smaller, lighter and higher performance electronic devices continues. Although invented in the 1960's, it is only in recent years that the flip-chip process has been investigated in detail to identify reliable low cost manufacturing routes that can match the forecast demands of consumer products. As a result of this interest, many different process routes have been devised for the solder bumping of bare die, their attachment to the circuit board and subsequent underfilling.

Presently, the majority of semiconductor devices use an Al alloy as the interconnection medium within the device and the terminations, or "bondpads," are similarly made of Al. However, the recent introduction of Cu as a replacement to Al within devices may eventually change this. The Al metallization is adequate for traditional packaging of components using wirebonding interconnection methods, however, for solder bumping, the Al bondpad requires an under bump metallization layer (UBM) to be deposited. This provides a solderable interface on top of the otherwise unsolderable Al surface and helps to protect the fragile bondpads from fluxes and solder dissolution during reflow.

A number of under bump metallization routes have been developed for the preparation of Al bondpads. Of these the electroless nickel plating process appears to offer a low cost, high volume solution, especially when combined with solder paste printing for solder bumping [1]–[6]. The technique is a chemical process and is maskless, such that no lithographic steps or tooling are required and is therefore cost effective. In addition, the electroless nickel process is well characterized and has been employed for many decades for the finishing of engineering components and, more recently, for the finishing of copper tracks on PCB's together with a gold flash to improve solderability [7], [8]. The extension of this technology to the bumping of small isolated Al bondpads, as compared to the relatively large area features for which electroless nickel has been previously employed, presents new challenges and this paper sets out to identify some of these issues.

The direct plating of electroless nickel onto Al is not reliable and if successful leads to poor quality deposits with low adhesion. It has been known for many years that only the Group 8 transition metals can be coated directly with electroless nickel [7], [8]. Therefore for other materials, a number of activation processes have been developed to create nucleation sites on the surface around which Ni can grow. Al falls into the class of metals that are not directly active to electroless Ni plating, but will begin to coat after some nuclei have formed at the surface. However, the deposits formed in this way are not of good quality and, in order to improve the adhesion and properties of the films, the pretreatment zincate process was developed [9]–[13], which involves the exposure of the component to a solution containing zincate ($[Zn(OH)_4]^{2-}$) ions. Generally, a highly alkaline solution is employed with the basic components being ZnO and NaOH. In addition, additives are often incorporated usually in the form of complexing agents such as potassium sodium tartrate and other metal ions such as Fe, Cu, and Ni [9], [13], [14].

The use of electroless nickel for the formation of the UBM in flip-chip assembly has prompted a number of studies of the zincate activation process applied to Al bondpads [15]–[20]. The etching and pretreatment stage of the bumping process has an important role to play in determining the overall properties

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and reliability of the final solder bumped flip chip. In particular, the adhesion of the nickel layer to the bondpad is critically dependent on the nature of the zincate process used. It is unclear exactly how the zincate process promotes the electroless nickel deposition on the Al surface, however, its importance in the improvement of the nucleation process has been clearly demonstrated [2]. Based on the earlier studies of aluminum alloy mechanical component coating, it has been found that the adhesion of the Nickel–Phosporous (NiP) deposits can be improved dramatically by partially etching off the previous zinc layer using nitric acid and then repeating the zincate process: a so-called "double zincate" treatment. A number of studies have shown that thin, fine grained deposits, produce better adhering Ni films and that this structure is promoted by multiple zincation treatments. This approach has therefore been carried forward into the process route developed for the treatment of Al bondpads.

In this paper, the activation of the Al surface and the subsequent adhesion of the electroless nickel deposit will be discussed based on experimental studies and information presented by other groups in the literature. In part 2 of this study, the key issues in the electroless nickel deposition process will be presented [21].

II. Experimental

The overall process route used in this work is shown in Fig. 1. Each stage was investigated to assess its impact on the bumping quality. The process begins with etching of the Al surface using sodium hydroxide and nitric acid which is followed by a zincate activation step, before the final electroless nickel plating step. The following sections describe the materials and bath compositions that were used in these studies.

A. Wafers

The majority of the bare test die (wafer Types A and B) used in these experiments. The majority of the bare test die (wafer Types A and B) used in these experiments consisted of a peripheral array of daisy chain interconnected bondpads at pitches of 225 μm and 300 μm. These provided bondpads octagonal in shape that measured 90 μm across the flats, but had circular openings in the silicon nitride passivation of 75 μm diameter. Wafer type B also had octagonal bondpads intended for BGA interconnection that were 600 μm across with a 580 μm diameter opening in the passivation. Many of these larger bondpads were electrically connected to the inner array of smaller bondpads. The Al thickness of all bondpads was 3 μm and consisted of Al with 1 Wt%Cu. In addition to bare die, macroscopic test samples of Al coated Si wafers were also supplied. These had an AlCu (1%) coating of 1 μm thickness that was deposited in the same manner as the interconnections on the die. For the majority of experiments, only segments of wafers were used, which were prepared by scribing and breaking into approximately 20 mm $\times$ 20 mm pieces.

A third type of test wafer (wafer Type C) was fabricated by the Queen's University of Belfast to examine the effect of bondpad pitch on the bumping process. These wafers had peripheral and full array daisy chain patterns with pitches of 150, 125, 100, and 90 μm. The bondpads were octagonal in shape, measuring 60 μm across the flats except for the 90 μm pitch pads that were 50 μm across. In all cases, the openings in the SiO_2 passivation were circular. These wafers were metallized with pure Al that was only 1 μm thick.

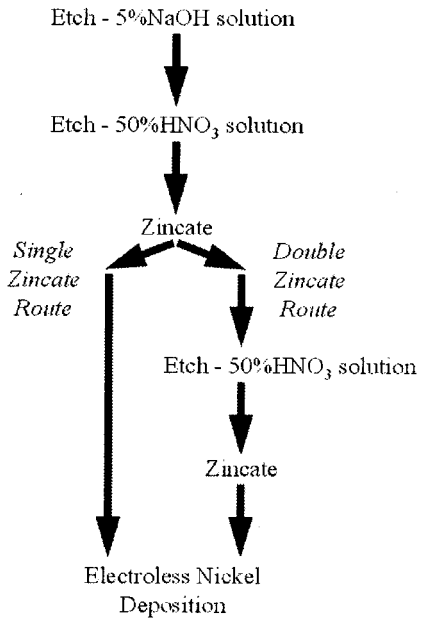


Fig. 1. Process route for electroless nickel bumping of Al bondpads.

