Influence of Noble Metal Coating on Wettability of Copper Substrate by Sn-Ag Eutectic Solder

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The influence of thin coatings (70 nm) of Au, Ag and Pd on the wettability of a Cu substrate by a Sn-3.5Ag eutectic solder was investigated using the contact angle measuring system combined with meniscograph tester. The wettability of the Cu substrate with Sn-3.5Ag was improved by the Au and Ag coatings, while reduced by a Pd coating, especially in terms of the contact angle. Namely, the contact angles were 29–30° on the Au coated-Cu substrate, 34–35° on the Ag coated-Cu substrate, 49–52° on the Pd coated-Cu substrate and 42–45° on the uncoated Cu substrate. On the wetted area, each coating layer dissolved into the solder and the Cu-Sn intermetallic compound was formed at the solder/substrate interface as well as on the uncoated Cu substrate, but the coating metal layers remained on the unwetted area. The difference in wettability of the noble metal coating on the Cu substrate was found to be attributed to the difference in the substrate-flux interfacial tensions (γ_{sf}).

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1. Introduction

From the viewpoint of global environmental preservation, the development of solders without containing Pb as a hazardous substance, the so-called Pb-free solders, has been conducted on a worldwide basis.¹⁾ Much insecurity about the mechanical and electrical reliability of Pb-free soldered connections remains, because the wettability of Pb-free solder is quite inferior to that of a Sn-Pb eutectic solder.^{2–5)} While Pb-free coating materials, for example, Au/Ni, Pd/Ni, Sn, Sn-Ag and Sn-Bi, on the electrode surface of a printed wiring board (PWB) and electronic device have been investigated,^{6–9)} the wettability of the Pb-free coating material on the electrode surface by Pb-free solder has not been sufficiently clarified.

The meniscograph method is one of the most useful methods to evaluate solder wettability. The wetting force can be measured as a product of the contact angle (θ) and the molten solder-flux interfacial tension (γ_{lf}), which are physical parameters of wettability. However, it is difficult to measure both θ and γ_{lf} separately by a conventional equipment. In the conventional method, the maximum wetting force, wetting time (between the time immersion starts and that when the force on a substrate returns to zero) and wetting rate, all of which are obtained from the wetting force-time curve, are used only as wettability indices.¹⁰⁾ Unfortunately, these indices are easily affected by the substrate size and shape, so that wettability cannot be quantitatively evaluated but only qualitatively.

We have developed a new system combined with an instrument to directly measure θ during the solder wetting process.^{11,12)} In this system, the contact angle (θ), interfacial tension (γ) and the wettability indices in the meniscograph method can be measured simultaneously. The principle for measuring the contact angle utilizes the regular reflection characteristic of the molten solder surface which possesses a specular gloss. Consequently, the direction angle of the solder fillet surface can be measured without contact.

In this study, using this newly developed system,^{11,12)} the

influence of the coating noble metals, Au, Ag, and Pd, on the wettability of a Cu substrate with the Sn-Ag eutectic solder was investigated on the basis of physical parameters, *i.e.*, the contact angle (θ) and interfacial tensions (γ) between the solder, flux and substrate.

2. Experimental

2.1 Contact angle measurement

A schematic illustration of the meniscograph method is shown in Fig. 1. The force-time curve during wetting of a substrate by the solder, the so-called wetting curve, is shown in Fig. 1(a). The t_0 , t_1 , t_2 and t_3 are times when the substrate contacts the solder surface ($t = 0$ s), the solder begins to wet the substrate, the force on the substrate returns to zero and the wetting becomes stable (the wetting force almost reaches a constant value), respectively.

Figure 1(b) shows the relationship among the wetting force (F), the solder-flux interfacial tension (γ_{lf}) and contact angle (θ). When F and θ are measured, γ_{lf} can be immediately obtained from eq. (1).¹³⁾

$$F = P \times \gamma_{lf} \times \cos \theta - B \quad (1)$$

where P is the perimeter of the substrate and B is the buoyancy force, both of which are known to be constants. Thus, the solder wettability can be analyzed from the viewpoint of interfacial tension.

