Research Article

Fabrication of Ni-Mn Microprobe Structure with Low Internal Stress and High Hardness by Employing DC Electrodeposition

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Due to its widely tunable mechanical property and incompatibility with most solders, Ni-Mn alloy can become a viable candidate in the fabrication of testing probe for microelectronic devices. In this study, the electrodeposition of Ni-Mn alloy in nickel sulphamate electrolyte with the addition of manganese sulphate was investigated under direct current (DC) power source. The effects of current density and Mn^{2+} concentration in the electrolyte on the coating composition, cathodic efficiency, microstructure and mechanical properties were explored. The results showed that the raise of the Mn^{2+} concentration in the electrolyte alone did not effectively increase the Mn content in the coating but reduce the cathodic efficiency. On the other hand, increasing the current density facilitated the codeposition of the Mn and rendered the crystallite from coarse columnar grain to the refined one. Thus, both hardness and internal stress of the coating increased. The fabrication of testing probes at $1 A/dm^2$ was shown to satisfy the high hardness, low internal stress, reasonable fatigue life, and nonsticking requirements for this microelectronic application.

1. Introduction

Accompanying the evolution of integrated circuits (ICs) chips toward miniaturization and complicated functionality, the demand on the development of high compaction probe card for quality assurance has posed a difficult engineering challenge. During the IC testing, the probe tip must have enough sharpness and hardness to penetrate the surface oxide layer of the solder pad or connection bump and conduct the electric circuit. On the other hand, the probe structure also needs to have high flexibility and strength to provide the acceptable contact force and fatigue life. Therefore, the development of the probe structure with high dimensional accuracy and high strength is crucial for the IC industry [1–3].

Electroforming has been used in fabrication of a variety of microstructures with high aspect ratio and precision [4]. Among the metallic alloys, nickel based one is most frequently employed in the industry because of its superior

mechanical properties and chemical stability [5]. For the fabrication of microprobe card, nickel alloys are more preferred than their monolithic nickel metal due to the improved strength and toughness [6]. For microstructure, the internal stress of the electroforming must be well controlled in order to avoid the distortion of the released structure and/or the peel-off during deposition process. Moreover, the cathodic efficiency must be maintained at suitable level to reduce excess generation of hydrogen bubbles which, if trapped on the cathode surface, could interfere with the local metal deposition process. Although the addition of wetting agent in the electrolyte can alleviate the problem associated with the hydrogen bubbles, the unavoidable inclusion of the organic chemical into the deposition usually causes the deterioration in its structural toughness [7]. Furthermore, for the special application in probe card, the potential alloy must not have the sticking problem with the material it tests. The material transfer due to adherence upon contact either from the solder

TABLE 1: Compositions of electrolyte and the associated process parameters.

Ingredient	Concentration (g/L)
Nickel sulphamate	514 mL/L (Ni^{2+} : 90 g/L)
$\text{MnSO}_4 \cdot 5\text{H}_2\text{O}$	0~175.5 (Mn^{2+} : 0~40 g/L)
$\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$	3
H_3BO_3	40
Wetting agent (sodium dodecyl sulfonate)	1.8 mL/L
Process parameters	
Temperature	50°C
Current density	0.2–12 A/dm ²
Agitation	Magnetic stirrer, 200 rpm

bump to probe tip or vice versa deteriorates the test object and the probe itself.

Ni-Mn alloy has superior mechanical strength and hardness and has been successfully used in preparation of microsystem devices, such as microgear and contact spring [8, 9]. Therefore, it serves as a potential candidate for the fabrication of microprobe structure. However, the properties of the electrodeposited structure depend on the composition and material microstructure [6, 10–14], which in turn are controlled by the parameters of electrodeposition process, such as electrolyte compositions and current density. Besides, the internal stress of the electrodeposition must also be brought under acceptable level. Although there are some detailed studies of the Ni-Mn alloy coating in the previously mentioned literature, the discussion on the control of the internal stress is still required for this application. In addition, the control of Mn content in the deposited alloy with the plating parameters is still not fully discussed, especially for the application in microprobe structure at higher current density. Furthermore, the special requirement for nonsticking property with most solder bumps and connecting pads was not reported. It is, thus, the goal of this study to investigate the plausible fabrication process of Ni-Mn microprobe structure with high strength and low internal stress by controlling the electrolyte bath and current density in the electrodeposition.