B. Etchants (Sodium Hydroxide and Nitric Acid)

For oxide removal and cleansing of the bondpads, sodium hydroxide (NaOH) and nitric acid (HNO_3) etchants were used. First, the die were etched in a 5 wt% (1.25 M) NaOH solution in water for a period of 5 to 30 s at room temperature. The degree of etching depended on the individual device and the etch time was kept to a minimum to prevent excessive removal of Al from the bondpad. Following the NaOH etch, the die were rinsed with water and then dipped into a 50% (10 M) nitric acid solution for 20 s, again at room temperature. The die were then rinsed with deionised water before proceeding to the zincate activation step. The concentrated nitric acid etch is used to remove the metal "smut" materials left behind by the NaOH etch and helps to remove any remaining organic residues on the pad surface. A number of studies have employed 5% hydrofluoric acid in this solution, however it was felt that this was not necessary for the Al or AlCu pads and could have led to damage of the SiO_2 wafer coating.

C. Zincate Activation

Initial studies employed a zincate solution prepared from individual components of ZnO and NaOH, similar in composition to a number of published baths. However, while these activated the surface sufficiently for electroless nickel bumping to take place, initial trials showed that they did not provide strong adhesion between the deposit and the bondpad surface. A commercially available solution was therefore used for the main trials, which was a modified, alkaline zincate bath.

The zincate treatment was applied to the die at room temperature, immediately after the etching processes, by dipping them into the solution for periods of 10 to 60 s and then rinsing with deionised water. For samples that were to have a double zincate treatment, a second etch in 50% HNO_3 for 10 to 20 s was carried out followed by a rinse and a repeat application of the zincate treatment.

D. Electroless Nickel Plating

After zincate activation of the Al surface, the die were plated in the electroless nickel bath. Again, a number of different baths were evaluated to determine any effects the composition of the plating solution might have on the bumping process. Details of these baths are given in Part 2 of this study. For the studies presented here, a standard hypophosphite based electroless nickel plating solution operating at a pH of 4.6–4.8, was prepared from individual compounds to provide complete control over the bath composition. In general a bath volume of 3.2 l was employed during these studies, with the temperature controlled at 85 °C producing a plating rate of 18–20 μm hr^{-1}.

III. Results: Wafer Type A and B

A. Al Bondpad Etches

The influence of each stage of the bumping process on the bondpads was investigated. Fig. 2 shows the morphology of the AlCu bondpad before and after etching for 20 s with 5% NaOH solution. For the untreated sample, the grain structure of the 3 μm thick AlCu is clearly visible. After etching, the surface still displayed the grain structure, which appeared slightly clearer and there were small etch pits across most of the surface. After etching the surface with 50% nitric acid solution for 20 s, the surface of the bondpad showed very little further change.

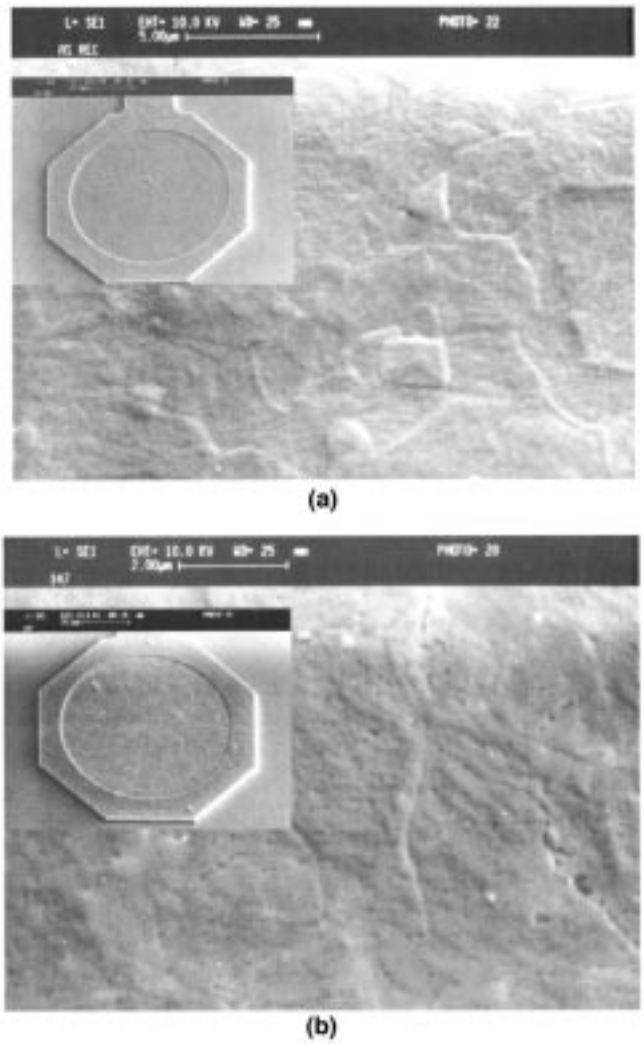


Fig. 2. SEM images of Al bondpads on wafer type A (a) before treatment and (b) after 20 s etching in 5% NaOH solution.

B. Zincate Treatments

The surfaces of the AlCu (1%) bondpads following single and double zincate treatments were investigated using SEM observation. Fig. 3 shows examples of the surface morphology of the bondpads following such treatments. In Fig. 3(a), a single zincate treatment of 20 s has been applied to the bondpad producing many large particles across the surface which were identified by energy dispersive analysis of X-ray (EDX) as zinc. The zinc clusters appear to be concentrated around the grain boundaries in the Al structure seen on the bare bondpads, which is similar to the observations of Monteiro *et al.* [12] who investigated the zincate treatment of large Al materials and Baggerman and Schwarzbach [19] who studied Al bondpads. Closer examination of the bondpad surface showed that the Al surface between the large zinc clusters had become pitted.

For a double zincate treatment, the first zincate layer was fully etched away by immersion in the 50% HNO_3 bath. Observation of the bondpad surface after this treatment (Fig. 4) revealed the complete removal of the zinc particles and showed the underlying pitting in the Al surface that could be seen in Fig. 3(a). This was again in agreement with Monteiro *et al.* [12] but, in contrast to other studies where residual amounts of zinc were detected [10], [22], EDX analysis indicated no zinc present on the surface. To determine if this was a result of the insensitivity of EDX to thin layers of material, this was further investigated by using X-ray photoelectron spectroscopy (XPS) analysis of Al films of the same composition sputtered onto Si wafers. The XPS results obtained from these larger samples (Fig. 5) supported the EDX results showing no zinc on the surface after only 5 s etching in 50% HNO_3 solution.