Figure 2 shows the construction of the contact angle and interfacial tension measuring system combined with the meniscograph method. The principle of the contact angle measurement utilizes the regular reflection characteristic of the molten solder surface which possesses a specular gloss. The multiple light sources with a distinctive brightness simultaneously irradiate the molten solder surface, and the reflection is recorded with a CCD camera. Subsequently, the reflection on each pixel received by the CCD camera is related to the position of the light source by using the regular reflection characteristic at the molten solder fillet surface. Consequently, the direction angle of the solder fillet surface

Fig. 1 Schematic illustration of meniscograph method; (a) wetting curve and (b) vertical schematic illustration of the substrate immersed in the molten solder.

Fig. 2 Construction of the contact angle and surface tension measuring system for soldering.

can be measured without contact.

The following three techniques are developed in order to achieve a high speed (15 frame/s) and highly accurate ($\pm 0.2^\circ$) measurement.

1) The first technique is that, in order to improve the angular resolution of this equipment, multiple light sources (37 LEDs) are accurately arranged at an interval of 2.2° so as to uniformly irradiate the solder fillet surface as shown in Fig. 2. The contact angle can be measured in the range of $4\text{--}71^\circ$ by changing the position of the CCD camera. The LED devices emitting near-infrared light (wavelength: 880 nm) were selected in order to suppress the influence of visible light.

2) The second technique uses brightness-ratio-coded light

Fig. 3 Schematic illustration of the brightness-ratio-coded method; (a) lighting pattern A (brightness: $La1 > La2 > La3$) and (b) lighting pattern B (brightness: $Lb1 < Lb2 < Lb3$).

sources, as shown in Fig. 3. This allows us to specify the position of the light source from the ratio of the reflection intensity obtained under two different flashing patterns. As can be seen in eq. (2), by reduction of the reflection rate, the ratio of the reflection intensity has to equal that of the light source brightness which is a known constant.

$$Ia/Ib = (La1 \times r)/(Lb1 \times r) = La1/Lb1 \quad (2)$$

where Ia and Ib are the reflection intensities, La1 and Lb1 are the brightness of the light sources, and r is the reflection rate of the solder surface. Therefore, by measuring the ratio of the reflection intensity, the position of the light source, hence, the direction angle (ϕ) of the solder fillet surface can be measured.

3) The third technique is the enlargement of the dynamic range of the CCD camera by synthesizing two pictures obtained under two different exposure times, which allows one to record the subject with a wide range of contrast such as the molten solder surface.¹⁴⁾

By continuously taking four pictures at fixed intervals (1/15 s) during the solder wetting test, the distribution data of the direction angle of the solder fillet surface were obtained, followed by measuring the contact angle and interfacial tension. The performance of the contact angle measuring instrument is shown in Table 1.

2.2 Wettability evaluation and analysis

Table 2 shows the soldering test conditions. The composition of the solder was 96.5 mass%Sn-3.5 mass%Ag (described as Sn-Ag hereafter). As the substrate, Cu (described as Au/Cu hereafter), Ag-coated Cu (described as Ag/Cu hereafter), and Pd-coated Cu (described as Pd/Cu hereafter) plates were used. The size of substrate was $10 \times 30 \times 0.3 \text{ mm}^3$, and each noble metal coating of 70 nm in thickness was deposited on the Cu plate by sputtering. Before the wettability test and coating with the noble metal, the Cu plate surface was cleaned by electrolytic polishing, followed by etching with 1 M HCl, washed in ion-exchange

Table 1 Performance of contact angle measuring instrument.

Sampling area (mm ²)	3.16(X) × 2.45(Y)
Resolution (pixel)	640(H) × 240(V)
Measured angle range* ¹	①4–42°, ②18–56°, ③33–71°
Accuracy of angle* ²	±0.2°
Sampling rate (frames max./sec)	15

*1: It can be selected by the setting angle of CCD camera on a horizontal level.

*2: Maximum fluctuation of one pixel is ±2°.

Table 2 Soldering test conditions.

