SAND--90-3248C

DE91 018348

SAND90-3248C

WETTABILITY OF LOW TEMPERATURE SOLDER ALLOYS FOR STEP-SOLDERING*

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ABSTRACT

The electronics industry relies on step-soldering during device attachment or subsequent reworking of heavily populated hybrid microcircuits and multilevel networks. Although there are many low to intermediate melting solder alloys that are commercially available, their wettability on typical base metals, such as Cu, Ni, Au, and Sn, are not well characterized. This investigation examines the wetting behavior of several In, Sn, Pb, Bi, and Ag containing solder alloys on Ni-Sn plated aluminum alloy substrates with soldering temperatures ranging from 145 to 200°C. Two rosin mildly activated (RMA) fluxes were included in the study. The wettability experiments were conducted with a Sandia designed wetting balance system. Wetting differences were observed between the two fluxes. The more active flux gave better wetting results and less variability in the meniscus terminations at the lower soldering temperatures. Wetting generally varied from adequate to very good. The Bi-bearing alloys generally gave the lowest wetting values. Work is in progress to determine the effects of aging on intermetallic growth and subsequent mechanical strength.

INTRODUCTION

The electronics industry generally relies on Sn-Pb solder alloys for attaching electronic devices to other discrete components or printed wiring assemblies (PWA). The principle compositions for making these solder joints are either the eutectic 63Sn-37Pb or near-eutectic 60Sn-40Pb alloys (wt. %). 63Sn-37Pb melts at 183°C and is typically soldered between 220-240°C. 60Sn-40Pb has a melt range of 183 to 188°C with soldering temperatures comparable to the eutectic composition. These alloys exhibit excellent wetting behavior on most base metals. The resulting solder joints have excellent thermal, electrical, and mechanical properties.

The use of lower melting temperature solder alloys during electronic manufacturing is becoming increasingly important as PWA population and network levels increase. Reworking of defective solder joints without remelting neighboring joints has added to this need. Step soldering provides a means in achieving these denser, more complex circuits.

Step soldering is defined as a method of sequentially producing several solder joints with different solder alloys and without remelting the previously solidified joints. Sn-Pb alloys are usually used in the first soldering step with other lower melting alloys to follow. Although these subsequent steps generally give good wetting results, the joint strengths may not approach the same value as obtained with a Sn-Pb joint.

There are several, commercially-available solder alloys that can be applied to step-soldering. These alloys are primarily based on Bi, Cd, In, Sn, Pb, and Ag compositions. Their soldering temperatures can range from 100°C for Bi-based alloys to 180°C for Sn-Pb-In alloys. Although they satisfy the melting requirements for step-soldering, their wetting behavior on most electronic base surfaces (Cu, Ni, Au, Sn, and SnPb) is not well

* This work was conducted at Sandia National Laboratories and supported by the U. S. Department of Energy under contract number DE-AC04-76DP00789.

established. The effects of intermetallic growth on subsequent mechanical strength must also be considered before fully integrating these lower melting solder alloys into actual electronic manufacturing.

The work reported in this paper examines the wetting behavior of several low to intermediate melting solder alloys on Ni-Sn electroplated aluminum alloy substrates. The particular application was the development of a step soldering process to attach PWAs to an aluminum backing plate. Parallel studies are in progress to address the intermetallic growth and strength issues of the lower melting alloys, but will not be discussed here.

EXPERIMENTAL PROCEDURE

MATERIALS AND PREPARATION - The base metal evaluated in this study was aluminum alloy 6061 in the T6 condition. 6061 has a nominal composition (wt. %) of 0.6 Si, 0.28 Cu, 1.0 Mg, 0.2 Cr, and the balance Al. Test coupons were cut from 0.86 mm sheet with individual dimensions of 25.4 x 25.4 x 0.86 mm. Since aluminum alloys form a very stable oxide on its surface that is difficult to reduce or wet during conventional solder processing, the 6061 substrates were metallized with Ni and Sn to promote wetting. The samples were initially plated with 5 μm (200 μin.) of electroless Ni-P to facilitate adhesion of the Ni and Sn layers. Electrolytic Ni (sulfamate bath) was then deposited on the Ni-P layer to a thickness of 8 μm (300 μin.), followed by 3 μm (100 μin.) of Sn (stannous sulphate bath without brighteners). The reported plating thicknesses were $\pm$ 0.6 μm (25 μin.). The Sn-Ni plated samples were then reflowed in a vapor phase system at 250°C to retain wettability of the deposited surfaces [1-3].

Nine solder alloys were evaluated. Their compositions were based on In, Sn, Pb, Bi, and Ag additions. Compositions, melting temperatures, and test conditions are listed in Table 1. Solder wettability test temperatures ranged from 145°C to 200°C and were generally 20-25°C above the melting temperature of each alloy. The 62.5Sn-36.1Pb-1.4Ag alloy served as the experimental control.

Two rosin mildly activated (RMA) fluxes were selected for the wetting tests. The first, K197, contains 37 wt. % solids in an isopropyl alcohol carrier. Its activator is an amine hydrochloride. The second flux, A35, has 35 wt. % solids in the same alcohol carrier with a brominated activator. Both fluxes are primarily rosin-based abietic acid. The concentrated fluxes were diluted 1:1 with isopropyl alcohol to simulate production use.

Samples were ultrasonically cleaned before fluxing with a three step process of trichloroethylene and isopropyl alcohol rinses and a final filtered nitrogen dry. Flux was uniformly applied to the cleaned test surfaces by dipping. The fluxed samples were allowed to dry ten minutes before conducting the wetting experiments.

SOLDER WETTABILITY EXPERIMENTS - Solder wettability [4] was determined with a Sandia-designed wetting balance system [5-8]. The apparatus (Fig. 1) consists of an electrobalance which measures wetting force as a function of time. The test methodology is based on the Wilelmy plate technique [9]. The contact angle ø (Fig. 2) formed between the top of the solder meniscus and the base plate can be calculated with equations derived from the elastica relationship [10-11]. The angle becomes a function of the meniscus height, surface tension between the flux and molten solder (or maximum wetting force), sample geometry, and solder density [6]:

$$\o = \sin^{-1} [(4W^2 - (pgPh^2)^2) \div (4W^2 + (pgPh^2)^2)]$$

where W is the maximum wetting force, p is the solder density, g is the acceleration due to gravity, P is the sample perimeter, and h is the meniscus height.

Contact angle represents the equilibrium condition of wetting and is one of several indicators of wettability. The best wetting occurs as the contact angle or surface tension between the molten solder and flux decreases and meniscus height increases. Wetting is also affected by the difference between the base metal-flux and base metal-solder surface tensions. The difference, which is the force measured by the wetting balance, represents the cleanliness of the base surface and metallurgical reaction between the base metal and solder alloy. This last parameter must be maximized to optimize wetting. Another important parameter to consider is the wetting rate since it has a direct influence on selecting soldering methods that will produce acceptable wetting within the process time constraints.