After removal of the first zincate layer, the zincate treatment was repeated to deposit a fresh layer. Fig. 3(c) shows SEM images of the surface morphology of the bondpads after a 20 s double zincate treatment. In agreement with other studies, this shows clearly that this treatment produces a more uniform zinc layer, which is comprised of many small zinc clusters, in contrast to the single zincate process. Some evidence of the grain boundary structure of the original Al can still be seen, but is now much less significant.

Similar results were obtained for bondpads given 60 s treatments. The single 60 s zincate treatment [Fig. 3(b)] produced a more uniform zinc layer compared to the 20 s single treatment, but this was still comprised of large individual particles.

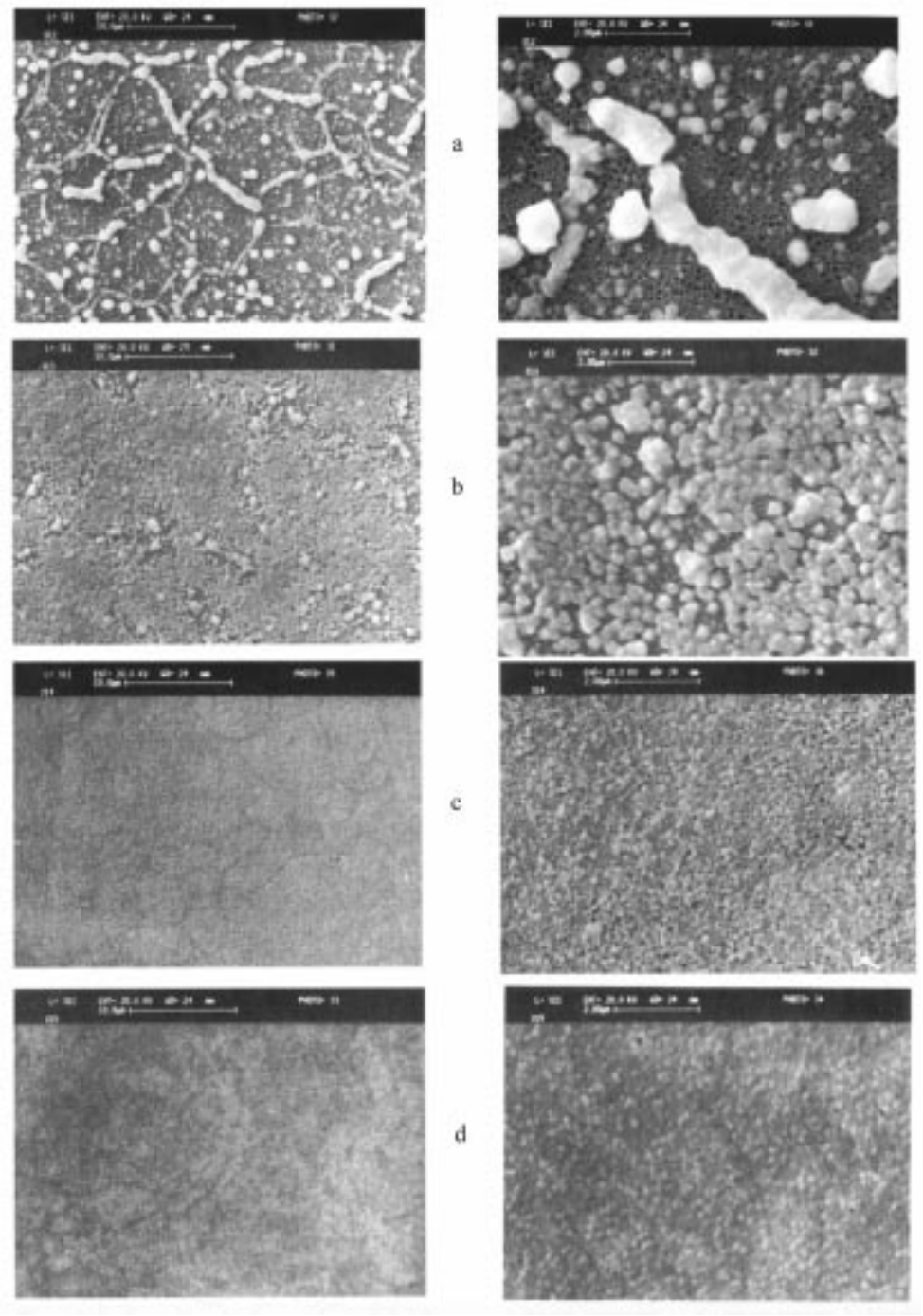


Fig. 3. Surface of bondpads on wafer type A following zincate pretreatment: (a) 20 s single zincate, (b) 60 s single zincate, (c) 20 s double zincate, and (d) 60 s double zincate. The images shown in each column were recorded with the same magnification for direct comparison (right hand column at higher magnification than left).

Similarly, a double zincate treatment of 60 s duration [Fig. 3(d)] produced a thinner, denser and more uniform zincate layer compared to the single zincate treatment.

For the results presented in Fig. 3, the zincate treatment produced a uniform effect across all the bondpads of the die. However, for other batches of wafers, the zincate treatment produced differing degrees of zincate growth on the bondpads across a particular chip. This was very noticeable for single zincate treatments and Fig. 6 shows an example of the different zincate layers that could be obtained. For double zincate treated samples, the zincate structure across the bondpads on the die was more consistent and resembled that observed in Fig. 3(c). The reason for this variation was unclear but comparison of the images in Figs. 3(a) and 6 indicates that the bondpad microstructures may have been different and might, therefore, be related to variations in the manufacture of the wafer batches.

C. Adhesion of Electroless Nickel Bumps to Bondpads

The effect of the pretreatment processes on the adhesion of the electroless nickel bumps to the bondpads was characterized

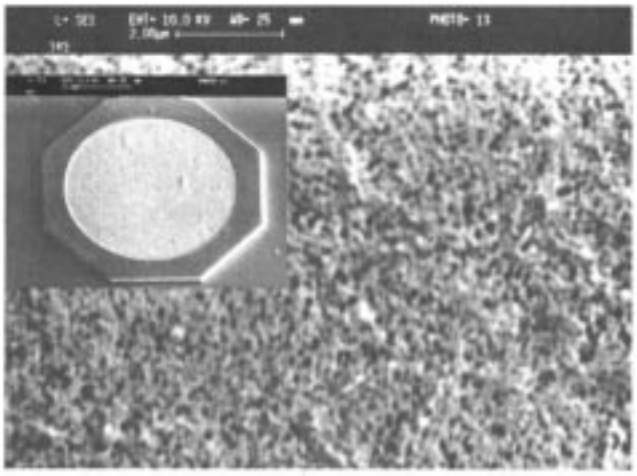


Fig. 4. SEM images of the bondpad surface following 50% HNO_3 etching for 10 s after a single zincate treatment.