Solder	96.5 mass%Sn-3.5 mass%Ag
Temperature	250, 270, 290°C
Substrate	Cu, Au, Ag, Pd
Coating metal on Cu substrate	Au, Ag, Pd (thickness: 70 nm)
Flux	PO-Z-7 (halogen-free)
Atmosphere	Air
Immersion time	10 s
Immersion depth	2 mm
Immersion rate	±10 mm/s
Repetition	3

water and then air dried. In addition, the wettabilities of pure Au (99.99%), Ag (99.99%) and Pd (99.95%) substrates, having a size of 10 × 30 × 0.3 mm³, were also investigated for the comparison with those of the Au/Cu, Ag/Cu and Pd/Cu substrates. The soldering temperature was measured at a depth of 3 mm from the surface of the molten solder alloy with a thermocouple.

The contact angle (θ) and wetting force (F) were measured 5 s after the immersion began. In this study, the wetting time (t_2 in Fig. 1(a)), maximum wetting force ($F_{\max}$) and contact angle (θ) were used to evaluate the wettability.

Figure 4 is a schematic illustration of the wetting. The contact angle is determined as a result of the balance of three interfacial tensions at the wetting tip of the solder, *i.e.*, the solder-flux (γ_{lf}), substrate-flux (γ_{sf}) and substrate-solder interfacial tension (γ_{sl}). This relationship¹⁵⁾ is described by Young's eq. (3).

$$\gamma_{sf} = \gamma_{lf} \times \cos \theta + \gamma_{sl} \quad (3)$$

The solder-flux interfacial tension (γ_{lf}) can be obtained by substituting F and θ in eq. (1). In addition, by using eqs. (1) and (3), the difference in γ_{sf} and γ_{sl} can be obtained. Consequently, the wettability between the molten solder and substrate can be analyzed from the viewpoint of the

Fig. 4 Sessile drop geometry determined by the equilibrium relationship between interfacial tensions which are the solder/flux, substrate/flux and substrate/solder.

interfacial tensions which are physical parameters.

Furthermore, the intermetallic compound, which is formed at the interface between the solder and each substrate, was investigated by X-ray diffraction. The sample was prepared by removing the solder on the substrate using an aqueous solution of 15 volume % nitric acid.

3. Results and Discussion

Figure 5 shows the wetting curves of the Au/Cu, Ag/Cu, Pd/Cu and Cu substrates wetted by the Sn-Ag solder between 250 and 290°C. In Fig. 6, the wetting time (t_2 in Fig. 1(a)) and the time to reach the stable wetting (t_3 in Fig. 1(a)) for each substrate wetted by the Sn-Ag solder are shown as a function of the soldering temperature. For all the substrates, both t_2 and t_3 decrease with an increase in temperature.

In addition, t_2 and t_3 for the Cu substrate wetted by the Sn-Pb eutectic solder at 230°C (eutectic temp.+50°C) is shown as a cross on the vertical axis for reference.

In Fig. 6(a), for the Au/Cu and Ag/Cu substrates, the t_2 values are comparable to that for the Cu substrate, while t_2 value for the Pd/Cu substrate is slightly longer than that for the other substrates. Additionally, many of the t_2 values for the Sn-Ag solder are longer than that for the Sn-Pb solder wetting Cu substrate (the cross in Fig. 6(a)).

In the case of the Sn-Ag solder, in order to obtain the wetting time (t_2) which is comparable to the case of the Sn-37Pb solder wetting Cu substrate at 230°C (*i.e.*, 0.76 s), the soldering temperature had to be shifted to a higher temperature, namely, 270°C for the Au/Cu and Ag/Cu substrates, and beyond 270°C for the Pd/Cu substrate.

On the other hand, as shown in Fig. 6(b), t_3 values for the Ag/Cu, Pd/Cu and Cu substrates show similar values, but t_3 value for the Au/Cu substrate is much longer than those of the other substrates. This is considered to be attributed to the difference in the dissolution rates between the coating metals and Cu into the molten Sn-Ag solder, or difference in the effect of the flux on the wettability among the substrates.

Figure 7 shows the maximum wetting force ($F_{\max}$). The maximum wetting force for the Cu substrate wetted by the Sn-Pb eutectic solder at 230°C is shown as a cross on the vertical axis for reference.

At each temperature, for the Au/Cu and Ag/Cu substrates, $F_{\max}$ is larger than that for the Cu substrate, while $F_{\max}$ for the Pd/Cu substrate is smaller than that of the Cu substrate. Also, for each substrate, there is a slight temperature dependence of $F_{\max}$. These differences in $F_{\max}$ among the substrates is due to the difference in the magnitude of θ and γ_{lf} , as will be described later.

