Paper

Evaluation of the Ni Diffusion to the Surface of Au Plating for Soldering Process Control

Midori Takano
Corporate Quality Innovation Center Panasonic Electronic Devices Co.,Ltd., 1006, Kadoma, kadoma, Osaka, 571-8506
takano.midori@jp.panasonic.com

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One of the factors inhibiting the soldering on an Au electrode is that the underlying metal diffuses onto the surface of Au electrodes and an oxide layer is formed. In order to prevent any faults, the evaluation method of the underlying metal diffusion was examined. Usually, surface analytical techniques such as Auger electron spectroscopy and X-ray photoelectron spectroscopy are used for the evaluation of the underlying metal diffusion; however, a more rapid method is required for process management. We examined the measurement of the diffused underlying metal by energy dispersive X-ray spectroscope equipped to a low voltage scanning electron microscopy (SEM-EDX). It is possible to evaluate the underlying metal diffusion without detecting the underlayer signal by using SEM-EDX. It is suggested that measurements of the diffused underlying metal with SEM-EDX can be applied to the process management.

1. Introduction

Recently, electronic products have become small more and more, applying to mobile utility with high-performance. Therefore, high integration reliability and strong impact resistance for electronic devices have become much important. Under these circumstances, improvement of solder joints reliability is an important factor for electronic products.

A poor solder wetting is one of the solder joint failure modes. It is caused by a soldering area surface contamination and/or an oxidized layer formed by metals diffused from underlayer of an electrode for soldering [1]. A soldering reliability would be improved by eliminating poor wetting by surface analysis for soldering areas. In this report, we describe the evaluation of Ni diffusion to the Au plated electrode surface which obstructs soldering.

2. Experimental

We prepared the approximately 90 nm thick Au electrodes deposited on Ni layer by using electron beam deposition. We used the X-ray photoelectron spectroscopy (XPS) and the scanning electron microscope equipped with the energy dispersive X-ray spectrometry (SEM-EDX) for the failure analysis. The XPS spectra were acquired using monochromatic AlKα X-rays (1486.6 eV) with a charge neutralizer (Quantera SXM, ULVAC-PHI INC.). The analysis area was about 20 μm in diameter. The SEM-EDX spectra were acquired at the acceleration voltages of 3 kV and 5 kV (ULTRA55, Carl Zeiss, equipped with INCA Energy 450, Oxford).

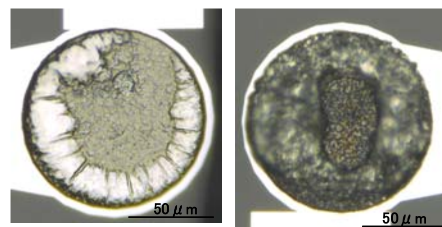

Fig.1 Optical micrographs of poor solder wetting electrodes.

3. Investigation of a poor solder wetting

Fig.1 shows the optical micrographs of the disassembled electrodes of the soldering failure samples. The solder do not wet at the center. By empirical knowledge on failure analysis, we can suspect that the causes are foreign particles, contaminations, and/or Ni oxide film formation on the surface.

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XPS measurements were performed on two Au electrode samples before soldering. One sample was taken from the solder wetting defective production lot (NG-lot sample), the other was from the normal production lot (OK-lot sample). Fig.2 shows the XPS survey spectra of the two sample surfaces. Ni and O peak intensities of the NG-lot sample are stronger than those of the OK-lot sample. Fig.3 shows Ni 2p spectrum of the NG-lot sample. The peak at 855 eV in binding energy is attributed to Ni oxide. Fig.4 shows the depth profile of NG-lot sample. In this depth profile, Ni and O layer are on top of the Au layer. These results indicate that Ni oxide film is formed on the Au electrode of the NG-lot sample. Accordingly, the cause of the poor solder wetting is concluded to be the diffusion of the Ni to the Au electrode surface from the underlayer, and the formation of the Ni oxide film as illustrated in Fig.5.

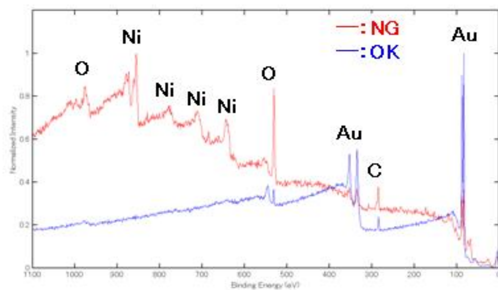

Fig.2 XPS survey scan spectra of NG-lot sample and OK-lot sample.