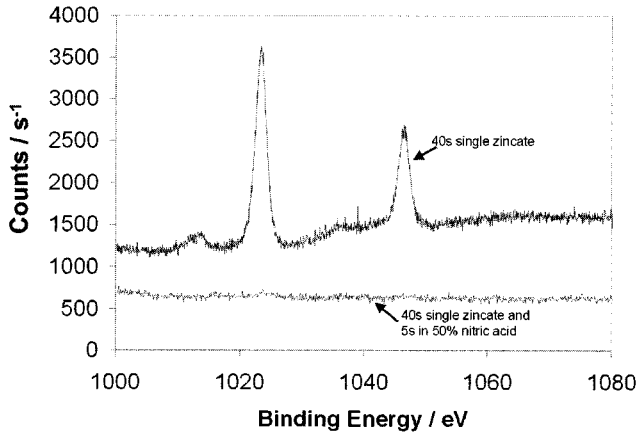


Fig. 5. Zn 2 p XPS spectra obtained from Si samples coated with AlCu (1 wt%). Top curve after 40 s single zincate treatment, bottom curve, after 40 s single zincate treatment and 5 s etch in 50% HNO_3 solution.

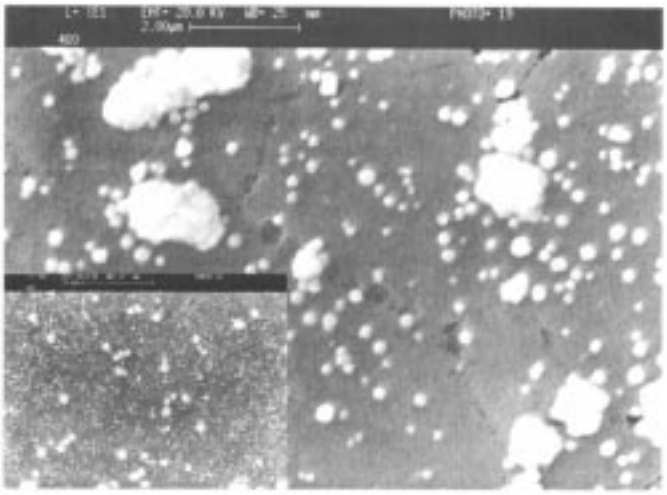


Fig. 6. SEM images obtained from a 20 s single zincate treated sample showing different surface finish to that obtained in Fig. 3(a).

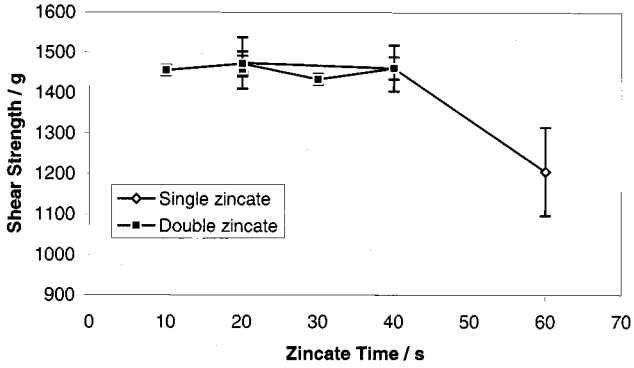


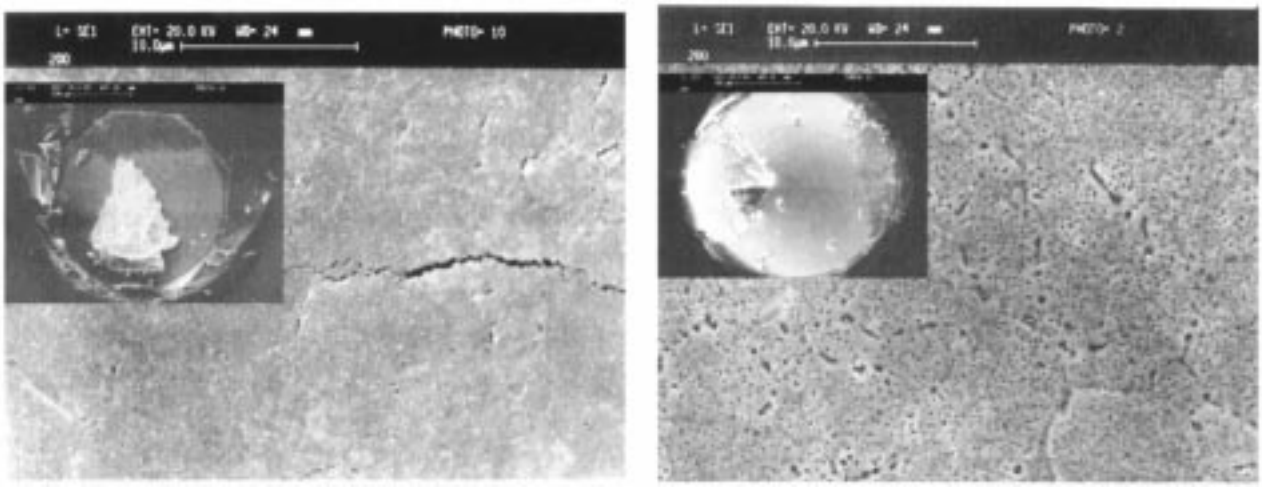
Fig. 7. Shear test results as a function of zincate activation time for BGA solder balls attached to the large bondpads (580 μm passivation opening) of wafer type B. Shear speed: 250 μms^{-1}, Shear height: 50 μm.

using shear testing with a range of structures. Due to difficulties in carrying out shear tests directly on NiP bumps, initial trials were conducted on the large BGA sized bondpads of wafer type B, which were solder bumped by reflowing BGA solder balls onto them. A number of these die were bumped, using a range of zincate treatments and were then characterized by shear testing. Fig. 7 shows the results of these tests and indicates that for the majority of single and double zincate treatments the shear force was very similar, which could be correlated with SEM observations of the fracture surface that clearly indicated that shear failure always occurred through the solder material. The only exception to this was the 60 s single zincate treated sample, which exhibited reduced shear strength and where many fractures could be seen at the NiP–Al interface (Fig. 8).