It also was found that between 250 and 290°C, $F_{\max}$ values for the Au/Cu and Ag/Cu substrates are larger and $F_{\max}$ value for the Pd/Cu substrate wetted by the Sn-Ag solder is smaller than that for the Cu substrate wetted by the Sn-Pb solder (the cross in Fig. 7).

The contact angle (θ) for the wetting between the Sn-Ag solder and each substrate is shown in Fig. 8. The contact angle for the Cu substrate wetted by the Sn-Pb eutectic solder at 230°C is shown as a cross on the vertical axis for reference.

As shown in Fig. 8, θ ranges from 29 to 38°, from 34 to 35°, from 49 to 52°, and from 42 to 45° for the Au/Cu, Ag/Cu,

Fig. 5 Wetting curves of Sn-3.5Ag solder on the metal-coated Cu substrate; (a) Au/Cu, (b) Ag/Cu, (c) Pd/Cu and (d) Cu, ----: 250°C, —: 270°C, —: 290°C.

Cu, Pd/Cu and Cu substrates, respectively. In other words, the wettabilities of the Au/Cu and Ag/Cu substrates are superior to that of the Cu substrate, while that of the Pd/Cu substrate is inferior to that of the Cu substrate.

The value of θ (36–38°) for the Au/Cu substrate at 250°C is larger than those at the other temperatures ($\theta = 29$ –30°). This is rationalized as follows: As can be seen in Fig. 5, the wetting has not yet finished 5 s after the immersion began, when θ was measured. In fact, θ for the Au/Cu substrate 9 s after the immersion began was 29° at 250°C. If this value is taken as the equilibrium contact angle (shown in Fig. 8 by $\circ^*$), θ at 250°C is comparable to that at the other temperatures. On the other hand, for the Ag/Cu, Pd/Cu and Cu substrates, such a temperature dependence of θ between 250 and 290°C is not shown.

Comparison of the wetting between the Sn-Ag solder and the Sn-Pb solder revealed that θ for the Au/Cu substrate wetted by the Sn-Ag solder is comparable to that for the Cu substrate wetted by the Sn-Pb solder (the cross in Fig. 8), while those for the Ag/Cu and Pd/Cu substrates are larger than that for the Cu substrate wetted by the Sn-Pb solder.

Table 3 summarizes the relative evaluation of the wettability (wetting time, wetting force ($F_{\max}$) and contact angle (θ)) for the noble metal coating as compared with that for the Cu substrate.

The influence of the Au and Ag coatings on the wettability of the Cu substrate wetted by the Sn-Ag solder is small for the wetting time, but large for $F_{\max}$ and θ . Especially, the Au coating decreases θ from 42–45° to 29–30°, that is, improves the wettability of the Cu substrate wetted by the Sn-Ag solder. This effect by the Au coating is remarkable because θ

for the Au/Cu substrate wetted by the Sn-Ag solder is comparable to the Cu substrate wetted by the Sn-Pb solder. On the other hand, the influence of the Pd coating on the wettability of the Cu substrate wetted by the Sn-Ag solder is large for the wetting time, $F_{\max}$ and θ . However, its influence is negative, and causes the wettability of the Pd/Cu substrate to be inferior to that of the Cu substrate.

Figure 9 shows the interfacial tension (γ_{if}) between the Sn-Ag solder and the flux evaluated using eq. (1). The Sn-Pb solder-flux interfacial tension (γ_{if}) on the Cu substrate at 230°C is shown as a cross on the vertical axis for reference. In Fig. 9, the γ_{if} value for the Au/Cu substrate at 250°C was not plotted because, as shown in Fig. 5, the wetting has not yet finished at the time when γ_{if} was measured 5 s after the immersion began.

There is no remarkable difference in γ_{if} , which shows 0.460–0.480 N/m for all substrates. It is considered that γ_{if} is determined by the interaction between the solder and flux, and unlikely to be influenced by the substrate. In other words, there is little change in the composition of the molten solder by the dissolution of the noble metal coating layer into the molten solder. It was also found that there is no remarkable temperature dependence of γ_{if} for any substrates.