2. Experiment

2.1. Electrodeposition. The effect of the process parameters controlled in this study included the concentration of Mn^{2+} and current density. As presented in Table 1, a nickel sulphamate bath was employed in this study in which the concentration of manganese sulphate was adjusted within the range of 0–40 g/L. Manganese sulphate provided the source of Mn ions for the electrolyte. A copper substrate was used as the cathode, while S-round nickel pallets enclosed in titanium net and nonwoven bag served as the anode. The electrodes were placed in vertical configuration inside a 1-l glass beaker. A magnetic stirrer rotated in 200 rpm drove the circulation of electrolyte and the bath temperature was controlled with a hot plate beneath the beaker. During electrodeposition, a DC power supply provided a constant electric current and

the current density was controlled to prepare the specimens at a range of 0.2–12 A/dm².

2.2. Composition, Morphology, and Microstructure Examination. The quantitative composition analysis of the coating was measured by using Electron Probe X-ray Microanalyzer (EPMA, JEOL JXA-8200). Based on the measured compositions, the cathodic efficiency of the electrodeposition was calculated by using Faraday's law.

The surface morphology of the coating was examined through the use of a scanning electron microscope (SEM, JEOL-JSM-7401F). Regarding the crystalline structure and texture of the coating, an X-ray diffractometer (XRD, Philips 1830/Mac) was employed at a sweeping rate of 2 deg/min. The X-ray was generated by a $\text{Cu-K}\alpha$ target operated at 30 kV and 20 mA with a wavelength of 0.15418 nm. With the measured diffraction spectrum, the associated grain size was calculated by using Scherrer equation. As for the microstructural examination over the cross-section of the coating, the specimen was prepared through mechanical polishing and ion-beam thinning and examined with a transmission electron microscope (TEM, FEI Tecnai G2 T20).

2.3. Hardness and Internal Stress Measurement. The hardness of the coating was measured on the cross-section of the resin-mounted specimen after polishing preparation. The microhardness measurement was performed at six different points of each sample using a diamond pyramid indenter, the Mitutoyo HM-113 microhardness tester, at a load of 50 g and loading duration of 15 s. The internal stress of the coating prepared under various electrode positions with the controlled process parameters was measured by a deposit stress analyzer (Model 683, Specialty Testing & Development Co.). A 3 μm thick coating was deposited on the copper test strip (PN 1194, Specialty Testing & Development Co.) and the internal stress was measured accordingly.

2.4. Sticking Test. The sticking test of the Ni-Mn coating was carried out in a bath of molten tin. The electrodeposited specimen was dipped into the molten tin bath for 3 s and the coating surface was examined with naked eyes for the residue of the attached tin. With the estimated area percentage of the nonstick surface, the antisticking property of the Ni-Mn coating was quantitatively determined.

3. Results and Discussion

3.1. The Effect of Mn^{2+} Concentration in the Electrolyte. For the nickel sulphamate electrolyte containing 0–40 g/L Mn^{2+} ion, the pH of the bath decreased from 3.5 to 3.0, as shown in Figure 1. Under the fixed current density of 8 A/dm², the current efficiency, Mn content, and the internal stress of the coating prepared in the bath with different Mn^{2+} concentrations are also presented in Figure 1. As seen in the result, the Mn content rose quickly when the Mn^{2+} concentration in the electrolyte was first increased. However, for the Mn^{2+} concentration was higher than 10 g/L, the Mn content in

FIGURE 1: The properties of Ni-Mn coating prepared in electrolyte with varying Mn^{2+} concentration but constant current density of 8 A/dm^2 .

FIGURE 2: The effect of current density on the current efficiency of the deposition and Mn content of the coating.

the obtained coating remained nearly unchanged around 1 wt%. In similar trend with the result of Mn content, the internal stress increased quickly with the initial addition of Mn^{2+} ion, reached a maximum at 5 g/L of Mn^{2+} in the electrolyte, and then gradually decreased at higher Mn^{2+} concentrations. In addition, there were serious cracks developed in the coating electrodeposited with Mn^{2+} concentration higher than 15 g/L. On the other hand, the current efficiency of the electrodeposition monotonically decreased with the raise of Mn^{2+} concentration. Quicker descent in current efficiency was observed when the Mn^{2+} concentration reached 30 g/L.