Following these initial measurements, further experiments were conducted to determine directly the shear strength of the electroless nickel bumps. These measurements need to be made with great care, as the size and shape of the NiP bumps can strongly influence the results. It is very difficult to test NiP bumps of only 5 μm height, as the shear tool cannot gain purchase on the bump edge and skids over the hard material surface. In order to overcome this issue, early trials used thick (30 μm) bumps that were deposited to create tall features that could be easily engaged by the shear test tool. However, the substantial isotropic overgrowth of the nickel bump onto the passivation surrounding the bondpad resulted in the shear strength of the Al pad attached to the Si surface being measured instead of the desired NiP–Al interface. Further samples were therefore prepared with a 15 μm bump height that allowed relatively easy testing without further complications. These results are presented in Fig. 9 and show a general decline in the shear strength of the interface as the zincate time is increased. Furthermore, for a fixed zincate time, the double zincate treatment provided a higher strength interface than the single treatment, in general agreement with other studies in the literature [15]–[17], [23].

D. Electrical Resistance of Bumped Structures

The electrical resistance of daisy chained bondpads on wafer A were measured before and after electroless nickel bumping to ensure that the treatments were removing the oxide layer on the Al surface and creating a low resistance interface between



(a) (b)

Fig. 8. Fracture surfaces of: (a) bondpad and (b) solder ball following shear testing. Sample was electroless nickel bumped after a 60 s single zincate treatment.

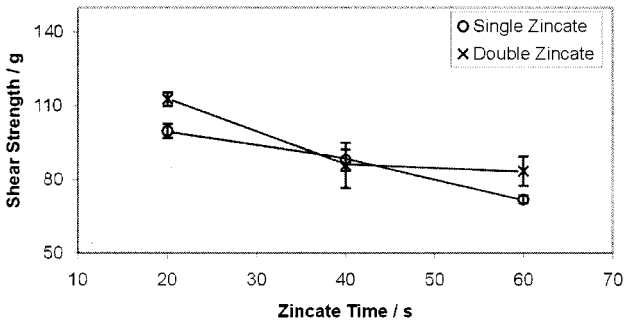


Fig. 9. Shear strength of electroless nickel bumps on wafer type A as a function of zincate treatment time. Shear test speed: 150 μms^{-1}, shear height: 7 μm.

TABLE I
ELECTRICAL RESISTANCES BEFORE AND AFTER ZINCATE TREATMENT OR ELECTROLESS NICKEL PLATING

Wafer Type		Wafer A	Wafer A	Wafer C
Pitch (μm)		300	225	150
Resistance (Ω)	Un-bumped	0.217 ± 0.001	0.182 ± 0.001	0.386 ± 0.001
	Ni-bumped	0.218 ± 0.001	0.183 ± 0.001	0.453 ± 0.004***
	After zincate treatment only	-	-	0.446 ± 0.004*
	After zincate treatment only	-	-	0.59 ± 0.01**

* 10s single zincate after 10 s etching in 5% NaOH solution
** 10s single zincate after 20 s etching in 5% NaOH solution
*** 5 μm Ni plated after 10s single zincate treatment with 10 s etching in 5% NaOH solution

the Al and the electroless nickel deposit. Four point electrical measurements were made of the pairs of bondpads joined on the die by Al tracks. Table I shows the effect of the electroless nickel bumping treatment on wafer A for which the resistance rose only slightly. The slight increase in resistance observed could be accounted for simply by the resistivity of the nickel bump itself, indicating that the zincate treatment being applied was successful in creating a low resistance interface. This was found to be the case for all zincate treatments investigated.

E. Investigation of the Zincate Activation Mechanism

In order to develop an understanding of the mechanism of the zincate activation of the Al surface to electroless nickel plating, a number of samples were given a zincate pretreatment and then immersed into the electroless nickel bath for a short period of time (10 s to 2 mins). Following this, they were immediately rinsed and dried to "freeze" the reaction. Fig. 10 shows SEM results obtained from these samples and Fig. 11 shows the EDX analysis from the same samples. For samples that received a 20 s double zincate treatment, a 10 s immersion into the electroless nickel bath produced a surface difficult to distinguish from the original smooth zincate structure, which with further exposure to the electroless nickel plating bath developed into the familiar electroless nickel surface finish. The results for samples prepared using a 60 s double zincate pretreatment showed very similar results. For a 20 s single zincate treated sample, the electroless nickel growth closely followed the original structure of the zincate layer, with electroless nickel growing in nodular chains associated with the grain boundaries of the Al bondpads and also nucleating and growing in between these areas.

The EDX analysis of these samples (Fig. 11) revealed a rapid decrease in the amount of zinc present at the surface as a result of the very brief immersion into the electroless nickel bath. Some attenuation of the zinc signal might be expected as the NiP layer covers it, however the extent of the zinc reduction would appear to indicate the dissolution of the zinc into the bath.

IV. RESULTS: WAFER TYPE C

A. Pretreatment and Activation of Bondpads

The bondpads on wafer type C were both thinner (1 μm) and a different composition (pure Al) to those on types A and B. This presented an opportunity to characterize the effects the different pretreatments would have on this less common composition. The morphology of the Al bondpads was examined using SEM (Fig. 12) and were found to have a grain structure with substantial etch pits in the vicinity of the grain boundaries. This led to concern regarding the effects that the pretreatment procedures could have on the bondpads. In addition, there were many