From these results in Fig. 8, Fig. 9 and eq. (1), the difference in the wetting force among the substrates in Fig. 7 was found to be attributed to θ , not γ_{if} .

Figure 10 shows values of $\gamma_{\text{sf}} - \gamma_{\text{sl}}$ for the wetting between the Sn-Ag solder and different substrates, evaluated using eqs. (2) and (3). Again, the cross shows $\gamma_{\text{sf}} - \gamma_{\text{sl}}$ for the wetting between the Sn-Pb solder and the Cu substrate at 230°C.

The $\gamma_{\text{sf}} - \gamma_{\text{sl}}$ values for the Au/Cu and Ag/Cu substrates are

Fig. 6 Wetting time and the time to reach the stable wetting of Sn-3.5Ag solder on the metal-coated Cu substrate; (a) wetting time (t_2), (b) the time to reach the stable wetting (t_3), $\circ$: Au/Cu, $\square$: Ag/Cu, $\triangle$: Pd/Cu, $\bullet$: Cu and $\times$: Sn-37Pb wetting Cu substrate at 230°C.

Fig. 7 Wetting force of Sn-3.5Ag solder on the metal-coated Cu substrate; $\circ$: Au/Cu, $\square$: Ag/Cu, $\triangle$: Pd/Cu, $\bullet$: Cu and $\times$: Sn-37Pb wetting Cu substrate at 230°C.

larger than that for the Cu substrate, while that for the Pd/Cu substrate is smaller than that for the Cu substrate. For all substrates, no remarkable temperature dependence of $\gamma_{sf}-\gamma_{sl}$ was found.

From the results mentioned above, it is concluded that the variation in θ and hence, the wettability by coating the Cu

Fig. 8 Contact angle for the wetting between Sn-3.5Ag solder and the metal-coated Cu substrate; $\circ$: Au/Cu, $\square$: Ag/Cu, $\triangle$: Pd/Cu, $\bullet$: Cu, $\times$: Sn-37Pb wetting Cu substrate at 230°C and $\circ^*$: Au/Cu substrate 9 s after the immersion began at 250°C.

Table 3 Influence of the noble metal coating on the wettability of Cu substrate by Sn-3.5Ag solder.

Wettability	Coating layer on Cu		
	Au	Ag	Pd
Wetting time	$\circ$	$\circ$	$\triangle$
Wetting force	$\odot$	$\odot$	$\triangle$
Contact angle	$\odot$	$\odot$	$\triangle$

$\odot$: better, $\circ$: same, $\triangle$: worse compared to uncoated Cu.

Fig. 9 Interfacial tension γ_{if} for the wetting between Sn-3.5Ag solder and the metal-coated Cu substrate; $\circ$: Au/Cu, $\square$: Ag/Cu, $\triangle$: Pd/Cu, $\bullet$: Cu and $\times$: Sn-37Pb wetting Cu substrate at 230°C.

surface with Au, Ag and Pd is attributed to the variation in $\gamma_{sf}-\gamma_{sl}$.

The substrate-flux interfacial tension (γ_{sf}) is considered to be different from one coated substrate to another because γ_{sf} is influenced by the surface of the noble metal-coated Cu substrate. On the other hand, γ_{sl} is likely to be affected by the interaction between the solder and intermetallic compound, which is formed at the interface between the solder and substrate.

The same intermetallic compound, Cu_6Sn_5 , was formed at

Fig. 10 Interfacial tension $\gamma_{sf} - \gamma_{sl}$ for the wetting between Sn-3.5Ag solder and the metal-coated Cu substrate; ○: Au/Cu, □: Ag/Cu, △: Pd/Cu, ●: Cu and ×: Sn-37Pb wetting Cu substrate at 230°C.

the solder/substrate interface, on all the substrates studied. This is in good agreement with the previous studies.^{8,16)} In other words, there was no difference caused by the noble metal coating. This is because the noble metal coating layer is very thin (70 nm) and easily dissolves into the molten solder, and disappears at the interface. This result shows that there is little difference in γ_{sl} among different substrates because γ_{sl} shows the interaction between the Sn-Ag solder and the intermetallic compound, Cu_6Sn_5 .

