

**Assessing The Effect Of Surface Roughness On
The Wetting Of Cu And Pd By Sn/Pb Solder**

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ABSTRACT

Here we examine the effect of varying substrate surface roughness on solder wettability (area of spread) and the time required to reach terminal area of spread. Results are given for solder wetting experiments that were performed on copper (Cu) substrates having chemically etched surfaces, as well as, Alumina (Al_2O_3) substrates electroplated with various thicknesses of palladium (Pd). The effect of etching on the Al_2O_3 /Pd specimens was also examined as related to surface roughness and solder spread. These surface treatments were found to significantly alter wettability. Substrate surface roughness properties were quantified using an optical profilometer. Area of spread and time to terminal area of spread were quantified using video recording and image analysis equipment.

Substantial improvements were observed in both solder wettability and time to wet with the uniformly etched Cu surfaces used in this study. For the Cu substrates, the average terminal area of spread is shown to be directly related to the substrates root mean square (RMS) surface roughness. The rate of wetting of the Cu surfaces is also shown to increase when chemical surface treatment is used. Maximum wetting on the Al_2O_3 /Pd specimens was found to be directly related to surface smoothness (i.e., inversely related to surface roughness). The average terminal area of spread of Al_2O_3 /Pd specimens is inversely related to the vertical distance from the highest surface peak to the deepest surface valley (i.e., peak-to-peak variation)

INTRODUCTION

Artificially enhancing the solder ability of a surface can at times prove to be advantageous. As chip packaging geometries become increasingly complex, the issue of solder wettability becomes significantly more important. Certain chip packaging designs (i.e., BGA, tab pack, flip-chip, and others) often conceal solder connections making it nearly impossible to visually inspect solder connection surety. Inability to visually inspect the solder connections necessitates additional functional testing which increases chip production time. Surfaces with artificially enhanced solder spreading

capacity can help to alleviate some of these problems by ensuring a better ratio of accepted-to-rejected parts.

The benefits resulting from an in depth, intuitive understanding of the effects of surface roughness on solder wettability will allow future chip designs to become increasingly more complex—both in geometry and density.

MATERIALS AND SUBSTRATE PREPARATION

Two types of substrate materials were used in this study. The first substrate material was an oxygen-free high conductivity (OFHC) Cu with dimensions of ~25 mm square by ~3 mm thick. Five different sets (of five substrates each) were etched with a 50% by volume HCl stock solution (with 235 g/L of $FeCl_3$ added) for one, three, five, and nine minutes. Another set (of two substrates each) was etched in the same solution for fifteen minutes. Immediately after being taken from the etching bath, the substrates were double rinsed in isopropyl alcohol and blown dry with argon. The substrates were then fluxed to inhibit oxidation prior to the testing procedure. The wetting experiments were performed within two minutes after completing the etch, rinse, and flux steps mentioned above.

The second substrate material used in the study was Al_2O_3 . Al_2O_3 was chosen as a substrate material because it is commonly used in manufacturing circuits by means of thin film vacuum deposition methods. The dimensions of the substrates were ~38 mm by ~32 mm by 0.635 mm. The ceramic substrates were first sputter cleaned, then coated on one side with an adhesion layer (~250 Å) of tantalum, and finally coated with a conductive layer (~750 Å) of Pd. After this vacuum deposition process, the substrates were electroplated in a Pd bath with no brightener at 130°C. Three different sets (of five substrates each) were electroplated with Pd. One of the sets was plated with a thin, smooth (0.5mm) Pd coating. The two other sets of substrates were electroplated with thicker, rougher (2.4 mm and 3.5 mm) layers of Pd. The Al_2O_3 substrates with 3.5 mm of Pd deposited on them were also subjected to a HCl/HNO₃ (3:1 solution by volume) etch for ten seconds (five substrates each) and 60 seconds (two substrates each) to further roughen the substrate surfaces. These substrates were stored in a desiccator after plating (and again

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immediately after roughness measurements and SEM micrographs were obtained), and remained stored in this manner until the wetting experiments were conducted.