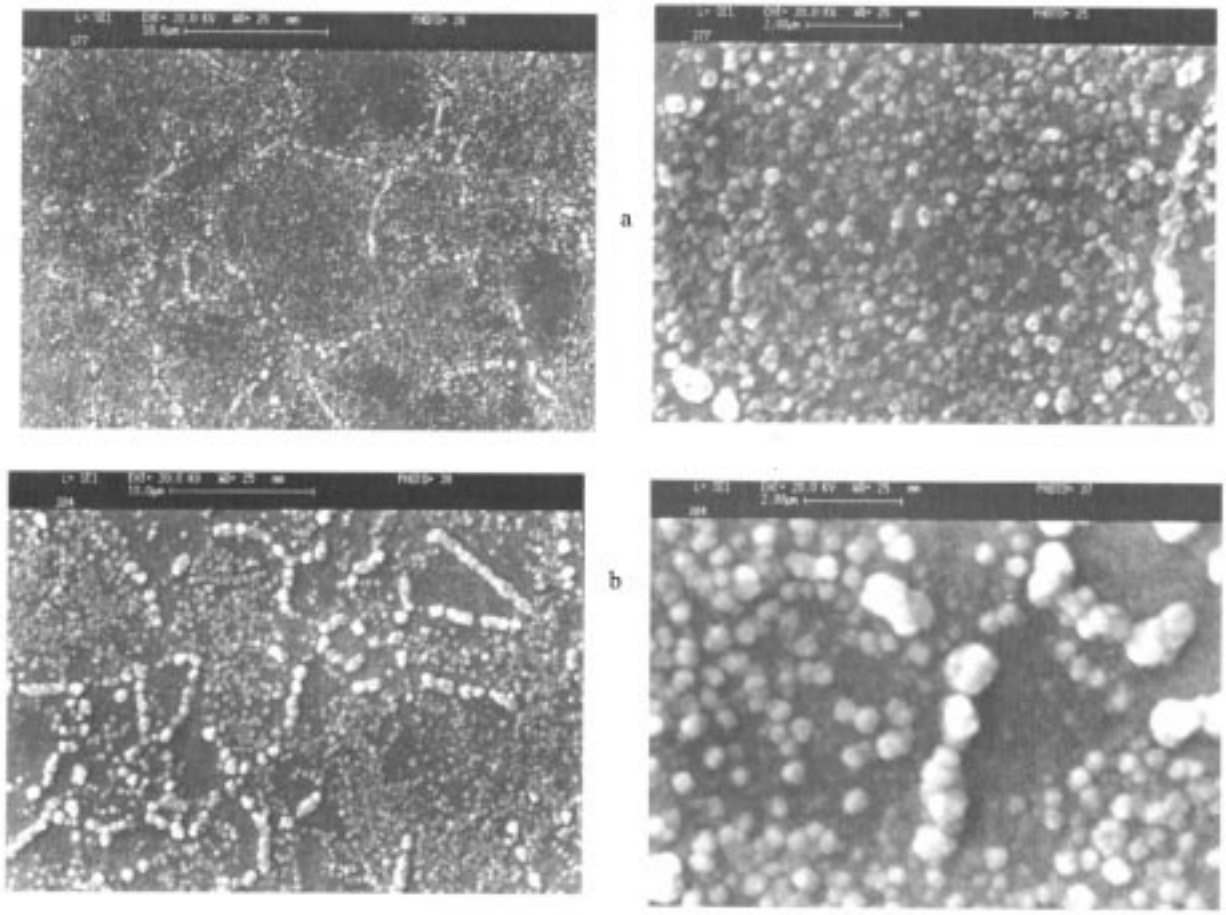


Fig. 10. SEM images of zincate treated samples exposed briefly to the electroless nickel bath. 20 s single zincate treatment followed by: (a) 10 s and (b) 60 s in the electroless nickel bath.

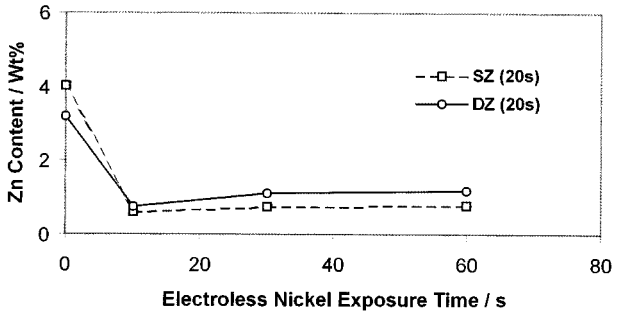


Fig. 11. EDX analysis of the bondpad composition following zincate treatment and exposure to the electroless nickel bath. SZ = single zincate, DZ = double zincate. Results show Zn content in wt%.

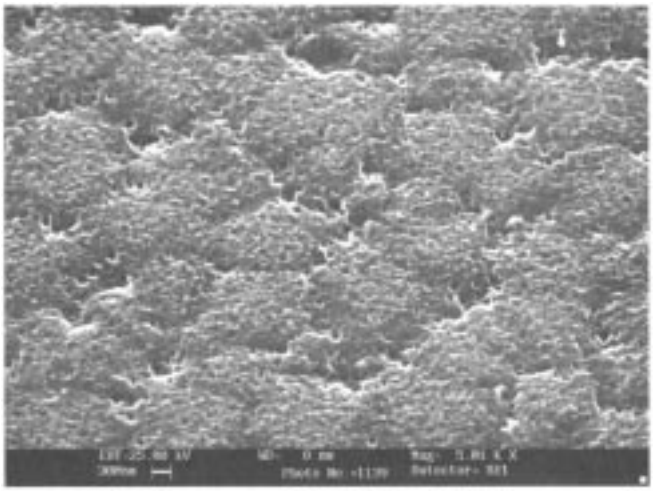
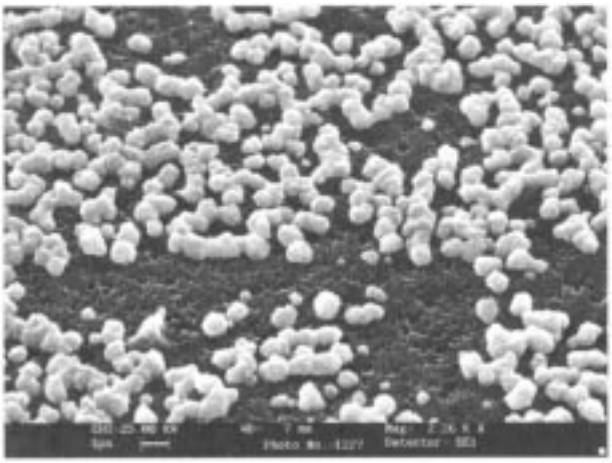


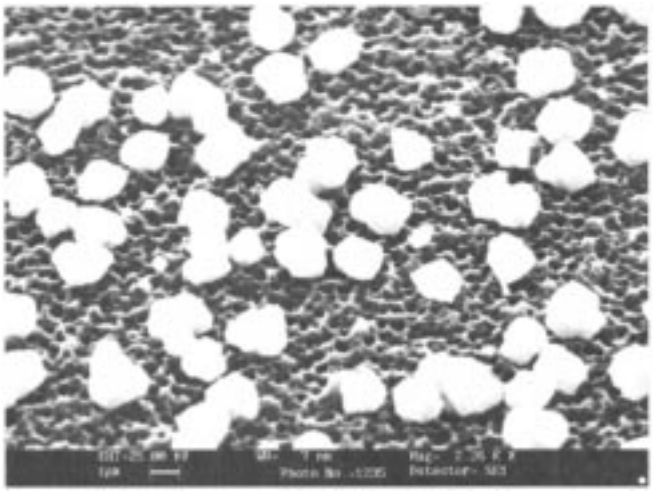
Fig. 12. SEM image of the untreated surface of the Al bondpads of wafer type C.

fragments of material covering the surface of the wafer that had been deposited by the manufacturing process.