Meniscus height, maximum wetting force, wetting rate, time to 90° turnaround (solder bath perpendicular to the immersed coupon), and time to maximum wetting force data were measured for the selected solder alloys and fluxes shown in Table 2. The sample immersion rate and depth were 12.5 mm/s and 4 mm, respectively. Since the reflowed Sn surface made it very difficult to accurately measure meniscus heights because of remelting near the meniscus edge, contact angles were not computed. Force and time data were taken from the wetting curves (Fig. 3). The measured values, however, were very representative of the wetting behavior of the lower temperature, step soldering alloys. Meniscus heights were independently determined with a meniscometer. The meniscometer uses a traveling microscope to measure the advancing solder meniscus. An immersion depth of 4 mm was also used in the meniscometer experiments.

Each balance and meniscometer data set consisted of six samples to satisfy statistical sampling. Both sets of experiments required preheating of the relatively thick test coupon to avoid heat transfer (temperature gradient) effects on the wetting results. The samples were preheated to 120-150°C with an air gun and immediately tested to minimize oxidation of the exposed Sn surfaces. The actual preheat temperatures were approximately 30-40°C below the soldering temperature being investigated (Table 1). The exception was with the 49Bi-21In-18Pb-12Sn solder alloy and 80°C test temperature, where no preheat was used.

RESULTS AND DISCUSSION

The wetting results are listed in Table 2. Significant differences in wetting were observed between the two fluxes on all of the solder alloys. The greatest differences occurred at the lower wetting temperatures (165°C or less). The A35 flux was generally more active than the K197 flux and gave the best wetting results. The A35 fluxed samples had larger meniscus heights, maximum wetting forces, and wetting rates and shorter wetting times. Their meniscus terminations were also more clearly defined and less variable.

Meniscus height is a relatively direct measurement of solder wettability. Experience has shown that a meniscus height above 2.5 mm generally represents adequate wetting, while a meniscus height exceeding 3 mm suggests good to excellent wetting. A bar chart of meniscus height as a function of the different solder alloys and fluxes is shown in Figure 4. The chart suggests adequate to excellent wetting on the A35 fluxed samples and only poor to good wetting on the K197 samples. The latter samples also had very uneven meniscus profiles, although the K197 appeared more active at the higher soldering temperatures. Negligible wetting was obtained for the 49Bi-21In-18Pb-12Sn alloy at 80°C and 150°C when fluxed with K197. The same alloy had adequate wetting at 150°C when fluxed with A35. Meniscus heights ranged from negligible to 3.08 ± 0.21 mm with K197 and from 2.53 ± 0.27 to 3.30 ± 0.19 mm with

A35. The average meniscus height of the control solder alloy, 62.5Sn-36.1Pb-1.4Ag, was 2.82 ± 0.10 mm with K197 and 2.96 ± 0.18 mm with A35.

Maximum wetting force corresponds to the weight of the solder meniscus. It is also the difference between the base metal/flux and base metal/liquid solder surface tensions. Wetting increases as this difference increases. A bar chart of maximum wetting force as a function of the different solder alloys and fluxes is shown in Figure 5. The wetting force differences between the K197 and A35 data were relatively greater compared to the meniscus height results. The K197 samples had generally larger experimental scatter. K197 was especially ineffective on the Bi-containing alloys and In-3Ag. Although wetting was significantly improved with the A35 flux, wetting forces were still low on the Bi alloys. Wetting forces varied from negligible to 188 ± 45.1 μN/mm with K197 and from 184 ± 45.3 to 433 ± 42.4 μN/mm with A35. The average wetting force for the control solder alloy, 62.5Sn-36.1Pb-1.4Ag, was 188 ± 45.1 μN/mm with K197 and 375 ± 23.1 μN/mm with A35.

Wetting rate is another important parameter that provides information on the wetting kinetics of the advancing solder meniscus. A bar chart of wetting rate as a function of the different solder alloys and fluxes is shown in Figure 6. There was a large difference in how the two fluxes affected wetting rate. Very low rates were obtained with the K197 flux, while the A35 flux gave significantly higher wetting rates. The results were generally independent of the solder alloy and meniscus height/maximum wetting force data. The phenomenon can be explained by the different chemical activities of the fluxes. Wetting rates varied from negligible to 61 ± 28.6 μN/mm·s with K197 and from 119 ± 50.5 to 332 ± 59.1 μN/mm·s with A35. The average wetting force for the control solder alloy, 62.5Sn-36.1Pb-1.4Ag, was 61 ± 28.6 μN/mm·s with K197 and 207 ± 65.4 μN/mm·s with A35.

A final measure of solder wettability is the time it takes to reach the 90° turnaround and the maximum wetting force. These values are useful to know when choosing the soldering process to fabricate production joints, since adequate time must be allowed to complete the wetting process. Bar charts of the times to 90° turnaround and maximum wetting force as a function of the different solder alloys and fluxes are shown in Figs. 7 and 8, respectively. The wetting times followed the same general trend as the wetting rate data with longer times for the K197 flux. The shortest times to 90° turnaround occurred with the A35 flux and higher temperature solder alloys. Time to 90° turnaround varied from 1.6 ± 0.6 s to 8.8 ± 5.2 s with K197 and from 0.1 ± 0.1 s to 0.8 ± 0.7 s with A35. Time to the maximum wetting force ranged from 12.7 ± 1.4 s to 16.8 ± 2.2 s with K197 and from 2.7 ± 0.3 s to 12.9 ± 2.6 s with A35. The control solder alloy, 62.5Sn-36.1Pb-1.4Ag, typically had the shorter wetting times.

The 40In-40Sn-20Pb solder alloy has been used to step solder printed circuit boards into housings [12] with satisfactory wetting and mechanical strength properties. The alloy is especially attractive for applications where thermal expansion differences exist between the base materials and creep deformation in the solder joint is required to reduce residual stresses on cooling from the intermediate soldering temperature. After examining the results of the wetting experiments above, the microstructures of the 40In-40Sn-20Pb dipped samples were investigated in more detail.

The 40In-40Sn-20Pb wetting experiments were consistent with excellent wetting on the A35 fluxed samples and poor wetting on the K197 fluxed samples. The A35 samples had an average meniscus height of 3.09 ± 0.17 mm, a maximum wetting force of 407 ± 27.3 μN/mm, a wetting rate of 289 ± 40.7 μN/mm·s, and a maximum wetting time of 6.3 ± 1.8 s. The A35 results were comparable to the Sn-Pb-Ag/A35 control sample data. The K197 samples conversely had an average meniscus height of 0.57 ± 0.07 mm, a maximum wetting force of 113 ± 63.2 μN/mm, a wetting rate of 41 ± 28.4 μN/mm·s, and a maximum wetting time of 14.3 ± 2.9 s. Fig. 9 shows the relationship between wetting force and the inverse of time (1/t) at 150°C for the In-Sn-Pb alloy and the two RMA fluxes. The force data was sampled from the time to 90° turnaround through the time to maximum wetting. The fitted lines ($r^2 = 1.0$) clearly demonstrate the effectiveness of the A35 flux over K197 with significantly better wetting results with A35.

Additional In-Sn-Pb and K197 meniscometer experiments were conducted at 200°C to determine if the K197 flux could yield better wetting through improved activation at the higher soldering temperature. The 200°C tests gave an average meniscus height of 3.40 ± 0.05 mm, a significant increase from the 0.57 mm meniscus height of the 150°C tests (Fig. 10). These results confirmed the sensitivity of K197 to temperature. Although no further K197 activation experiments were performed, the wetting behavior of the other solder alloys at temperatures exceeding 165°C suggested that satisfactory wetting is probable with K197 and 40In-40Sn-20Pb between 170 and 200°C and the results could be applied to step soldering.

