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Pb-Free Solders: Part III. Wettability Testing of Sn-Ag-Cu-Bi Alloys with Sb Additions

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Maximum bubble pressure, dilatometric, and meniscographic methods were used in the investigations of the surface tensions, densities, wetting times, wetting forces, contact angles, and solder-flux interfacial tensions of liquid Sn-Ag-Cu-Bi alloys with various additions of Sb. Density and surface tension measurements were conducted in the temperature range 230 to 900 °C. Surface tensions at 250 °C were measured in air and under a protective atmosphere of Ar-H_2 and were combined with data from meniscographic studies done under air or with a protective flux. Meniscographic data with a nonwetted Teflon substrate provided data on solder-flux interfacial tensions, and meniscographic data with a Cu substrate allowed determinations of wetting times, wetting forces, and calculations of contact angles. Additions of Sb to quaternary Sn-Ag-Cu-Bi alloys improve wettability, move the parameters closer to those of traditional solders, and affirm, as found in previous studies of Bi additions to the Sn-Ag-Cu near-eutectic compositions, that interfacial tensions and contact angles are the two parameters most important as a metric of wettability. However, in contrast to results found in studies of quaternary Sn-Ag-Cu-Bi alloys, the changes in quinary Sn-Ag-Cu-Bi-Sb alloys of interfacial tensions and contact angles do not correlate with decreasing wetting time and increasing wetting force.

Keywords density, meniscographic studies, Pb-free solders, surface tension

1. Introduction

Part 1 of these extended studies[1] on Bi additions to two near-eutectic alloys Sn-2.76Ag-0.46Cu and Sn-3.13Ag-0.74Cu (the numerical values represent at.%) have combined surface tension and density data with interfacial tension and contact angle data to find links between basic data and industrial application of Pb-free solders. In Part 2 of this investigative program,[2] the modeling of surface tensions by the Butler method[3] was compared with experimental results and was extended to a calculation of the phase diagram and simulation of solidification with the use of the ADAMIS database;[4] these studies were done in cooperation with Tohoku University. Both Parts 1 and 2 clearly indicate that changes of surface tension under a protective atmosphere, interfacial tension, surface tension in air, and the calculated contact angle correlate with wetting force and wetting time over an extensive range of concentrations of alloys starting with the binary and ternary eutectics, Sn-Ag and Sn-Ag-Cu-Bi, and proceeding through four quaternary Sn-Ag-Cu-Bi alloys.

The present report is Part 3 of an overall investigation that deals with measurements of the surface tension by the maximum bubble pressure method, density by dilatometry under a protective atmosphere of Ar-H_2, and both interfacial tension and surface tension in air from meniscographic studies that also provide wetting force and wetting time and allows a calculation of contact angle for liquid quinary Sn-Ag-Cu-Bi-Sb alloys. The main purpose of the current investigative program was to prove the previous initial results presented at the 2005 Annual TMS meeting,[5] but it was found that, contrary to the results from studies of quaternary Sn-Ag-Cu-Bi alloys, the quinary Sn- Ag-Cu-Bi-Sb alloys do not show changes in interfacial tensions and contact angles that correlate with decreasing wetting time and increasing wetting force.

2. Experimental Procedure

Details connected with measurements of the surface tensions and densities under a protective atmosphere of Ar-H_2 using the maximum bubble pressure method and a dilatometric technique as well as meniscographic measurements in air and under a protective atmosphere to determine interfacial tensions and surface tensions, wetting forces, and wetting times with subsequent calculations of contact angles have been summarized in previous papers[1,6-8] In the present report, surface tensions and densities under a protective atmosphere of Ar-H_2 were measured over the