The solder used was a 63Sn/37Pb eutectic alloy. When received, the solder was in wire form. The wire was cut into sections having a mass of 10 ± 0.5 mg after which they were re-flowed in a mildly activated rosin (RMA) flux to form spherical solder balls. The spherical shape approximates a non-wetting condition (or lowest solder surface energy state) at the onset of the wetting event. The fluxing agent used for the wetting study was a 50% (by volume) IPA/RMA flux solution.

To monitor the Cu substrate temperature, each Cu substrate had a 1.5 mm (diameter) by 4 mm (depth) hole which was filled with high temperature, high conductivity grease. This hole served as a thermocouple well. A thermocouple was attached between the substrate and the heat source to ensure a substrate temperature of 220 °C. To verify Al₂O₃/Pd substrate temperature, a spare Al₂O₃/Pd substrate was selected and soldered to a thermocouple. The thermocouple's temperature was then monitored to ensure a substrate temperature of 220°C nominal. A hot plate was used as the heat source during the study.

EXPERIMENTAL

The substrates were placed onto the hot plate, allowed to come to temperature, and lightly fluxed (fluxed again in the case of the Cu substrates). The substrate's temperature was then allowed to stabilize (the substrate's temperature decreases momentarily as flux is applied). After substrate temperature stabilized, a video tape recorder was started, and a solder sphere was placed onto the substrate. The spreading event was recorded until no visible motion of the three phase line was observed.

All experiments were performed without the aid of an environmental chamber. The entire wetting event was recorded from directly overhead. For calibration purposes, several seconds of video were recorded using a 10 mm² reticule located in the position where the substrates would later be placed and videotaped. The video recorder used in this study was equipped with a time code generator board so that accurate (30 frames per second) area of spread versus time data could be acquired. A computer equipped with a digital frame grabber board and image analysis software (Image Pro Plus) was used to collect digital 'snapshots' which were later manipulated to achieve a high contrast image. The digital images were then analyzed--resulting in 'area of spread versus time' raw data. The estimated error is approximately three to five percent in regard to the area of spread measurements. This error is due to the subjectivity involved in the placement of the measurement cursor when using the image analysis software.

Both the etched Cu and Al₂O₃/Pd substrate surface roughnesses were characterized by means of optical profilometry. A WYCO, RST Plus, optical surface profilometer was used to quantify the extent of substrate surface morphology variation. The WYCO profilometer uses white light as the source in an interferometer and measures the degree of fringe modulation or coherence present. The peak of the

coherence function provides a measure of the surface geometry (ASME 846.1 85, 1992; and WYCO Corporation, 1994).

The WYCO unit provided measurements of the RMS surface roughness, R_q; arithmetic average roughness, R_a; the vertical distance from the highest peak to the deepest valley, R_t; and the surface index, which is the ratio of the measured surface area to the planar surface area. The surface index is also known as the Wenzel roughness coefficient. The instrument is capable of profiling surfaces with vertical steps in excess of 100mm, with R_q values ranging from 1 Å to 20mm.

RESULTS AND DISCUSSION

Solder spreading on chemically roughened Cu surfaces can attain final areas of spread that are orders of magnitude greater than those Cu surfaces that have not been similarly treated (Hosking, et al., 1996).

Figure 1 shows representative scanning electron micrographs of the Cu substrates used in the study. The unetched Cu (a) was observed to have a much larger, smoother surface morphology than that of the etched substrates. The etched substrates had progressively deeper recesses and taller surface peaks as the etch durations were increased.