40In-40Sn-20Pb wetting samples were cross-sectioned to examine the reaction layer between the plated layers of the aluminum coupon and the solder alloy. Scanning electron microscopy (SEM) and energy dispersive spectrometry analysis (EDS) was conducted to identify the reaction products. EDS revealed a Ni-Sn reaction layer (Fig. 11) that corresponds to the Ni_3Sn_4 intermetallic. The intermetallic is formed during reflowing of the Sn plating and subsequent wetting of the In-Sn-Pb solder alloy. SEM (Fig. 12) showed the Ni_3Sn_4 layer to be relatively thin, 1 μm or less. No other elements were identified in the reaction zone, although In could substitute for Sn during the wetting reaction. Since SEM analysis has a limited spatial resolution of the order of the Ni_3Sn_4 layer, a more detailed analysis is in progress with electron microprobe analysis to better characterize the reaction product. Similar microanalysis is being performed on other low melting temperature solder alloys to identify the intermetallic reactions on Au, Cu, and Ni surfaces and to determine the effects of aging on intermetallic growth.

CONCLUSIONS

The solder wettability of several low to intermediate melting solder alloys containing In, Sn, Pb, Bi, and Ag was investigated. The wetting samples were Sn-Ni plated aluminum alloy substrates. Two RMA fluxes were evaluated. The Bi-containing alloys generally gave the lowest wetting results. Wetting of all the alloys was very sensitive to the flux chemistry. The A35 fluxed samples had significantly better wetting than the K197 samples with larger meniscus heights, maximum wetting forces, and wetting rates and shorter wetting times. The meniscus terminations of the A35 samples were also more clearly defined and less variable.

40In-40Sn-20Pb was identified as an intermediate melting solder alloy for step soldering. Samples fluxed in A35 and dipped into In-Sn-Pb at 150°C gave wetting results similar to the control samples using 62.5Sn-36.1Pb-1.4Ag. The reaction layer between the In-Sn-Pb solder and the Sn-Ni plated samples was identified as Ni_3Sn_4. Wetting of the In-Sn-Pb alloy was limited with the K197 flux at 150°C, although significantly improved at 200°C.

ACKNOWLEDGEMENTS

The authors would like to acknowledge the work of Jerry Rejent, Alice Kilgo, and Bonnie McKenzie of Sandia National Laboratories in Albuquerque, New Mexico. The manuscript was reviewed by John Stephens, also of Sandia National Laboratories.

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Table 1 - Low to Intermediate Melting Solder Alloys for Wettability Experiments

Composition, wt. %	Melt Range, °C	Coupon Preheat, °C	Soldering Temperature, °C
49Bi-21In-18Pb-12Sn	58	none and 120	80 and 150
50In-50Sn	118-125	120	145
40In-40Sn-20Pb	120-130	120	150
34Pb-34Sn-32Bi	96-133	120	155
56.84Bi-41.16Sn-2Pb	128-133	120	155
97In-3Ag	146	130	165
80In-15Pb-5Ag	148-149	130	170
70Sn-18Pb-12In	153-161	140	180
62.5Sn-36.1Pb-1.4Ag	179	150	200

Table 2 - Wettability Data for Low to Intermediate Melting Solder Alloys

Composition, wt. %	Flux	Meniscus Height, mm	Maximum Wetting Force, µN/mm	Wetting Rate, µN/mm-s	Times: 90° Wetting, s	Times: Maximum Wetting, s
49Bi-21In-18Pb-12Sn*	K197	---------------------------- very poor wetting, data not anaylzed ---------------------------				
" "	A35	2.55 ± 0.07	184 ± 45.3	119 ± 50.5	0.8 ± 0.7	6.4 ± 4.7
50In-50Sn	K197	1.29 ± 0.51	114 ± 101.0	36 ± 40.0	6.0 ± 5.7	14.5 ± 2.8
" "	A35	2.67 ± 0.34	430 ± 34.4	332 ± 59.1	0.8 ± 0.1	7.0 ± 1.8
40In-40Sn-20Pb	K197	0.57 ± 0.07	113 ± 63.2	41 ± 28.4	2.8 ± 1.8	14.3 ± 2.9
" "	A35	3.09 ± 0.17	407 ± 27.3	289 ± 40.7	0.2 ± 0.1	6.3 ± 1.8
34Pb-34Sn-32Bi	K197	1.42 ± 0.46	58 ± 67.0	20 ± 39.0	8.8 ± 5.2	16.8 ± 2.9
" "	A35	2.53 ± 0.27	310 ± 66.3	253 ± 73.0	0.8 ± 0.2	6.3 ± 1.3
56.84Bi-41.16Sn-2Pb	K197	2.08 ± 0.37	174 ± 64.0	44 ± 37.5	3.6 ± 3.7	15.6 ± 5.7
" "	A35	2.64 ± 0.19	312 ± 58.0	206 ± 78.0	0.4 ± 0.2	5.9 ± 2.3
97In-3Ag	K197	2.02 ± 0.63	28 ± 19.0	8 ± 2.8	3.5 ± 2.4	12.7 ± 1.4
" "	A35	3.30 ± 0.19	427 ± 40.5	301 ± 61.9	0.4 ± 0.2	11.2 ± 5.5
80In-15Pb-5Ag	K197	2.93 ± 0.42	154 ± 107.0	13 ± 4.9	3.0 ± 1.2	18.1 ± 2.2
" "	A35	3.21 ± 0.11	433 ± 42.4	243 ± 69.2	0.2 ± 0.1	12.9 ± 2.6
70Sn-18Pb-12In	K197	3.08 ± 0.21	120 ± 42.7	16 ± 17.8	4.9 ± 2.9	18.9 ± 0.8
" "	A35	3.06 ± 0.19	404 ± 39.8	189 ± 48.7	0.2 ± 0.1	5.7 ± 1.6
62.5Sn-36.1Pb-1.4Ag	K197	2.82 ± 0.10	188 ± 45.1	61 ± 28.6	1.6 ± 0.6	16.8 ± 2.2
" (control) "	A35	2.96 ± 0.18	375 ± 23.1	207 ± 65.4	0.1 ± 0.1	2.7 ± 0.3

* 49Bi-21In-18Pb-12Sn wetting data for 150°C test temperature only; no wetting observed at 80°C.