The application of a single zincate treatment to the bondpads produced a number of zinc clusters across the surface of the bondpads and small etch pits in the underlying Al material (Fig. 13). This was similar to the effect observed with wafer A, however, a double zincate treatment did not produce a smoother finer grained zinc deposit, instead larger more isolated clusters were formed with more substantial etch pits in the Al surface. EDX analysis of the bondpads revealed that almost all of the Al had been removed by the treatments even for a brief 10 s double zincate exposure. As expected, this effect was made worse by prolonged etching in the 5% NaOH solution at the start of the bumping process. This indicated a much faster etch rate of the pure Al bondpads in the alkaline NaOH and zincate baths compared to AlCu (1%). It was therefore necessary, for reliable electroless nickel bumping, to minimize the etching and zincate treatment times on these samples.



(a)



(b)

Fig. 13. Zincate structure on wafer type C after: (a) single and (b) double treatments.

B. Adhesion of Electroless Nickel Bumps to Wafer C Bondpads

The adhesion of electroless nickel bumps plated onto the bondpads of wafer C was investigated using shear testing in the same manner as for wafers A and B. Table II shows the results of these tests for samples with a variety of pretreatments. In contrast to the results obtained for wafer A, the length of the initial NaOH etch had a substantial effect on the shear strength of the bumps. In particular, for a 10 s double zincate treatment, the shear strength could be more than doubled by etching with NaOH for only 10 s instead of 20 s.

C. Electrical Characterization of Electroless Nickel Bumped Wafer C

Table I shows the results of four point resistance measurements made on pairs of bondpads linked by an Al track. After electroless nickel bumping, the resistance had increased significantly in contrast to the results obtained with wafer A, however in order to identify whether this was entirely due to the electroless nickel bump or another factor, results were taken for samples at various stages of the pretreatment process. These revealed that the majority of the resistance change was taking place as a result of the activation steps and was strongly affected by the NaOH etch time, leading to the conclusion that the resistance change was a result of the thinning of the Al pad rather than the incomplete removal of an oxide layer.

TABLE II
SHEAR STRENGTH (IN GRAM FORCE) OF ELECTROLESS NICKEL BUMPS ON WAFER TYPE C AS A FUNCTION OF ZINCATE TREATMENT TIME

	Shear Strength / g		
Etch time in 5% NaOH / s	10 s double zincate treatment	10 s single zincate treatment	20 s double zincate treatment
10	124 ± 8	124 ± 7	-
20	48 ± 5	-	41 ± 12

V. DISCUSSION

While there have been many studies in the literature regarding the zincate activation of Al, many of them relate to the activation of large engineering parts rather than small features such as Al bondpads. In addition, the studies that characterize the adhesion of deposits are based on a variety of electroless nickel bath formulations and zincate treatments, which could significantly influence the results. The following discussion attempts to correlate the results of many of these studies with observations made during the experiments reported here and also aims to provide an overview of the key features of the zincate process that influence the reliability of the bumping process.

A. Mechanism of Zincate Deposition

The mechanism of the initial zincate deposition onto the Al surface has been described by a number of other research groups [24], [25], [11]–[14], [2], [19]. The process is a redox one involving the dissolution of Al metal from the surface thereby releasing electrons to reduce Zn ions from solution. This can be summarized by the following equations [2]:

Anodic dissolution of aluminum:

$$\mathrm{Al}_{(s)} + 3\mathrm{OH}^- \rightarrow \mathrm{Al(OH)}_3 + 3e^-$$

Cathodic deposition of zinc:

$$\mathrm{Zn(OH)}_4^{2-} \rightarrow Zn^{2+} + 4\mathrm{OH}^-$$
$$\mathrm{Zn}^{2+} + 2e^- \rightarrow \mathrm{Zn}_{(s)}.$$

The SEM images of the single zincate step [Figs. 3(a) and 13] indicate that this process is not completely uniform and produces large clusters of zinc preferentially at the grain boundaries of the Al pad. What appears to be occurring is the dissolution of the Al between the grain boundaries leading to the formation of the pits that are visible in the background of Figs. 3(a). These pits persist after the Zn is etched away by the nitric acid and presumably provide a new set of nucleation sites for the second zincate deposition step leading to a more uniform and regular growth. The enhanced growth at the grain boundaries could be expected due to the higher surface free energy of these regions, which capture the Zn atoms during deposition. Baggerman and Schwarzbach [19] suggest that this is due to a thinner aluminum oxide layer in these regions, which is broken down more rapidly than elsewhere by the zincate solution, leading to the early nucleation of zinc clusters at these sites. Once these nuclei form

they would seem to grow preferentially to other potential nucleation sites that exist across the surface. However the 60 s single zincate treated sample showed a relatively uniform distribution of large zinc clusters that were smaller in size than those on the 20 s single zincate sample. This observation of reduced cluster size with extended zincate treatment time is in agreement with Ng *et al.* [16]. In their work, they used AFM to monitor the formation of the zinc clusters and also noted that the initially large zinc clusters formed by the zincate solution immersion, decreased in size with continued exposure time to the zincate solution, implying dissolution of the zinc layer.

The reason for the variation in the zincate activation of the bondpads across some of the die is unclear, but may be a result of passivation residues [18], electrochemical cell effects, or Al/oxide composition variations. Similar effects have been observed by Tan *et al.* [20] who monitored the "coloration" of the bondpads under an optical microscope following a zincate treatment. They found five different levels of coloration and related these to the electrical resistance of the deposited electroless nickel bumps and strength of the interface. Overall, they found that certain coloration levels produced weaker adhesion of the electroless nickel deposit to the Al, but this was still at an acceptable level to produce reliable flip-chip assemblies. In our experiments, we have not noticed any significant variation in shear strength across a sample.

B. Mechanism of Zincate Activation of Al Surface for Electroless Nickel Plating

It is clear from the shear test results for wafer A that a thinner and more uniform zincate layer leads to stronger adhesion of the electroless nickel deposit to the Al substrate. In addition, it can be seen that shorter zincate times also provide a higher degree of adhesion which is in agreement with a number of other studies [15]–[17].