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temperature range 250 to 900 °C by the maximum bubble pressure method, and data are presented in Table 1 and are shown graphically in Fig. 1(a) and (b). Two plots are used to avoid a mixed jumble of experimental points. For comparison purposes, data for the binary Sn3.8Ag near-eutectic alloy are included in both figures and are shown by dashed lines. Also included in Fig. 1 (a) are data for the ternary Sn3.13Ag0.74Cu near-eutectic alloy and are shown by filled points; similarly in Fig. 1(b), data for the Sn2.76Ag0.46Cu near-eutectic alloy are shown by filled points. An earlier report[7] is the source of these ternary data. In all cases, the data points were fitted by the least-squares method, and lines in the figures show these fits. On average, the data points scatter ~2 to 3% along the fits. Results for dilatometric density measurements are shown in Table 2 and graphically in Fig. 2(a) and (b). Again the duality in the graphical displays is to make the data more easily distinguishable, and data for the Sn-Ag and Sn-Ag-Cu eutectic alloys are again shown for comparison. The thermodynamically important molar volumes were derived from the density data, and the resultant values at 250 C and at 900 °C for a variety of different compositions are shown in Fig. 3(a) and (b).

Table 1 Temperature dependencies of the surface tension, σ, of four Sn-Ag-Cu-Bi alloys together with eight Sn-Ag-Cu-Bi-Sb alloys with comparative values for two near-eutectic alloys and eutectic Sn-Ag with uncertainties in slope and intercept (in protective atmosphere Ar + H_2)

Alloys(a), mass%	Alloys(a), at.%	$\sigma = A + BT$, mN/m	$\sigma_{(250\ C)}$, mN/m	Error (A), mN/m	Error (B), mN/m^1 · K
Sn3.65Ag(b)	Sn3.8Ag	= 585.1 − 0.0881T	539.0 ± 8.2	±8.1	±0.0086
Sn2.56Ag0.26Cu(b)	Sn2.76Ag0.46 Cu	= 587.0 − 0.0964T	536.6 ± 7.8	±7.4	±0.0086
Sn2.86Ag0.40Cu(b)	Sn3.13Ag0.74Cu	= 582.1 − 0.0867T	536.7 ± 6.9	±5.8	±0.0068
Sn2.77Ag0.25Cu6.91Bi(c)	Sn3.13Ag0.48Cu4.02Bi	= 555.3 − 0.0623T	522.7 ± 6.2	±5.1	±0.0062
Sn2.56Ag0.27Cu11.45Bi(c)	Sn2.95Ag0.53Cu6.81Bi	= 540.1 − 0.0541T	511.9 ± 4.5	±4.8	±0.0053
Sn2.4Ag0.45Cu6.65Bi(c)	Sn2.7Ag0.86Cu3.86Bi	= 574.6 − 0.0778T	533.9 ± 8.8	±7.8	±0.0094
Sn2.49Ag0.52Cu11.17Bi(c)	Sn2.86Ag1.01Cu6.62Bi	= 550.4 − 0.0581T	520.0 ± 7.9	±7.3	±0.0082
Sn3.17Ag0.26Cu5.19Bi3.02Sb	Sn3.55Ag0.5Cu3Bi3Sb	= 561.5 − 0.0880T	515.5 ± 5.9	±7.2	0.0086
Sn3.10Ag0.26Cu5.19Bi5.04Sb	Sn3.48Ag0.5Cu3Bi5Sb	= 538.7 − 0.0823T	495.7 ± 8.8	±7.8	±0.0094
Sn3.03Ag0.26Cu8.52Bi2.98Sb	Sn3.48Ag0.5Cu5Bi3Sb	= 523.2 − 0.0782T	482.3 ± 7.4	±7.7	±0.0088
Sn2.99Ag0.26Cu8.52Bi4.96Sb	Sn3.40Ag0.5Cu5Bi5Sb	= 531.5 − 0.0693T	495.3 ± 6.3	±7.2	±0.0089
Sn3.16Ag0.53Cu5.20Bi3.03Sb	Sn3.53Ag1Cu3Bi3Sb	= 552.3 − 0.0645T	518.6 ± 5.9	±7.1	±0.0081
Sn3.09Ag0.53Cu5.20Bi5.00Sb	Sn3.46Ag1Cu3Bi5Sb	= 525.2 − 0.0492T	499.4 ± 3.8	±3.8	±0.0042
Sn3.05Ag0.52Cu8.54Bi2.99Sb	Sn3.46Ag1Cu5Bi3Sb	= 515.6 − 0.0489T	490.0 ± 6.1	±5.7	±0.0063
Sn2.98Ag0.52Cu8.54Bi4.97Sb	Sn3.38Ag1Cu5Bi5Sb	= 511.8 − 0.0486T	486.4 ± 4.2	±4.4	±0.0051