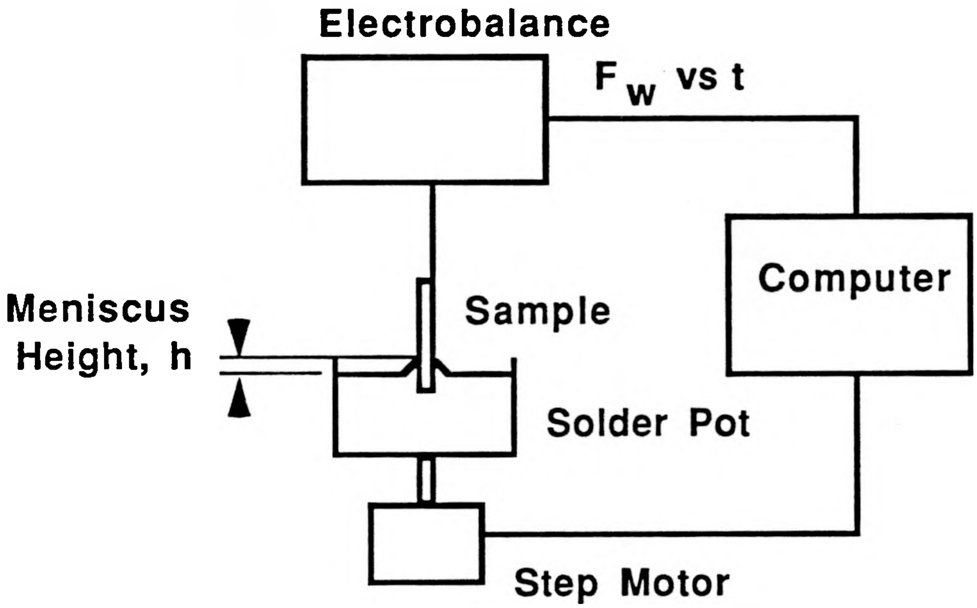
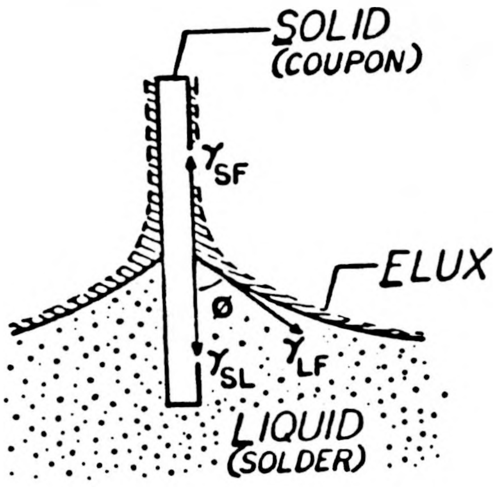


Figure 1. Schematic of the Sandia-designed Solder Wettability Test System



$$\gamma_{SF} - \gamma_{SL} = \gamma_{LF} \cos \phi$$

Figure 2. Schematic of the liquid solder, base metal, and flux surface tension equilibrium conditions for wetting of a vertical plate.

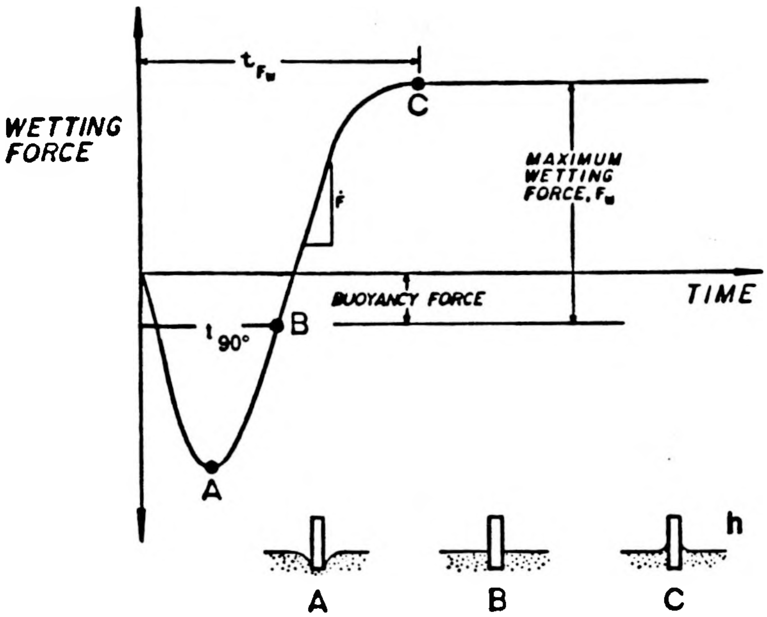


Figure 3. Typical wetting curve of force versus time measured by the wetting balance technique.

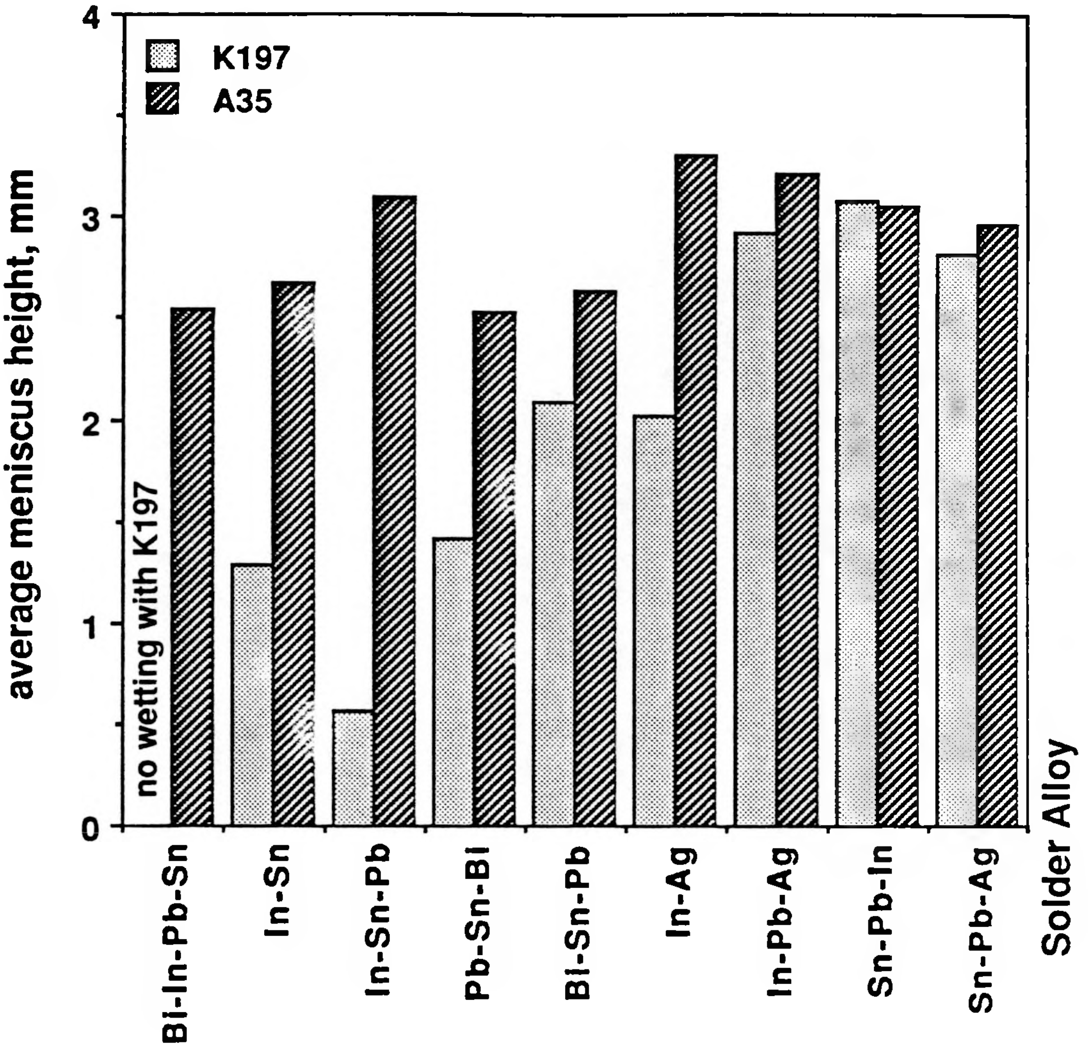


Figure 4. Average meniscus height as a function of solder alloy and flux for Sn-Ni plated aluminum alloy wetting substrates.