From these results, the difference in $\gamma_{sf} - \gamma_{sl}$ for different substrates, as shown in Fig. 10, is considered to result from the difference in γ_{sf} . The decrease in θ and hence, the improvement in wettability by coating the Cu surface with Au and Ag is attributed to the increase in γ_{sf} by the Au and Ag coatings. On the other hand, the increase in θ and hence, the degradation of wettability by coating the Cu surface with Pd is attributed to the decrease in γ_{sf} by the Pd coating.

Consequently, for the improvement of the wettability by the noble metal coating, it is important that the same intermetallic compound as that on the Cu substrate without a coating is formed, and that the noble metal coating layer remains in the non-wetted area.

The influence of the intermetallic compound on the wettability, especially θ , was evaluated using the pure Au, Ag, and Pd substrates. Figure 11 shows θ and γ for the pure Au, Ag and Pd substrates wetted by the Sn-Ag solder in comparison with those for the Au/Cu, Ag/Cu, Pd/Cu and Cu substrates mentioned above. The test temperature was 270°C.

The values of θ for the pure Au, Ag and Pd substrates are larger than those for the Au, Ag and Pd-coated Cu substrates, respectively. From the comparison of γ for different substrates, while γ_{lf} shows almost the same values independent of the substrate species, the $\gamma_{sf} - \gamma_{sl}$ values for the pure Au, Ag and Pd substrates are smaller than those for the Au, Ag and Pd-coated Cu substrates, respectively. It is suggested that the decrease in $\gamma_{sf} - \gamma_{sl}$ results from the increase in γ_{sl} because there is less difference in γ_{sf} when the surface of the substrate is consisted of the same kind of metal, such as the Au and Au-coated Cu, Ag and Ag-coated Cu, Pd and Pd-coated Cu substrates, respectively. The difference in γ_{sl} is

Fig. 11 Contact angle and interfacial tensions for the wetting between Sn-3.5Ag solder and the metal-coated Cu substrate, and the pure metal substrate at 270°C.

considered to result from that in the intermetallic compound which is formed at the interface between the Sn-Ag solder and each substrate. In fact, by X-ray diffractometry, it was identified that, on the pure Au, Ag and Pd substrates wetted by the Sn-Ag solder, the intermetallic compound is AuSn_4 , Ag_3Sn and PdSn_4 , respectively, all of which were different from that on the noble metal-coated Cu substrate. These results also suggests that the wettability of the Cu substrate decreases if the thickness of the Au and Ag coatings is too thick to disappear at the solder/substrate interface through dissolution into the molten solder.

Based on these results, it is concluded that, in order to improve the wettability of the Cu substrate with the Sn-Ag solder by the noble metal coating, it is important to select the coating metal which has the large γ_{sf} value, and to optimize the coating thickness to suppress an increase in γ_{sl} .

4. Conclusion

The influence of the noble metal coating on the wettability of a Cu substrate by Sn-3.5Ag solder was studied using the meniscograph method combined with the contact angle measuring system developed by us. The following conclusions were derived from the present study.

- (1) The wettability of the Cu substrate with Sn-3.5Ag solder is improved by Au and Ag coatings on the Cu substrate, showing that θ decreases and $F_{\max}$ increases though there is little influence on the wetting time.
- (2) The wettability of the Cu substrate with Sn-3.5Ag solder is degraded by the Pd coating on the Cu substrate, showing that θ increases and $F_{\max}$ decreases, and the wetting time becomes slightly longer.
- (3) It was clarified that, by measuring the contact angle using the meniscograph method, the difference in the wetting force by the Au, Ag and Pd coatings on the Cu substrate is attributed to the difference in magnitude of θ , not that of γ_{lf} for each substrate.
- (4) The decrease in θ by the Au and Ag coatings on the Cu substrate is attributed to the following: For the Au and Ag coatings on the Cu substrate, the coating layer remaining at the non-wetted area up to the tip of the molten solder resulted in an increased γ_{sf} .
- (5) The increase in θ by the Pd coating on the Cu substrate is attributed to the decrease in γ_{sf} by the Pd coating.
- (6) In order to improve the wettability of the Cu substrate with the Sn-Ag solder by the noble metal coating, it is important to select the coating metal which has the large γ_{sf} value, and to optimize the coating thickness to suppress an increase in γ_{sl} .

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