(a) Numbers preceding an element. (b) Ref 7. (c) Ref 1

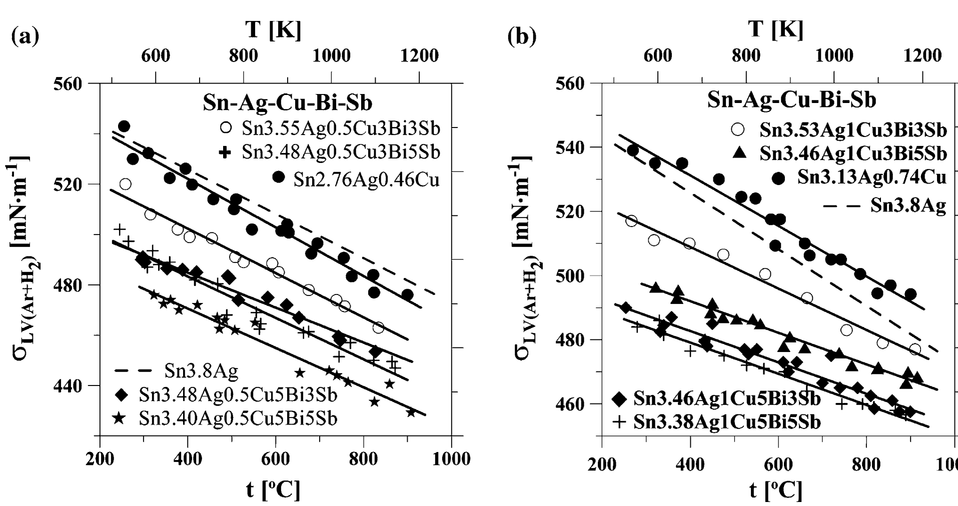

Fig. 1 Temperature dependence of surface tension of eight quinary Sn-Ag-Cu-Bi-Sb alloys. The data are divided into two plots to avoid a confusion of data points. (a) Data for four alloys with 0.5 at. % Cu. (b) Data for four alloys with 1 at.% Cu are shown (see Table 1). The various symbols represent individual data points. The solid lines represent least-squares fits to the data points. Data for eutectic Sn-Ag solder are shown in both plots as a dashed line and the solid points are for a ternary alloy with 0.46 at.% Cu and in (b) for a 0.74 at.% Cu

Table 2 Temperature dependencies of the density of four Sn-Ag-Cu-Bi alloys together with eight Sn-Ag-Cu-Bi-Sb alloys with comparative values for two near-eutectic alloys and eutectic Sn-Ag with uncertainties in slope and intercept (in protective atmosphere Ar + H_2)