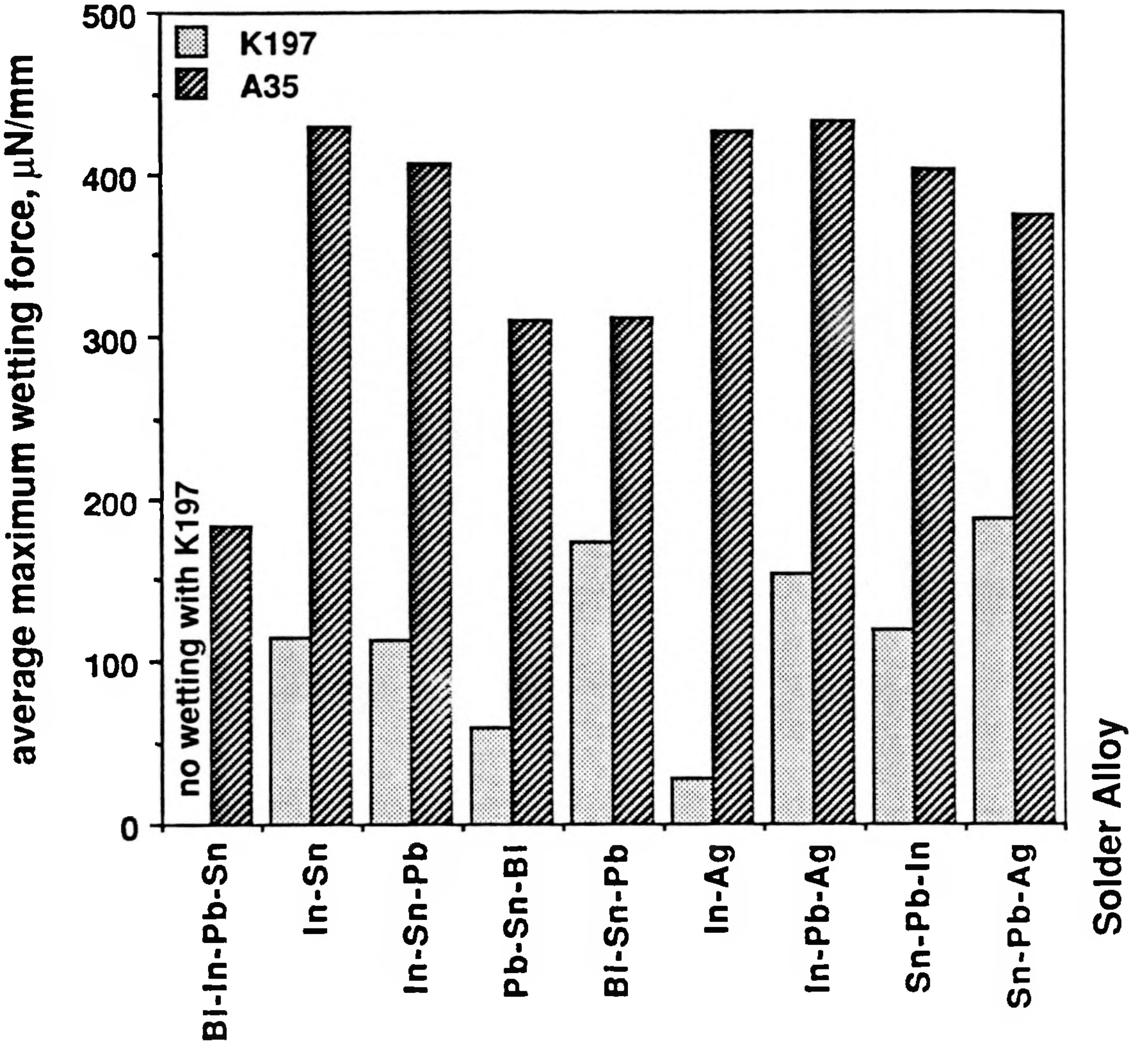


Figure 5. Average maximum wetting force as a function of solder alloy and flux for Sn-Ni plated aluminum alloy wetting substrates.