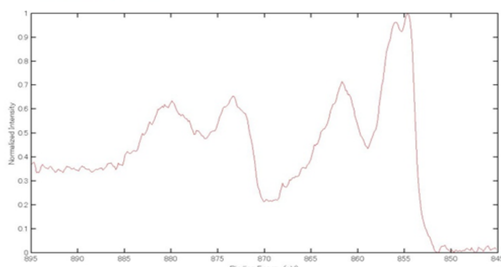

Fig.3 Ni2p spectrum of the NG-lot sample.

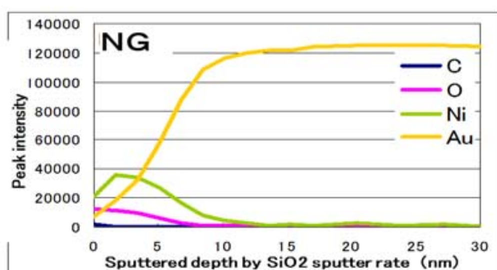

Fig.4 Depth profile of the NG-lot sample.

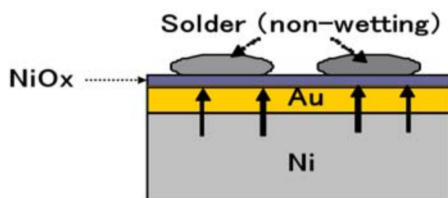

Fig.5 Ni atoms diffuse through the Au electrode and they form Ni oxide layer.

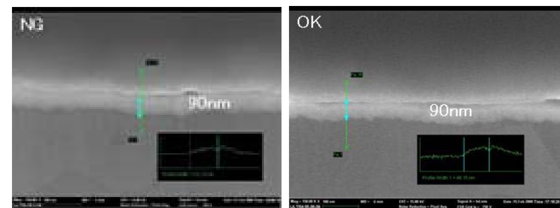

Fig.6 The cross-section SEM images.

In order to explain Ni diffusion onto the Au surface and the oxide layer formation on the NG-lot sample, the two reasons were considered. The first, it was that the Au film thickness might be thinner than the specific value due to an abnormality in the deposition process. The second, the Au deposition film of NG lot might be exposed higher temperature allowing Ni diffusion.

The thicknesses of the Au film of NG-lot and OK-lot samples were estimated by cross-section view of SEM. The cross-section of the electrodes was processed with focused ion beam. Fig.6 shows SEM images of the cross-section. The bright parts, middle of the image, are Au films. The Au film thicknesses of these samples are almost same, about 90 nm. There are no differences between NG-lot sample and OK-lot sample.

For evaluating the second reason, XPS measurements were conducted for the four processes after Au film deposition, namely processes A, B, C, and D, in order to determine surface atomic compositions. From XPS quantification results, the atomic concentration ratios of Ni to Au of the electrode were calculated and plotted against the process as shown in Fig.7. The solid, dashed, dotted lines correspond to the samples in process D from equipment 1, from equipment 2, and from equipment 2 for short time (0.75 times to the other sample), respectively. Equipments 1 and 2 carried out the same process treatment but

equipment 2 started in operation later. The ratio of Ni to Au determined by XPS (Ni/Au $_{XPS}$) corresponding to the amount of Ni diffused to the surface of Au, tends to increase in process D. Particularly, the solid line showed drastic increase which indicates that the Ni atoms diffuse into Au and segregated on the Au surface using equipment 1 in process D. The occurrence of poor solder wetting using equipment 1 is higher than those of others, which suggests that the ratio of Au to Ni on the electrode surface are correlated with the poor solder wetting. Additionally, the ratio of Au to Ni decreases by shortening the treatment time in process D (dashed line and dotted line).

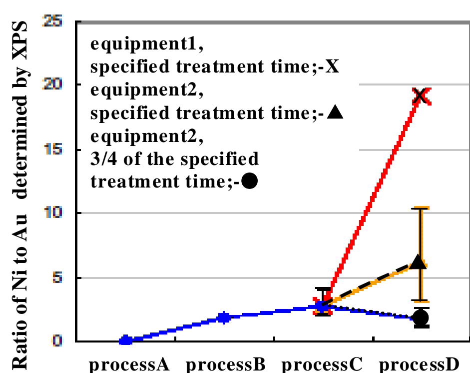

Fig.7 Atomic concentration ratio Ni/Au of the electrodes.