Alloys(a), mass%	Alloys(a), at.%	$\rho = A + BT$, g/cm^3	$\rho_{(250\ C)}$, g/cm^3	Error (A), g/cm^3	Error (B), g/cm$^3 \cdot$ K
Sn3.65Ag(b)	Sn3.8Ag	= 7.445 − 0.000739T	7.059 ± 0.054	±0.042	±0.000051
Sn2.56Ag0.26Cu(b)	Sn2.76Ag0.46 Cu	= 7.458 − 0.000691T	7.096 ± 0.037	±0.050	±0.000058
Sn2.86Ag0.40Cu(b)	Sn3.13Ag0.74Cu	= 7.462 − 0.000703T	7.094 ± 0.027	±0.028	±0.000035
Sn2.77Ag0.25Cu6.91Bi(c)	Sn3.13Ag0.48Cu4.02Bi	= 7.539 − 0.000802T	7.119 ± 0.043	±0.049	±0.000059
Sn2.56Ag0.27Cu11.45Bi(c)	Sn2.95Ag0.53Cu6.81Bi	= 7.678 − 0.000836T	7.241 ± 0.121	±0.126	±0.000148
Sn2.4Ag0.45Cu6.65Bi(c)	Sn2.7Ag0.86Cu3.86Bi	= 7.542 − 0.000767T	7.141 ± 0.078	±0.092	±0.000103
Sn2.49Ag0.52Cu11.17Bi(c)	Sn2.86Ag1.01Cu6.62Bi	= 7.722 − 0.000859T	7.272 ± 0.082	±0.096	±0.000105
Sn3.17Ag0.26Cu5.19Bi3.02Sb	$(SnAg)_{eut}$ + 0.5Cu + 3Bi + 3Sb	= 7.540 − 0.000751T	7.147 ± 0.027	±0.037	±0.000049
Sn3.10Ag0.26Cu5.19Bi5.04Sb	$(SnAg)_{eut}$ + 0.5Cu + 3Bi + 5Sb	= 7.409 − 0.000736T	7.024 ± 0.072	±0.092	±0.000100
Sn3.03Ag0.26Cu8.52Bi2.98Sb	$(SnAg)_{eut}$ + 0.5Cu + 5Bi + 3Sb	= 7.724 − 0.000866T	7.271 ± 0.063	±0.093	±0.000114
Sn2.99Ag0.26Cu8.52Bi4.96Sb	$(SnAg)_{eut}$ + 0.5Cu + 5Bi + 5Sb	= 7.611 − 0.000805T	7.190 ± 0.057	±0.081	±0.000095
Sn3.16Ag0.53Cu5.20Bi3.03Sb	$(SnAg)_{eut}$ + 1Cu + 3Bi + 3Sb	= 7.554 − 0.000756T	7.159 ± 0.036	±0.042	±0.000050
Sn3.09Ag0.53Cu5.20Bi5.00Sb	$(SnAg)_{eut}$ + 1Cu + 3Bi + 5Sb	= 7.472 − 0.000719T	7.095 ± 0.027	±0.041	±0.000045
Sn3.05Ag0.52Cu8.54Bi2.99Sb	$(SnAg)_{eut}$ + 1Cu + 5Bi + 3Sb	= 7.748 − 0.000821T	7.318 ± 0.083	±0.096	±0.000104
Sn2.98Ag0.52Cu8.54Bi4.97Sb	$(SnAg)_{eut}$ + 1Cu + 5Bi + 5Sb	= 7.645 − 0.000757T	7.249 ± 0.046	±0.066	±0.000078