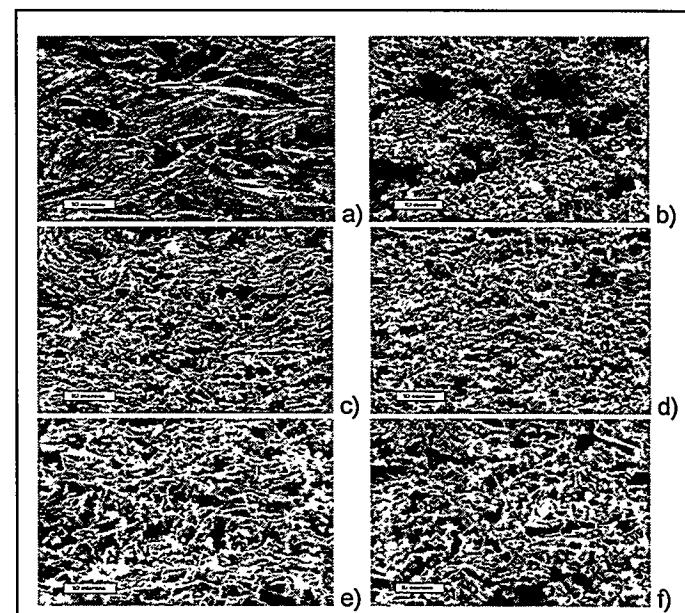


FIGURE 1: SEM micrographs of unetched and etched Cu. Unetched Cu (a); 1 (b), 3 (c), 5(d), 9(e) and 15 (f) minute HCl/FeCl₃ etched Cu. NOTE: The marker length is 10µm.

The area of solder spread (in mm²) as a function of time (in seconds) data for the Cu etched substrates are shown in Figure 2. The initial rate of solder spread (as well as area of spread) is similar for all etch durations (one minute through fifteen minutes). As time progresses, however, the Cu substrates subjected to longer etch times

approached their terminal area of spread more rapidly than those whose etch durations were shorter in length. The difference in wetting response is noted as very significant, considering the nominally constant volume of solder that was used in the study.

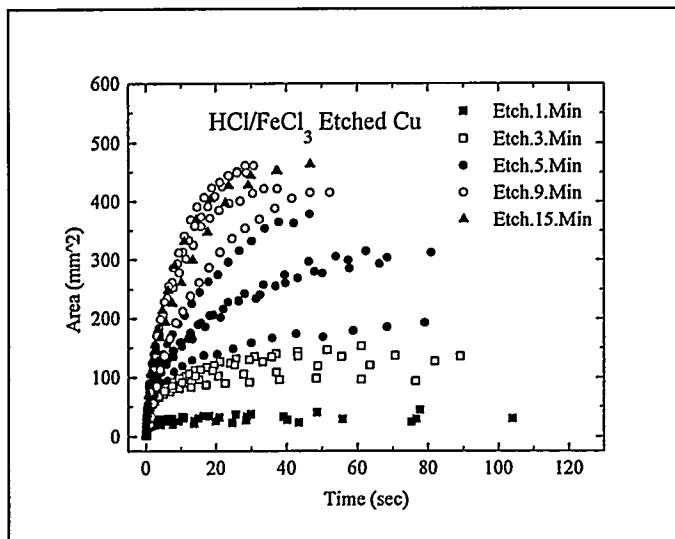


FIGURE 2: Raw data for the HCl/FeCl₃ etched Cu substrates with 63Sn/37Pb solder alloy.

The Cu substrates whose etch durations were nine and fifteen minutes provided areas of spread that were roughly nine times greater than those areas of spread observed with the one minute etch time. The average terminal area of spread is roughly inversely proportional to the time required to reach the terminal area of spread. The trend clearly shows that as etch time increases, both the terminal area of spread and time to terminal area of spread increases. Clear divisions are shown between the 1, 3, 5, and 9 minute data sets (i.e., there are no overlapping data points as the substrates approach terminal area of spread).

This is not the case with the nine and fifteen minute data—there is distinct data overlap present. Capillary action (induced by high peaks and low valleys) appears to be diminished with a fifteen minute etch. The elongated Cu peaks present with a nine minute etch may be etched away during a fifteen minute etch, giving the solder less incentive to flow via capillary action. Figure 3 expresses area of spread as a function of RMS roughness (R_q) for the Cu etched substrates.

As inferred by the data overlap seen in Figure 2, the average roughness (R_q) of the Cu etched nine minute substrates exceeds the R_q value of the fifteen minute substrates. The difference between these two R_q values (~0.1 μm); however, is marginal; as is the difference between their respective maximum terminal area of spread values (2.9 mm^2). The area of spread data shown in Figure 3 (i.e., the one through nine minute etch times) shows a very positive relationship between area of spread and roughness—and therefore etch time.