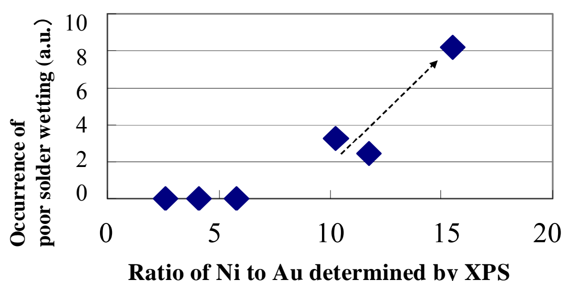

Fig.8 Relation of the atomic concentration ratio Ni/Au and the occurrence of poor solder-wetting.

For obtaining different occurrence of poor solder wetting, the electrode forming process was changed, then the atomic composition of prepared samples were evaluated with XPS. Fig.8 shows the relation of (Ni/Au $_{XPS}$) and the occurrence of poor solder wetting. When (Ni/Au $_{XPS}$) is higher than about 10, the occurrence of poor solder wetting increases. Thus, the high occurrence lot of poor solder wetting can be monitored by measuring (Ni/Au $_{XPS}$) of production lot, which can also be applied to the lot process control.

These results mean found that Ni diffusion to the Au surface is promoted in process D, especially in the treatment with equipment 1, it results more frequent poor solder wetting than that with equipment 2. Therefore, we introduced two countermeasures in process D. (1) Review of the treatment condition with equipment 1. (2) Introduction of the evaluation of Ni diffusion to the Au surface (the Ni/Au ratio) after process D as for the lot process control.

4. Study the process control method

Introducing the evaluation of Ni diffusion to the Au surface as for the lot process control, the requirements of evaluation method are as follows.

(a) The Ni under layer should not be detected, but should be detected only the Ni on Au, and Au.

(b) The measurement should be as fast and easy as possible.

A time for XPS measurement is not fast enough because XPS measurement is performed under ultra-high vacuum analysis, which may be difficult to apply to the lot process control. Therefore, we performed SEM-EDX analysis. The information depth of SEM-EDX analysis is determined by the electron penetration depth that varies with the acceleration voltage of an electron beam. From following reasons, the measurement requires low acceleration voltage such as few kV.

(1) The information depth should be few tens nm for satisfying the previous mentioned requirement (a).

(2) The acceleration voltage can excite NiLα line (0.85 keV) and AuMα line (2.12 keV) at more than 3 kV. We estimated the electron penetration depths into the sample by Monte Carlo simulation. The model sample structure was a 90 nm thick Au film on Ni substrate. The density of the Au film was assumed to be that of bulky Au. Fig.9 shows the result.

It is suspected that the suitable range of acceleration voltage is 3 to 5 kV. Analysis of NG-lot samples and OK-lot samples were performed using SEM-EDX. The spectral analysis was performed at the acceleration voltages of 3 and 5 kV. Fig.10 shows the SEM-EDX spectra. The aperture diameter of electron gun was 60 μm and the measurement time was 60 seconds. Almost equal intensities of the Ni peak are

obtained in both NG-lot samples and OK-lot samples. A clear difference is not observed between NG-lot and OK-lot at the acceleration voltage of 5 kV. On the other hand, at the acceleration voltage of 3 kV, strong peaks of Ni and O are detected from NG-lot samples; but very weak peaks of Ni and O are detected from OK-lot samples.

The electron penetration depths at the acceleration voltage of 3 kV, 5 kV are approximately 30 nm and 90 nm, respectively and the electron penetration depths at 5 kV is similar to the Au film thickness. However, the actual sample was a deposited film, suggesting that the deposited film density was smaller than the bulk density. Therefore, it is considered that the Ni underlayer is detected at the acceleration voltage of 5 kV, and that Ni on the Au film is only detected at the acceleration voltage of 3 kV. Fig.11 shows the observed SEM-EDX spectra at 3 kV acceleration voltage with 30 μm aperture diameter. This aperture parameter is the same that is used for SEM observation. The measurement time was 60 seconds. Compared to the spectra of Fig.10, the sensitivity is much lower, which explain the difficulty to detect the small amount of Ni.