3.2. The Effects of Current Density. The results in Figure 1 revealed that the raise of Mn^{2+} concentration in the electrolyte higher than 10 g/L had little improvement on the Mn content in the coating. Hence, for investigating the effect of current density, the Mn^{2+} concentration in the electrolyte was fixed at 10 g/L. Figure 2 presents the current efficiency and

FIGURE 3: The influence of current density at electrodeposition on the grain size, internal stress, and hardness of the coating.

Mn content in the coatings prepared with current density increased from 0.2 to 12 A/dm^2 . It was seen that the current density could effectively control the Mn content in the coating and raising the current density increased the Mn content. Nevertheless, the overpotential became higher as the current density was rigorously increased. The high overpotential rendered the electrodeposition with more hydrogen reduction. Therefore, the current efficiency was dropped accordingly at high current density.

Along with the change in its composition, the microstructure of the coating was also influenced by the current density. Figure 3 shows the variations of grain size, internal stress, and hardness with respect to the current density. Basically, the grain size of the coating reached a maximum at intermediate current density around 4 A/dm^2 . Then, the grain size decreased with increasing current density. Regarding the internal stress, the coating was deposited in compressive internal stress at low current density. The internal stress changed from compressive to tensile mode and raised its magnitude as the current density increased. At high current density, the internal stress became very high such that cracks developed in the coating. This highly developed cracking relieved slightly the internal stress at 12 A/dm^2 . From this measurement result, it was possible to conceive that a coating with nearly no internal stress can be obtained by suitable control of the current density.

Also shown in Figure 3 is the variation of the coating's hardness with respect to the current density employed in the electrodeposition. From the aforementioned changes in microstructure and composition of the coating at different current densities, it is an instinctive suspect that the hardness of the coating may alter accordingly. Overall speaking, the hardness increased with the current density. With the alloying of Mn in nickel substrate, the hardness was effectively raised from below 300 to above 600 kg/mm^2 . This strengthening should be ascribed to the finer grain, higher Mn content, and more crystalline defects in the microstructure. In spite of the effective increase in the hardness by electrodeposition at high current density, the corresponding high level in internal stress, as seen in Figure 3, would cause serious distortion in

FIGURE 4: The cross-sectional micrographs of TEM examinations on the coatings prepared at different current densities: (a) 2 A/dm², (b) 8 A/dm², (c) 10 A/dm², and (d) 12 A/dm². Single arrow denotes the direction of columnar grain, while double arrows indicate the presence of twins. The corresponding insets are the SAED patterns measured at location of pointed arrows.

structural dimensions, not to mention the possible cracking that threatens the integrity of the structure. Fortunately, the singular jump in hardness for coating prepared at 1 A/dm² was worth noting. Although the corresponding Mn content was not high, a significant grain refinement was seen from Figure 3. This grain refinement should be responsible for the abrupt hardness improvement. Because of this hardness increment and the associated low internal stress characteristics, the electrodeposition of the Ni-Mn alloy coating under this controlled process became a viable technique for its application in microstructure fabrication.

The photomicrographs in Figure 4 present the TEM examinations on the cross-section of the coatings prepared at different current densities. Columnar grains were clearly observed at low current densities such as 2 and 8 A/dm². The selected area electron diffraction (SAED) pattern as seen in the inset of the figure confirmed the coating's FCC columnar microstructure. At 8 A/dm², twins were observed inside the columnar grain. This should be related to the lattice distortion caused by the increased incorporation of Mn in the coating. The twin plane was in 45° angle with the axis of the columnar grain. Moreover, the SAED confirmed the twin belongs to [110] zone axis and the extra diffraction points were due to the twin defect. A change from columnar grain of 200 nm in width to the more refined columnar ones was observed as the current density further increased, accompanied with the increase in Mn content. Figures 4(c) and 4(d) depict the fine

FIGURE 5: The photograph of specimen after being dipped in molten tin for testing its nonsticking characteristic.

columnar grain structures at 10 and 12 A/dm², respectively. The spacing between the continuous circular rings of the SAED measurement in the insets verified the lattice still was FCC crystal of nickel.