(a) Numbers preceding element. (b) Ref 7. (c) Ref 1

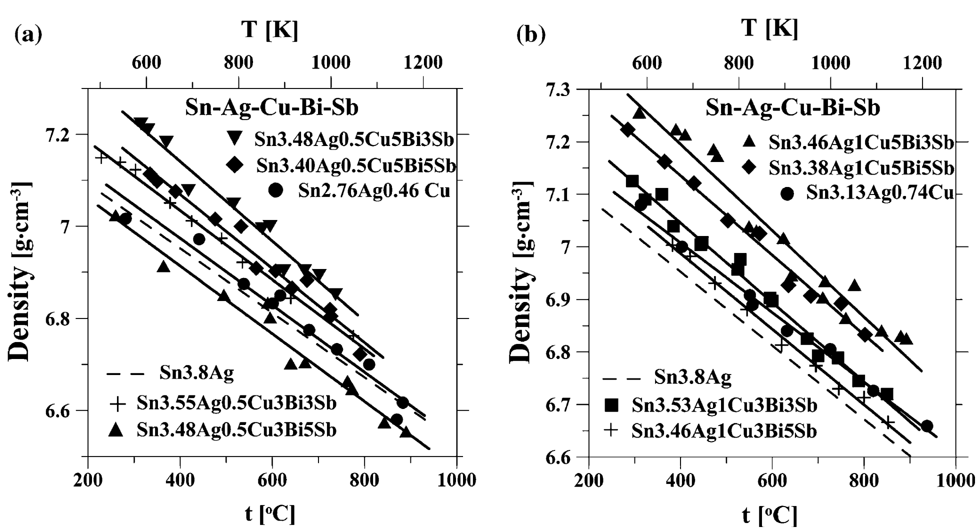

Fig. 2 Temperature dependence of density of eight quinary Sn-Ag-Cu-Bi-Sb alloys. The data are divided into two plots to avoid a confusion of data points. (a) Data for four alloys with 0.5 at.% Cu. (b) Data for four alloys with 1 at.% Cu are shown (see Table 1). The various symbols represent individual data points. The solid lines represent least-squares fits to the data points. Data for eutectic Sn-Ag solder are shown in both plots as a dashed line and the solid points are for a ternary alloy with 0.46 at.% Cu and in (b) for a 0.74 at.% Cu

Measurements were made in the manner described in the initial report.[1] The maximum bubble pressure measurements under a protective atmosphere provide data for surface tensions, $\sigma_{LV(Ar-H_2)}$, meniscographic measurements with a Cu substrate provide data for surface tension under air, $\sigma_{LV(air)}$, and the meniscographic method with a Teflon substrate provides data for interfacial tension, σ_{LF}, for a solder/flux interface. Figures 4 and 5 illustrate data that was accumulated for these three parameters from a number of related alloys during the present series of measurements. On the ordinate axis of the figures are found values at 250 °C for each of the three different tensions for the binary Ag-Sn eutectic, for an Ag-Sn-Cu ternary alloy with the alloy composition changing slightly between Fig. 4 and 5, and for an initial Ag-Sn-Cu-Bi quaternary alloy with 3 at.% Bi in Fig. 4(a) and 5(a) and 5 at.% Bi in Fig. 4(b) and 5(b) and with the Cu content shifting from 0.5 at.% in Fig. 4(a) and (b) to 1 at.% in Fig. 5(a) and (b). The abscissa in each case then shows the change in the three tension values as Sb additions change the quaternary alloy into quinary alloys.