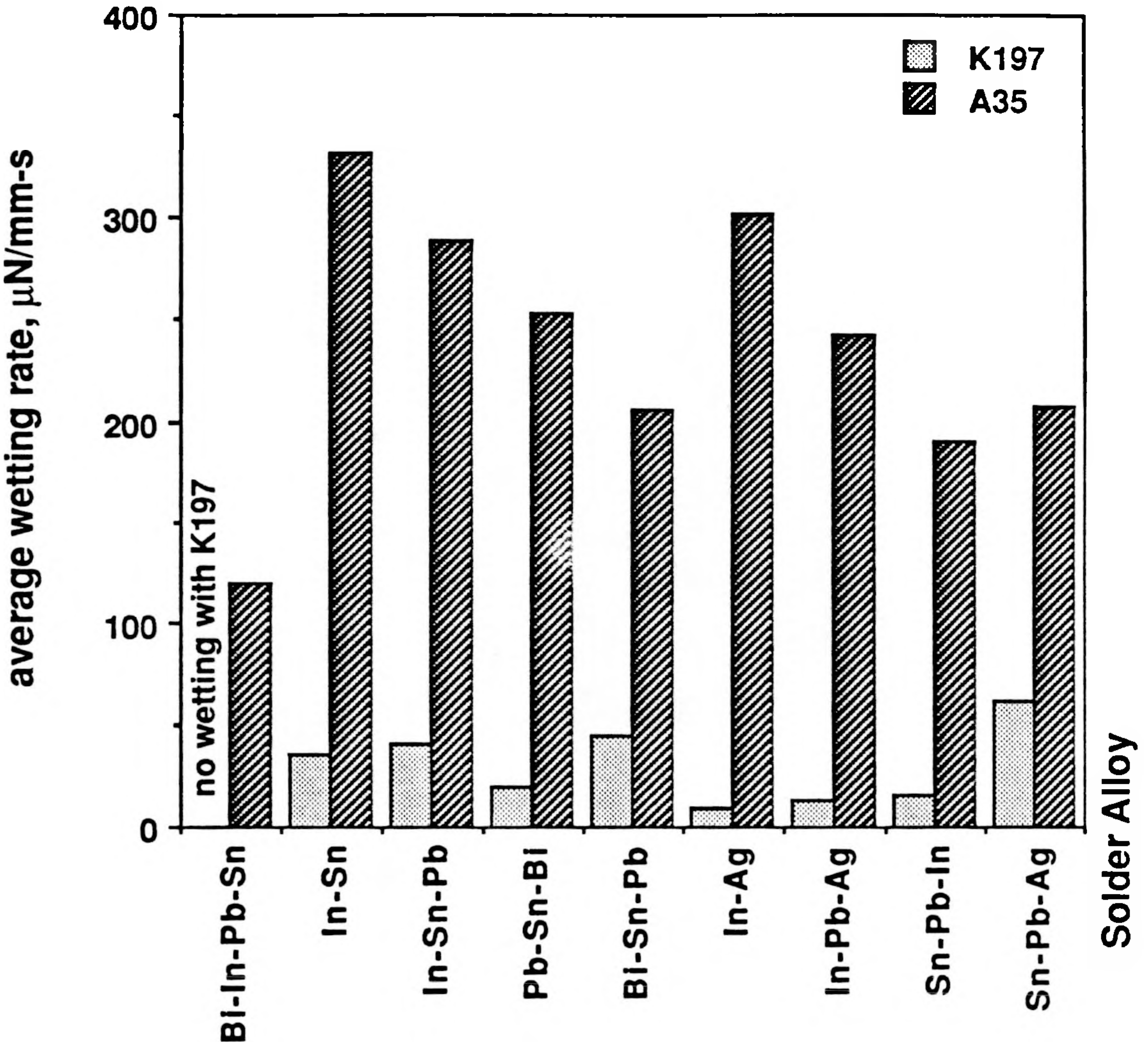


Figure 6. Average wetting rate as a function of solder alloy and flux for Sn-Ni plated aluminum alloy wetting substrates.

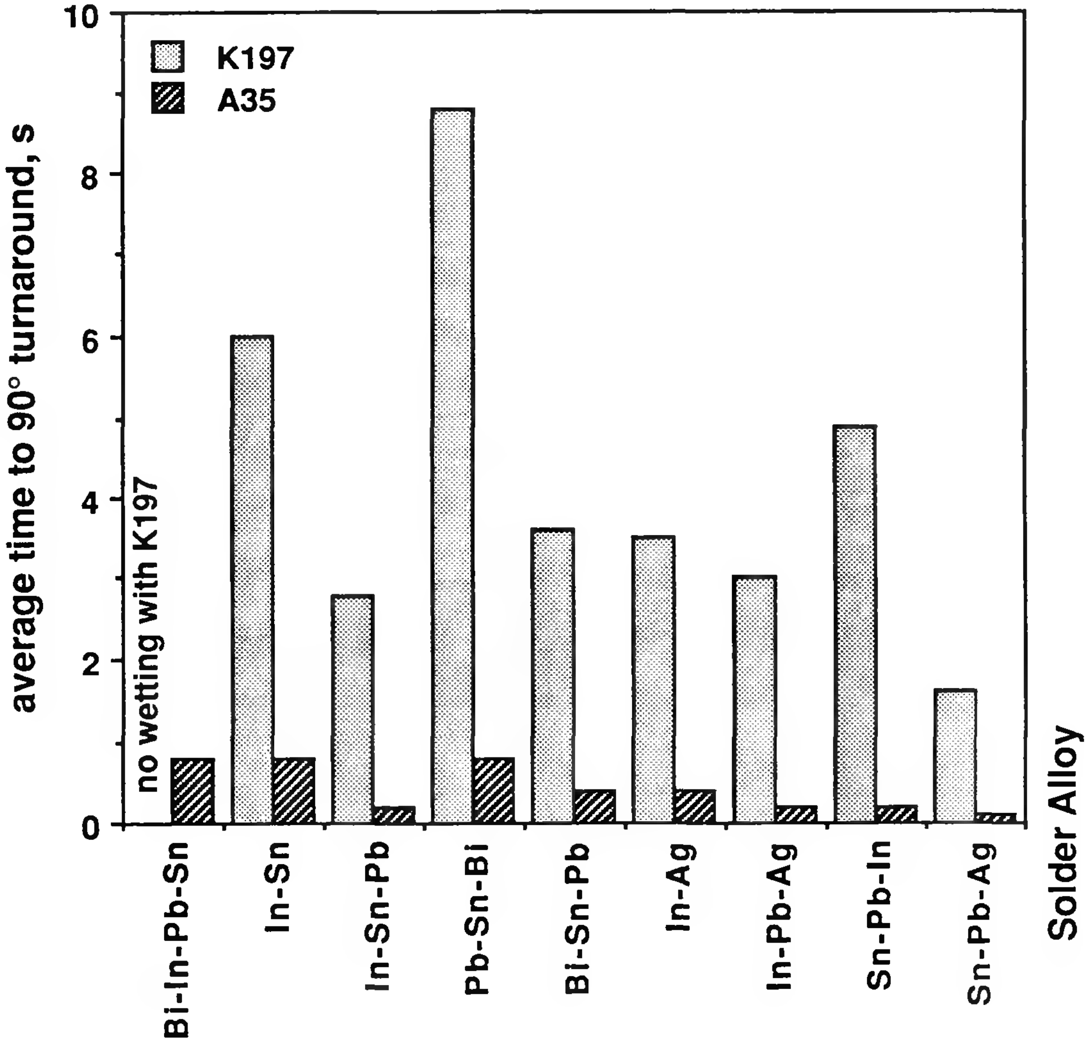


Figure 7. Average time to 90° turnaround as a function of solder alloy and flux for Sn-Ni plated aluminum alloy wetting substrates.