3.3. Nonsticking Characteristics of Ni-Mn Alloy. Since one of the proposed applications of the Ni-Mn alloy was in the fabrication of probe in microelectronic testing, the nonstick characteristic of the probe material must be assured. Figure 5 is a picture showing the surface of the Ni-Mn coating which had been dipped in the molten tin bath. There was less than 30% of area covered with residual tin and most of them were around the edges with contingent contact of brass

FIGURE 6: The fabricated probe structure of Ni-Mn alloy: (a) dimensions of the probe, (b) the enlarged view of the electrodeposited probe before being released from the pattern.

substrate. The central portion of the coating was basically free of tin residue. The brass substrate, on the other hand, had nearly 80% of area covered with tin residue. Therefore, the antisticking characteristic of the prepared Ni-Mn coating with respect to tin and its alloys should be satisfactory.

3.4. Fabrication and Testing of Probe Structure. Based on the results of the parameter study on the electrodeposition of Ni-Mn alloy, the microprobe was fabricated in a prepared micropattern under 1 A/dm^2 . Figure 6(a) shows the dimensions of a single probe and insets are the magnified photographs near its two ends. The further locally enlarged view of the probes before they were released from the electrodeposited pattern is presented in Figure 6(b). The thickness of the probe was $50\text{ }\mu\text{m}$. The quality of the fabricated probe was satisfactory both in surface finish and dimensional conformity. However, the performance of the probe needed to be further examined. Therefore, the probe was installed and tested in a fatigue test stand as depicted in Figure 7. As shown in the schematic diagram of Figure 7, the upper end of the probe was installed loosely inside a cylindrical hole at upper part of the fixture, while the lower end was constrained inside another vertical cylindrical hole for providing the vertical and lateral support. The vertical movement of the rhodium plate, which contacted the upper end of the probe during testing, was controlled for a specified overdrive displacement by a vertical platform, not shown in the figure. In the meantime, the contact loading for the overdrive displacement was measured by the load cell above the rhodium plate. During

FIGURE 7: The schematic diagram of the test setup for probe fatigue life.

the contact stroke, the contact resistance was calculated from the current supply and the associated voltage.

The contact and retreat strokes were executed continuously for life cycle testing. The overdrive of the probe was fixed at 0.1 mm and the data were recorded every ten cycles. Figure 8(a) presents a typical measured result over the 0.1 mm overdrive stroke. With the downward movement of the rhodium plate and the contact with the probe upper end, the probe force started increasing while the contact resistance decreasing. When the overdrive was larger than $40\text{ }\mu\text{m}$, the contact resistance dropped to a value around $1000\text{ m}\Omega$ and probe force maintained larger than 3 g_f for overdrive reaching 0.1 mm . The probe force and contact resistance at the end of the 0.1 mm stroke were recorded each 1×10^5 cycle and were shown in Figure 8(b). After 5×10^5 cycles, the probe still functions properly. In other words, the fatigue life of the fabricated probe was larger than half a million cycles.

4. Conclusion

The Ni-Mn alloy electrodeposited from a nickel sulphate electrolyte with the addition of manganese sulphate and under DC current density was examined microscopically. Although the codeposited Mn content in the coating increased, which boosted the hardness of the coating, with the raise of Mn^{2+} concentration in the bath and the applied current density, the cracking associated with the overgrown internal stress made the process infeasible in high current density. The grown internal stress with the Mn content of the coating was due to the transition of the crystalline morphology from large columnar grains to finer ones, which, on the other hand, increased the hardness. The prepared probe structure in 1 A/dm^2 current density showed low internal stress and moderate improvement in hardness over its pure nickel counterpart. Moreover, the life and elastic property of the thus prepared Ni-Mn probe structure was

FIGURE 8: (a) The typical probe force and contact resistance curves with respect to the overdrive stroke during cyclic testing; (b) the performances of the probe after selected number of testing cycles.

proved satisfactory. These results can provide a feasible electrodeposition system with its associated process parameters to engineers in the microelectronic industry.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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