A number of papers have suggested mechanisms for the zincate activation of Al surfaces before electroless nickel plating [26], [27], [23]. Zinc, like Al, is not directly active to the deposition of electroless nickel so it would appear unlikely that the zincate acts simply as an intermediate layer between Al and Ni. Generally, it is reported that the zincate layer is dissolved in the electroless nickel bath and it is this process which activates the surface. Whether this route exposes clean unoxidized Al onto which Ni adsorbs from solution or whether the dissolution of zinc establishes an electrochemical cell which reduces the Ni ions, is unclear. According to the work of Thurlow [13], the zincate film is dissolved and then redeposited together with the Ni producing a zinc rich zone (ZRZ) near the interface. However, Armyanov and co-workers [26], [27] have used depth profiling combined with Auger electron spectroscopy to examine the interfacial region between NiP deposits and Al. They found little or no zinc at the interface for a number of samples. Interestingly, they observed more zinc remaining within a sample that was nickel plated at a higher temperature, and consequently higher plating rate, than for lower temperature. They attributed this to the more rapid nucleation and isotropic growth of nickel at the higher temperature, covering over areas of undissolved zincate and entrapping them at the interface. They also noted that the presence of zinc at the interface, resulted in a weakening of the adhesion between NiP and Al. The results of the shear tests presented here and by Lin *et al.* [15] would appear to support this picture.

The idea that the zinc dissolves into the plating solution is in agreement with observations made as part of these studies. The samples exposed briefly to the electroless nickel solution showed a significant decrease in the amount of zinc present. While the deposition of some nickel (approximately 0.05 μm during the 10 s immersion time) is expected to attenuate the zinc X-ray signal in EDX analysis, the dramatic decrease in the apparent zinc content cannot be explained by this alone. Furthermore during the first few seconds of immersion of the wafers into the electroless nickel bath, gas could be seen bubbling up from the surface which rapidly died away, indicating an initial vigorous reaction. It is therefore suggested that this is the zinc layer dissolving into the bath and providing the electrons necessary to reduce the nickel ions in solution near the surface. Once a layer of nickel ions has been formed, the conventional autocatalytic electroless nickel plating process can take place, with any remaining zinc no longer dissolving due to the nickel covering.

Observation of the surfaces produced by short exposure to the electroless nickel bath indicated the uniform nucleation of the nickel coating for double zincate treated samples. However in contrast to this, the SEM observations of 20 s single zincate treated samples (Fig. 10) showed structures similar to those observed for the zincate coating alone [Fig. 3(a)], indicating that the nickel was growing over the original zinc structure as well as nucleating in the areas between clusters. For the double zincate treated samples, more zinc was detectable on the 60 s treated samples compared to the 20 s treated ones for the same immersion time in the electroless nickel solution. This appears to suggest a mechanism whereby the zinc is quickly etched by the electroless nickel solution creating electrons to reduce the Ni ions on the surface. For a uniform thin zincate layer, this takes place efficiently across the surface, creating many nucleation sites. However, for the thicker and more nodular coatings, some areas of zinc are not etched completely before they are covered with nickel, so that they become trapped under the coating. This could produce two effects: first the trapping of large zinc particles under the nickel layer which could represent weak points in the structure and secondly, the curtailment of the zinc dissolution process preventing the reduction of additional nickel ions on bare areas resulting in limited contact. Some evidence for this can be seen in the high magnification SEM image of the sheared solder ball bumped bondpad on wafer B after 60 s single zincate treatment [Fig. 8(b)]. For the 60 s single zincate treated sample, fracture occurred predominantly at the Al/NiP interface. The presence of the impressions on the underside of the Ni surface after shear matched quite closely the arrangement of the large zincate crystallites on the bondpad surface before plating. These could indicate that the nickel nucleated on the thinner areas of zincate first, where its dissolution was most rapid and then grew over the large zincate crystallites. However, this mechanism is most strongly supported by the shear test results (Fig. 9) that indicate that a double zincate treatment is preferable to a single treatment and that extended zincate times lead to weaker adhesion of the Ni coating.

It is clear from the results that for wafer type C, having pure Al bondpads, the pretreatment regime was critical to maintaining the structural integrity of the bondpad. The AlCu (1 wt%) pads of wafers A and B did not show significant etching by either the NaOH or zincate treatments. In addition, these pads were much thicker and would not have been as strongly affected by the same degree of etching. Lu *et al.* [28] studied the zincate activation of Al and AlCuSi bondpads and noted that the AlCuSi pads etched much faster than the pure Al. In this study it was found that the Al pads etched faster than the AlCu ones. This difference between the two studies may be attributable to the more uneven surface morphology of these pads to begin with. It is the high rate of etching and potential for damage of the bondpad by the pretreatments that has prompted some researchers to examine alternative activation procedures that use either modified zincate baths [29] or alternative direct nickel deposition techniques [30]–[32].

Shear testing of solder bumped bondpads showed that most of the zincate treatment regimes used, produced sufficient adhesion to ensure that shear took place through the solder material rather than at the NiP–Al interface. For wafer type C, despite the severe etching of the bondpad by the pretreatments, the shear strength could be higher than for wafer type A. This may be a result of the severe pitting induced by the pretreatments that roughened the surface leading to more mechanical interlocking of the NiP deposit with the bondpad. Bump pull tests would be a better way of determining the adhesion of the NiP deposit to the Al bondpad. In particular, this type of measurement would avoid some of the bump shape and size issues that affected the shear test values reported above. At present there do not appear to be any studies in the literature that report this type of measurement.

VI. Conclusion

The activation of Al bondpads using a zincate pretreatment has been investigated and compared with other data presented in the literature. In general it was found that a short double zincate treatment gave the best adhesion of the electroless nickel deposit and leads to minimal etching of the Al bondpad, which is in agreement with data presented by other groups. The other pretreatment steps, especially the sodium hydroxide etch, must also be carefully controlled to ensure that the Al bondpads are not etched severely, which can result in weaker adhesion and increased electrical resistance.

The mechanism of the zincate activation process has been investigated by briefly exposing zincate treated samples to the electroless nickel bath and the results support the suggestion that the zinc dissolves into solution releasing electrons to reduce the nickel ions, thereby creating nucleation sites on the surface. Again, the results indicate that the thinner more uniform zincate layers produce more uniform nucleation and growth of the electroless nickel coating.

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