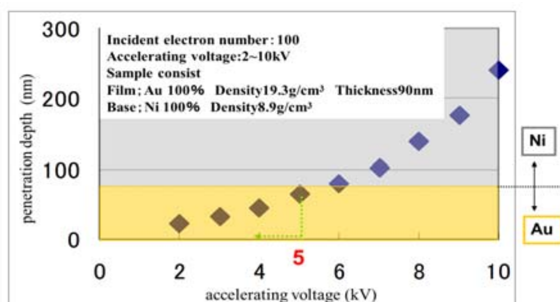

Fig.9 Electron penetration depth into the sample by Monte Carlo simulation at an acceleration voltage of 2-10 kV.

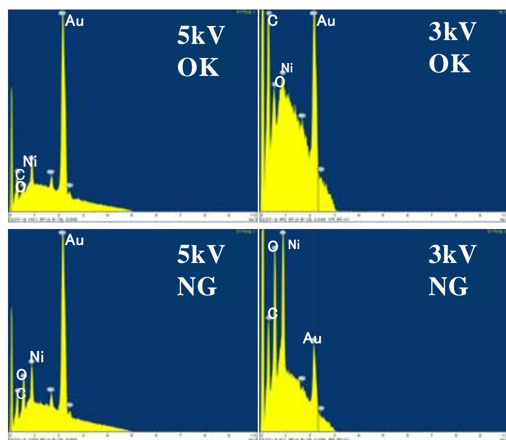

Fig.10 SEM-EDX measurement spectrum at the acceleration voltage of 3 kV, 5 kV with 60 μm aperture diameter.

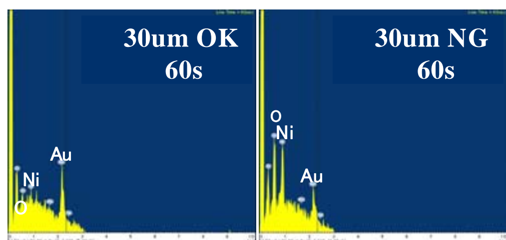

Fig.11 SEM-EDX measurement spectrum at the acceleration voltage of 3 kV with 30 μm aperture diameter.

From these results, the measurement condition was determined as the acceleration voltage 3 kV, the aperture diameter 60 μm, and the measurement time 60 seconds. In these experimental conditions, the results of SEM-EDX showed the occurrence of poor solder wetting tends to increase when (Ni/Au $_{XPS}$) is higher than about 10. If there is a correlation between the intensity ratio Ni/Au with SEM-EDX (Ni/Au $_{SEM-EDX}$) and (Ni/Au $_{XPS}$), the evaluation of Ni diffusion to the Au surface with SEM-EDX can be applied to the process control.

Then, some samples that were picked up from the lots showing the occurrence of poor solder wetting were analyzed by both XPS and SEM-EDX. Fig.12 shows (Ni/Au $_{SEM-EDX}$) as a function of (Ni/Au $_{XPS}$) which suggested the correlation between (Ni/Au $_{SEM-EDX}$) and (Ni/Au $_{XPS}$). When (Ni/Au $_{XPS}$) is higher than 10, (Ni/Au $_{SEM-EDX}$) is higher than about 1.5. Those results suggest that the evaluation of Ni diffusion to the Au surface is possible by using with SEM-EDX at low acceleration voltage

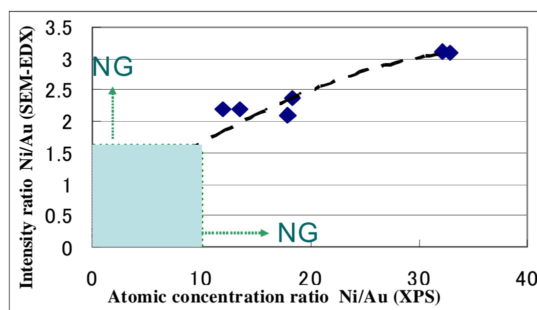

Fig.12 Correlation between (Ni/Au $_{SEM-EDX}$) and (Ni/Au $_{XPS}$).

The mass production trial samples were evaluated by SEM-EDX at the measurement condition described for fig.12. The samples were chips cut out from the wafer, which had several Au electrodes. Nine wafers

picked up from a film deposition lot, were evaluated using the following tests.