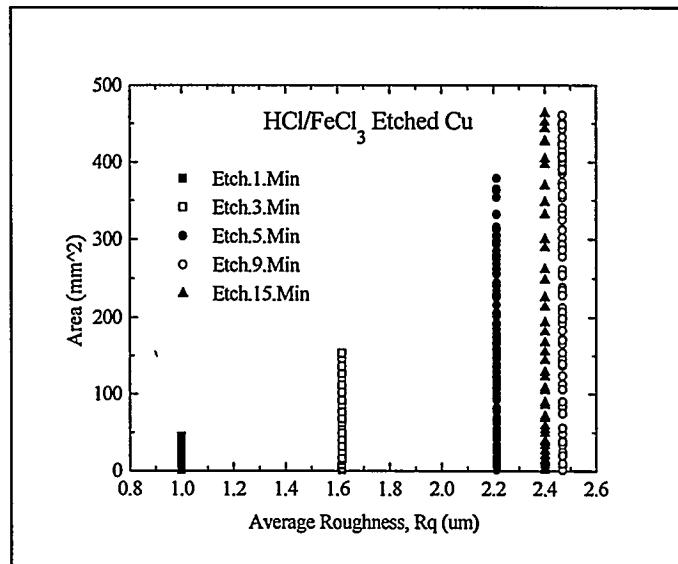


FIGURE 3: Average roughness versus area of spread data for the HCl/FeCl₃ etched Cu substrates.

By examining the average values of the Cu substrate surface morphology in Table 1, no distinct reasons are apparent for the increased average area of spread values present with the 15 minute etched substrates. Note that the WYCO data for the 15 minute substrates show a decrease in average roughness values. These results are unexplained at this time.

TABLE 1: Average area of spread and WYCO surface roughness measurements for the HCl/FeCl₃ etched Cu substrates.

Etch Time (min)	Avg. R _q (μm)	Avg. R _a (μm)	Avg. R _t (μm)	Avg. Wenzel Ratio	Avg. Terminal Area of Spread (mm^2)
1	1.00	0.77	12.63	3.12	33.70
3	1.62	1.30	16.12	3.63	129.98
5	2.21	1.82	19.98	3.76	298.29
9	2.47	2.01	21.11	3.83	439.22
15	2.40	1.95	20.61	3.68	457.61

The next series of experiments investigated the wetting of Pd coated Al₂O₃ by 63Sn/37Pb eutectic solder. Since Pd and Sn are known to interact, reactive mechanics (wetting) forces are expected to be present in this study. Reactive wetting has been observed to create intermetallic boundaries at the perimeter of the three phase line; as well as along the solder/substrate interface in previous studies (Wang and Tu, 1995).

Representative SEM micrographs of the 0.5 μm , 2.4 μm , and 3.5 μm (etched with the HCl/HNO₃ solution for 60 seconds) Al₂O₃/Pd substrates are shown in Figure 4. The 0.5 μm (a) and 2.4 μm (b) specimens have a fairly homogeneous surface morphology with the 0.5 μm specimen's surface having a smaller variation in profile-

amplitude and a larger variation in profile-wavelength. The $3.5\mu\text{m}$ substrate subjected to a 60 second HCl/HNO₃ etch has a more heterogeneous surface morphology with significant variation in both profile-amplitude and profile wavelength across the specimen's surface. A SEM micrograph of a $3.5\mu\text{m}$ substrate subjected to a 10 second HCl/HNO₃ etch was not available. It is suspected that the substrate's surface morphology would also be heterogeneous.

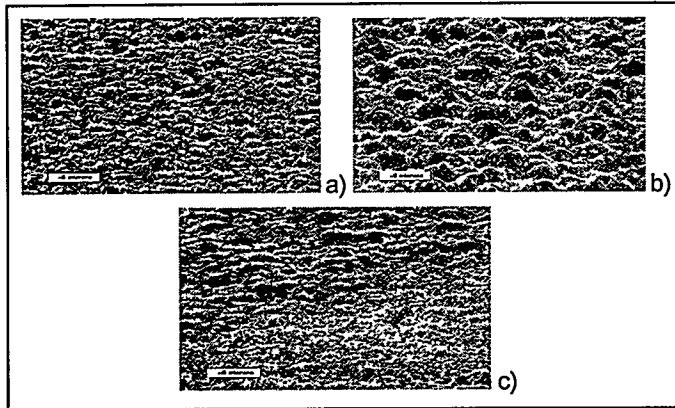


FIGURE 4: SEM micrographs of $0.5\mu\text{m}$ (a), $2.4\mu\text{m}$ (b), and $3.5\mu\text{m}$ (with 60 second HCl/HNO₃ etch) (c) Al₂O₃ / Pd substrates. NOTE: The marker length is $5\mu\text{m}$.