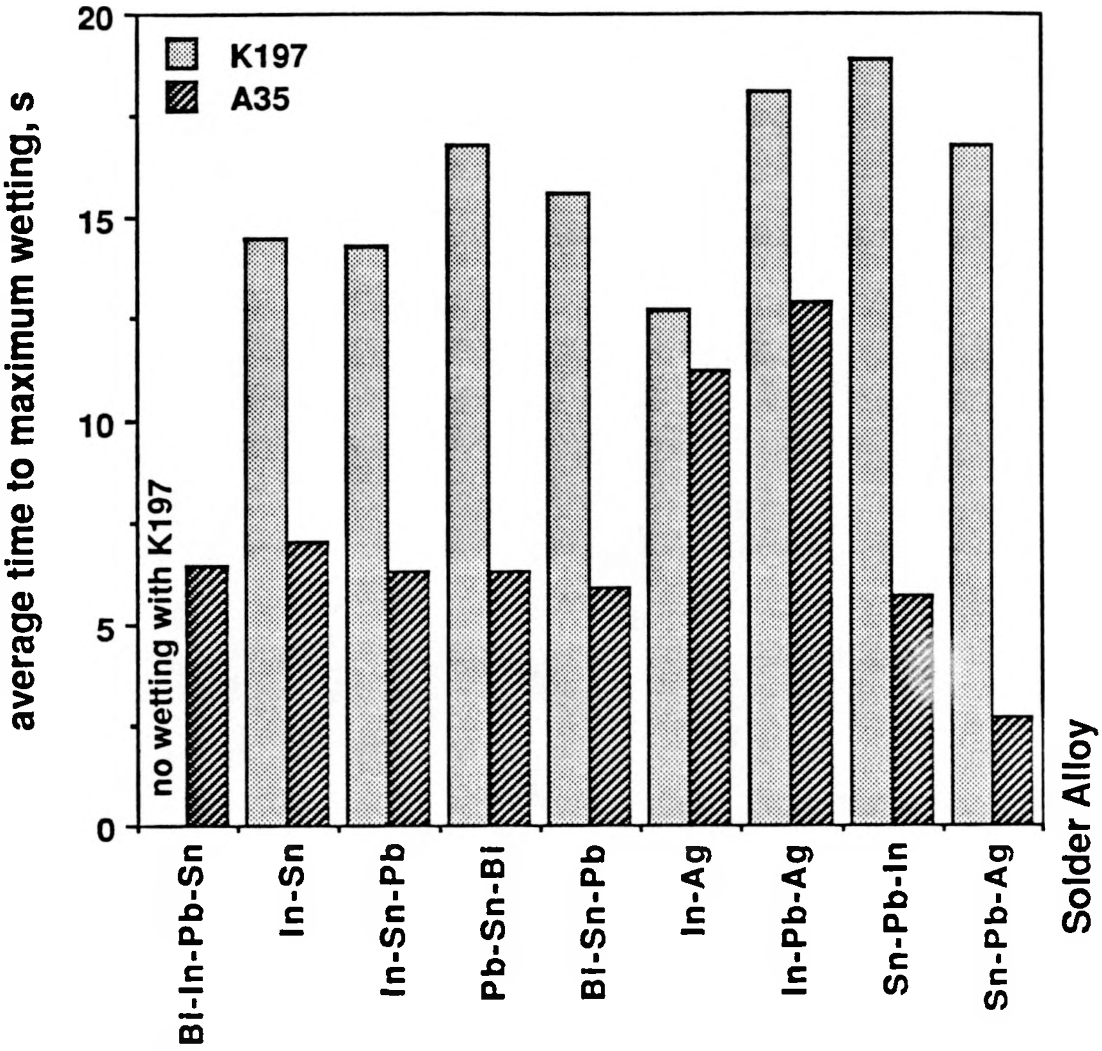


Figure 8. Average time to maximum wetting as a function of solder alloy and flux for Sn-Ni plated aluminum alloy wetting substrates.

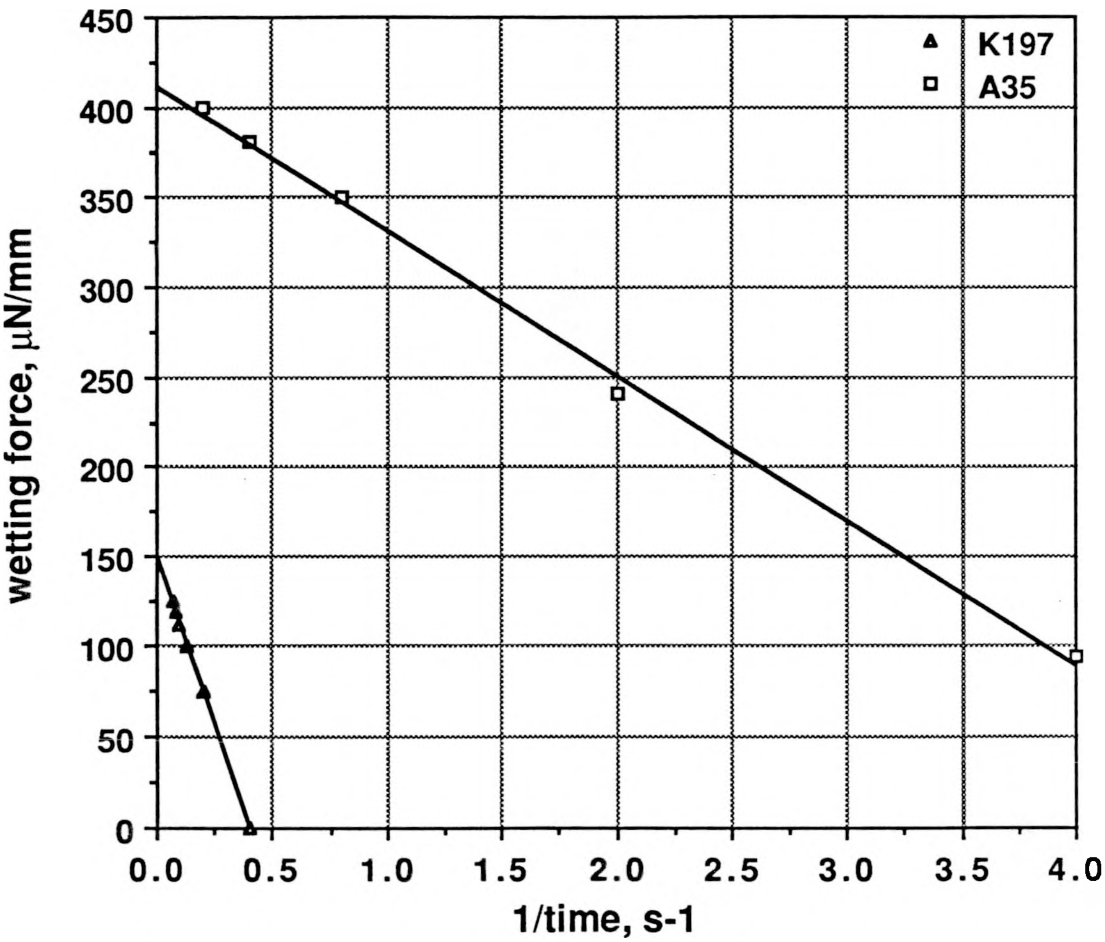
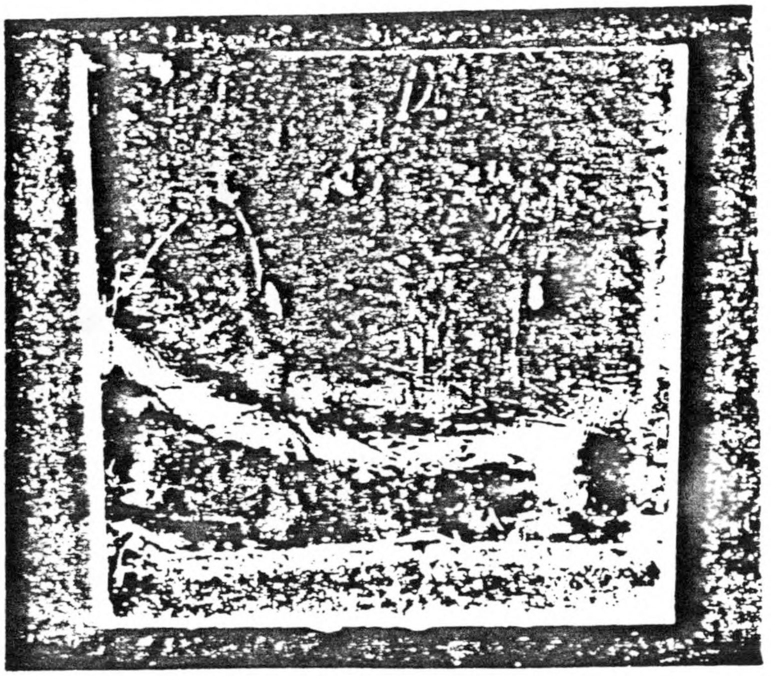
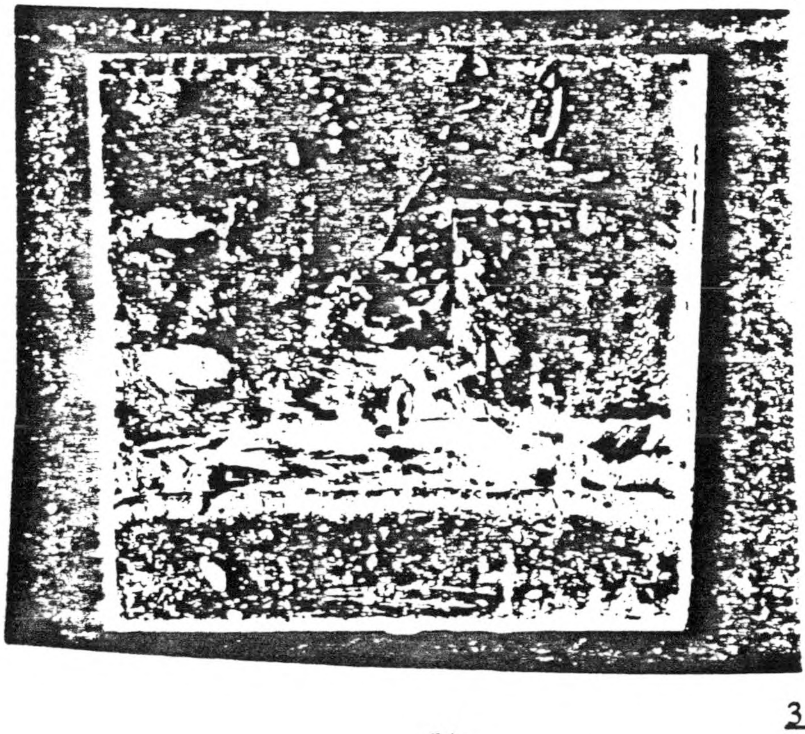