Test1: The variation among electrodes on a chip (The dependence on position in a chip)

Test2: The variation among chips in a wafer (The dependence on position in a wafer)

Test3: The variation among wafers in a film deposition lot

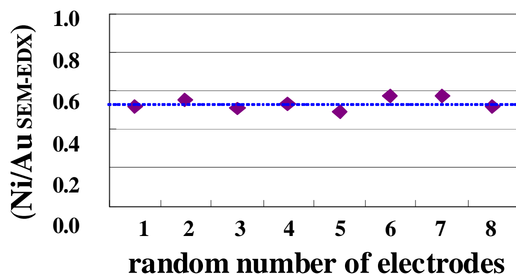

Fig.13 The variation among electrodes in a chip.

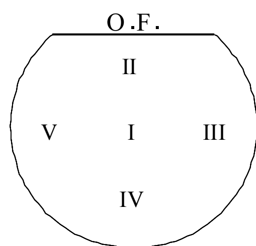

Fig.14 The sampling position in a wafer. O.F. is the orientation flat part.

Fig.13 shows the (Ni/Au $_{SEM-EDX}$) by the random number of electrodes (Test1). The mean value of (Ni/Au $_{SEM-EDX}$) was 0.53 and the standard deviation σ was 0.032. All of the measured values of the electrodes in a chip were in ±3σ, therefore, it suggested that (Ni/Au $_{SEM-EDX}$) was independent of the electrode position in a chip. Hence, test2 was carried out with the electrode at the center position in a chip.

On the test2, five chip samples were picked up from five points of a wafer as shown in Fig.14, and three wafers were evaluated. Fig.15 shows the mean value of (Ni/Au $_{SEM-EDX}$) of 3 wafers measured at each position (I to V). The mean value of (Ni/Au $_{SEM-EDX}$) for all positions was 0.59, and the standard deviation σ was 0.075. (Ni/Au $_{SEM-EDX}$) at position I (center position) tended to be lower than those at position II to V (peripheral position), however, all the measured values were in ±3σ.

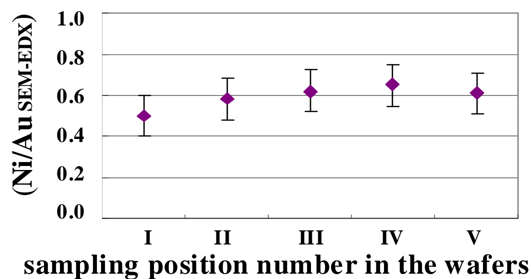

Fig.15 The variation among chips in a wafer.

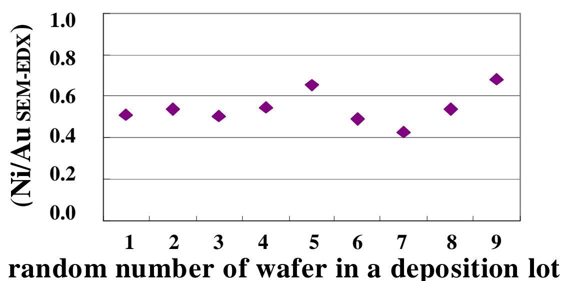

Fig.16 The variation among wafers in a film deposition lot.

On the test3, chip samples were picked up from the center position (position I) of 9 wafers in a deposition lot. Fig.16 shows the (Ni/Au $_{SEM-EDX}$) by the random- wafer- number in a deposition lot. The mean value of (Ni/Au $_{SEM-EDX}$) was 0.54, and the standard deviation σ was 0.079. In a deposition lot, all measurement values were within ±3σ.

5. Conclusion

We showed that the cause of the electrode poor solder wetting was Ni oxide film formation on the Au surface by XPS analysis. It is convinced that the poor soldering on the Au deposited electrode can be judged from the electrode surface evaluation by the random wafer measuring from the production lots by low acceleration voltage SEM-EDX. Considering the measurement error and preventing judgment error, the judgment criteria of (Ni/Au $_{SEM-EDX}$) set to higher than 1.0.

Low acceleration voltage SEM-EDX can be applied to the evaluation of Ni diffusion to the electrode Au surface, as for process control method.

6. Reference

[1] Hideyuki Sanpei, *Journal of The Surface Finishing Society of Japan*, **58**, 219 (2007).