The area of solder spread (in mm²) as a function of time (in seconds) data for the Al₂O₃/Pd substrates are shown in Figure 5. The initial rate of solder spread (as well as area of spread) appears to be similar for all test conditions. The Pd/Sn interaction causes a decrease in response time as related to wetting. The Al₂O₃/Pd, Sn/Pb, study should be noted as having a distinctly different solder wetting phenomena occurring than the Cu, Sn/Pb, study. At the onset, the Al₂O₃/Pd, Sn/Pb, system exhibited a pause before wetting occurred. However, the Cu, Sn/Pb system began to wet immediately.

Of all the Al₂O₃/Pd substrates, the unetched $0.5\mu\text{m}$ Al₂O₃/Pd substrates provided the highest terminal area of spread value (27.88 mm^2); as well as the largest standard deviation ($\pm 7.98\text{ mm}^2$) between substrates. The unetched $2.4\mu\text{m}$ Al₂O₃/Pd substrates provided the next highest terminal area of spread value (26.10 mm^2) with an improved standard deviation ($\pm 6.72\text{ mm}^2$). The third best terminal area of spread value (16.80 mm^2) and standard deviation ($\pm 4.38\text{ mm}^2$) was obtained from the $3.5\mu\text{m}$ substrates that were subjected to a 60 second HCl/HNO₃ etch. The $3.5\mu\text{m}$ substrates which were subjected to a 10 second HCl/HNO₃ etch were seen to have the lowest terminal area of spread value (15.05 mm^2) with a slightly better standard deviation ($\pm 4.36\text{ mm}^2$) than the 60 second etch substrates.

In comparing the results of the Cu etched substrates to that of the Al₂O₃/Pd substrates, the relationships visible in Figure 5 are not as clearly delineated as were those with the etched Cu data of Figure 2. The maximum areas of spread with the Cu etched substrates are approximately twenty times that of the maximum for the Al₂O₃/Pd substrates. Although, the Al₂O₃/Pd substrates were stored in a desiccator prior to performing the experiments, it is possible that some oxide buildup may have occurred on the substrates during WYCO and SEM testing. This supposed oxide layer could possibly

be removed (after some period of time) as a result of flux application. The solder would then be able to flow as expected.

Although the respective area of spread data are significantly different for the two substrate types, the information presented can still be related in regards to apparent differences in surface morphology. Figure 6 expresses area of spread as a function of RMS roughness (Rq) for the Al₂O₃/Pd substrates tested.

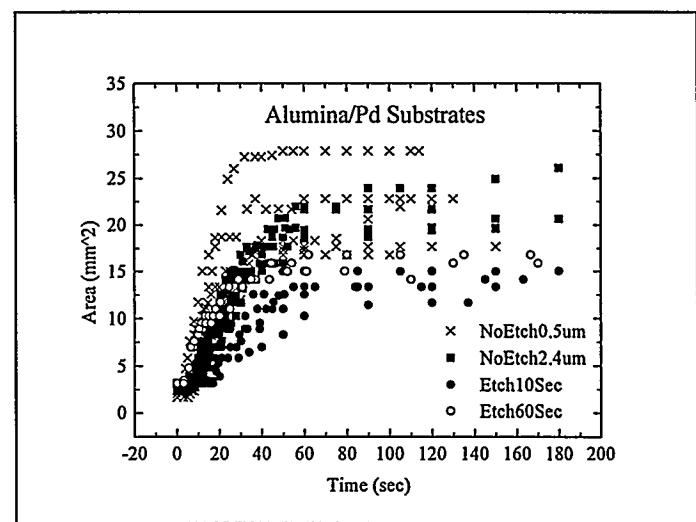


FIGURE 5: Raw data for the Al₂O₃/Pd substrates with 63Sn/37Pb solder.