Figure 9. Wetting force as a function of time^{-1} for the K197 and A35 RMA fluxes at 150°C with the 40In-40Sn-20Pb solder alloy.



(a)



(b)

Figure 10. Optical photographs of Sn-Ni plated aluminum alloy substrates that were fluxed with K197 and dipped in a 40In-40Sn-20Pb solder alloy at (a) 150°C and (b) 200°C.

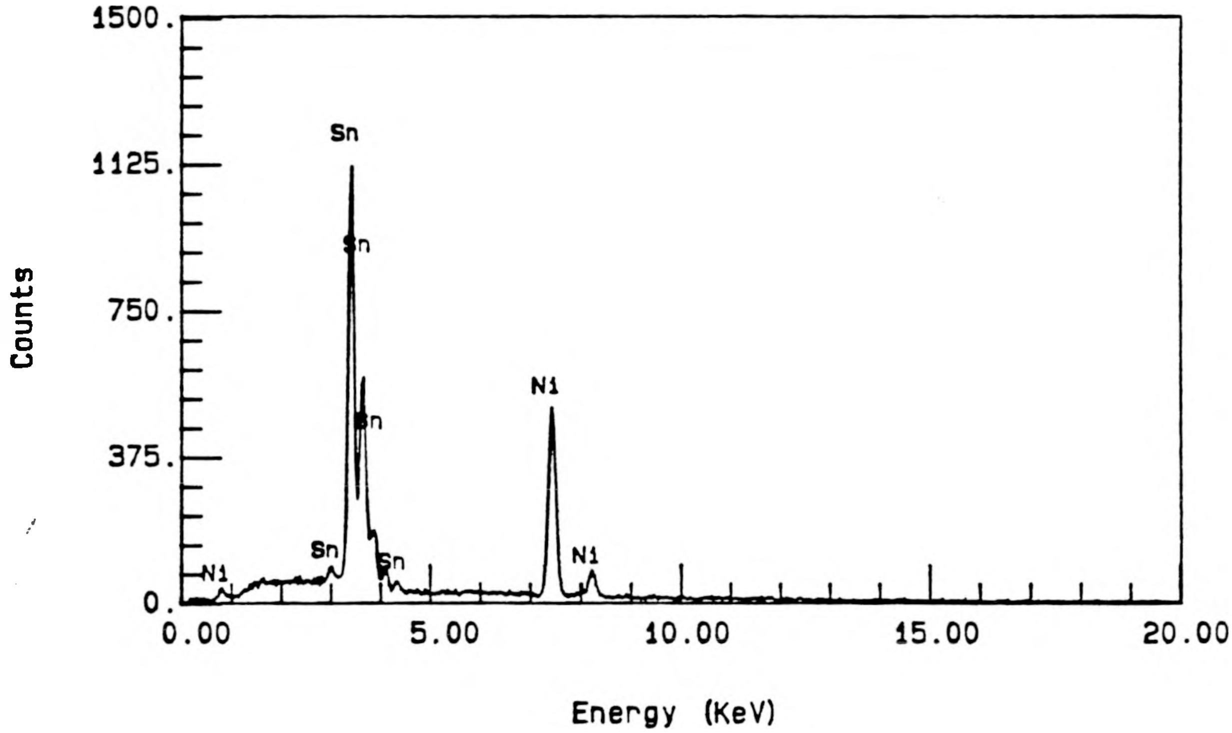


Figure 11. Energy dispersive spectrometry spectra of the Ni-Sn reaction layer between a 40In-40Sn-20Pb solder alloy and a plated Ni interface.

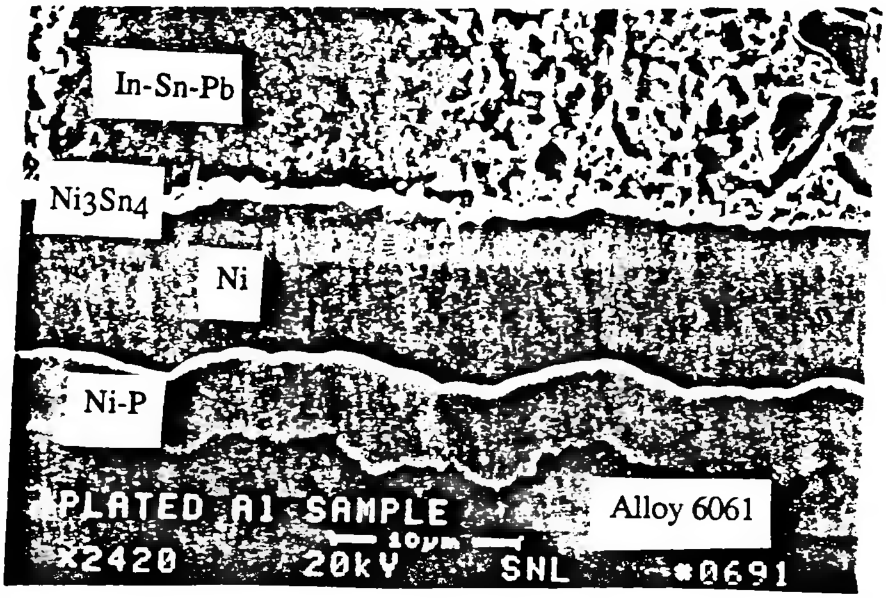


Figure 12. Secondary electron micrograph of a Sn-Ni plated aluminum alloy sample that was fluxed with A35 and dipped in 40In-40Sn-20Pb solder alloy at 150°C.