The average roughness (Rq) of the Al₂O₃/Pd substrates is shown to adversely effect the ability of the 63Sn/37Pb solder to wet the substrate surfaces. The Figure 6 data show that as the thickness of the electroplated Pd increases ($0.5\mu\text{m}$ to $2.4\mu\text{m}$), as does average roughness (Rq). As a result, there is an apparent decrease in wetting as roughness increases with Pd thin film thickness.

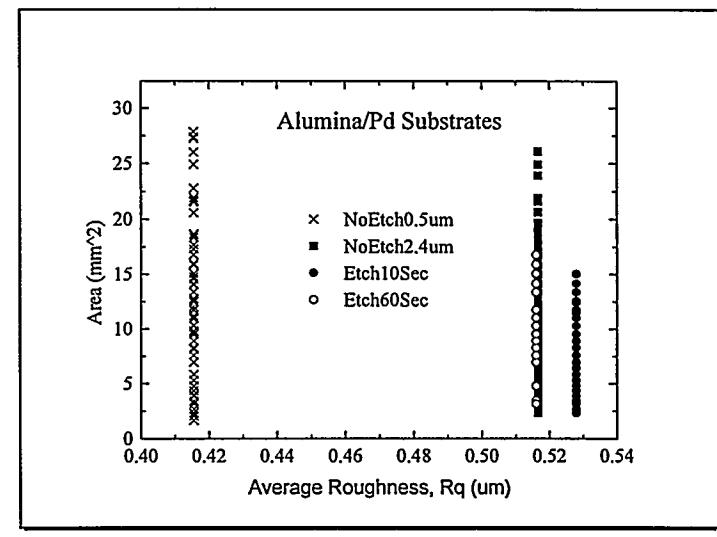


FIGURE 6: Average roughness versus area of spread data for the Al₂O₃/Pd substrates.

The pre-etch R_q value of both etched substrates was on average $0.52\mu\text{m}$. The $3.5\mu\text{m}$ substrates subjected to a 10 second HCl/HNO₃ etch had an increase in average roughness and a decrease in wettability (area of spread). Alternatively, the $3.5\mu\text{m}$ substrates subjected to a 60 second HCl/HNO₃ etch had a decrease in average roughness and an increase in wettability.

By examining the average surface morphology values for the Al₂O₃/Pd substrates in greater detail (see Table 2), it would seem that surface profile-amplitude sensitivity (Average R_t or peak-to-valley variation) is the driving factor in regard to the Al₂O₃/Pd type of substrates wettability with 63Sn/37Pb solder (i.e., the average R_t value is seen to follow the average terminal area of spread in an inverse manner).

TABLE 2: Average area of spread and WYCO surface roughness measurements for the Al₂O₃/Pd substrates.

Al ₂ O ₃ /Pd Substrate Type	Avg. R_q (μm)	Avg. R_a (μm)	Avg. R_t (μm)	Avg. Wenzel Ratio	Avg. Terminal Area of Spread (mm^2)
0.5 μm	0.42	0.32	3.83	2.36	21.66
2.4 μm	0.52	0.42	4.17	2.74	21.22
3.5 μm (10 sec etch)	0.53	0.40	4.85	2.48	13.86
3.5 μm (60 sec etch)	0.52	0.40	4.53	2.35	16.35

Note that in the case of the etched Cu substrates, the maximum average terminal area of spread corresponds to one of the intermediate levels of surface roughness (i.e., the 15 minute data set). As the Cu substrate's surface becomes rougher, the solder spreads quickly to its final area of spread, possibly due to capillary action (Rye, et al., 1996). Since the solder is no longer confined to a spherical cap, and because the solder is pulled across the surface due to wicking effects, the time to terminal area of spread decreases proportionately. With the Al₂O₃/Pd substrates, the maximum average terminal area of spread corresponds to the lowest level of average peak-to-peak surface variation. With the rougher Al₂O₃/Pd substrates, there is a reduction in area of spread. The longest time to final area of spread is in the case of the 2.4 μm substrates.

CONCLUSIONS

The rate and area of spreading of 63Sn/37Pb solder on chemically treated Cu substrates show a significant increase over those substrates that have not been surface treated. The opposite effect is observed on the spreading of 63Sn/37Pb solder on Al₂O₃/Pd substrates. The rate and area of spreading are greater for the smoother surfaces in that case.

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