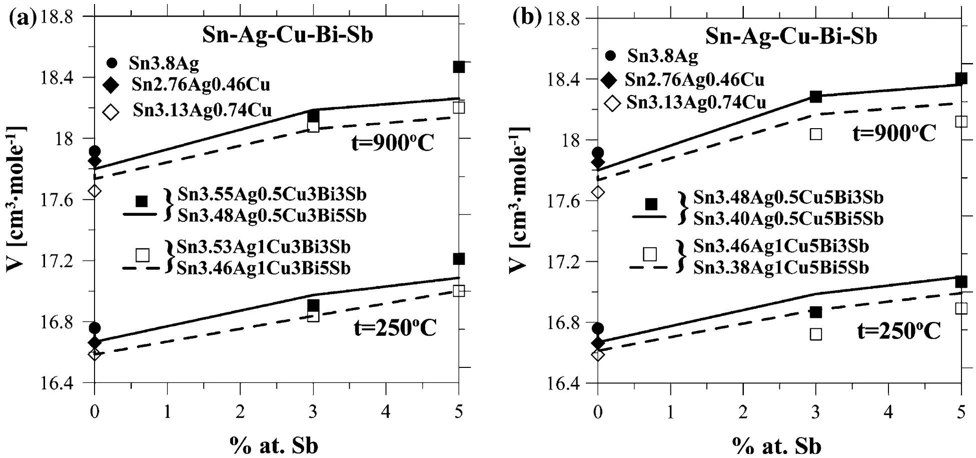

Fig. 3 Isotherms of the molar volume of the quinary Sn-Ag-Cu-Bi-Sb alloys compared with binary eutectic Sn-Ag and ternary alloys. (a) Sn2.76Ag0.46Cu and Sn3.13Ag0.74Cu with 3 at.% Bi. (b) Sn2.76Ag0.46Cu and Sn3.13Ag0.74Cu with 5 at.% Bi

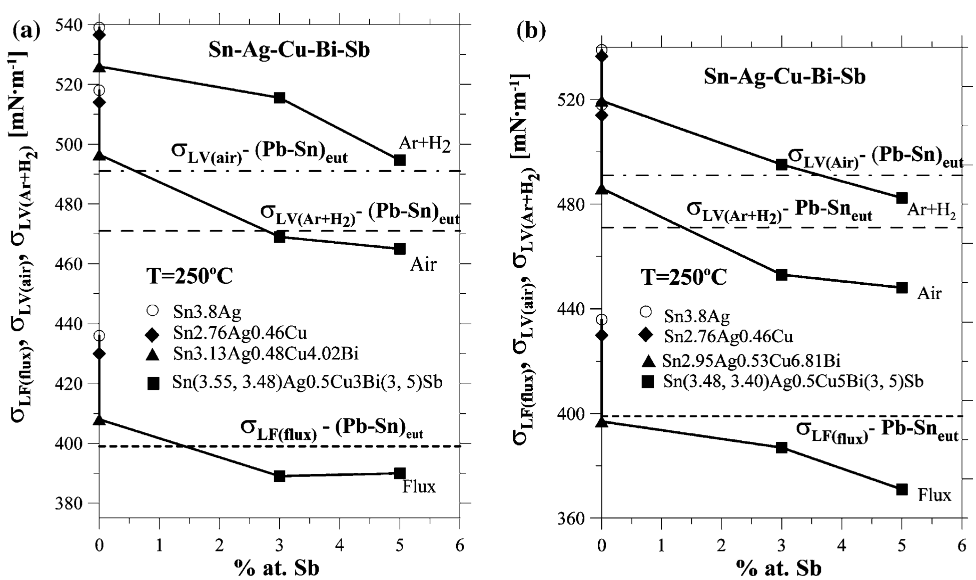

Fig. 4 Comparison of surface tension σ_{LV} from the maximum bubble pressure method (upper plots) under Ar-H_2 protective atmosphere with meniscographic data (middle plot) in air. The lower plots are for interfacial tension σ_{LF} from meniscographic data for the solder/flux interface. (a) and (b) are for the same ternary alloy Sn2.76Ag0.46Cu. According to Table 1, in (a) the following alloys are plotted Sn(3.55, 3.48)Ag0.5Cu3Bi(3.5)Sb and in (b) Sn(3.48, 3.40)Ag0.5Cu5Bi(3.5)Sb. Sn-Pb values are shown for comparison

The values for the Pb-Sn eutectic are dashed across the plots for easy comparison. The 250 °C temperature was chosen because it is a commonly used temperature for industrial soldering.

Meniscograpic studies also evaluate wetting time and wetting force that are indicative of wettability. Thus the present measurements added to previous measurements provide an extensive set of data that may be used with the Miyazaki method[9] to calculate contact angles and thus proceed toward the development of a metric for wettability. In Table 3, the final values for wetting time, wetting force, contact angle, interfacial tension, surface tension with flux, and surface tension under a protective atmosphere are tabulated for all alloys that have been studied beginning with Sn-Ag and ending with eight Sn-Ag-Cu-Bi-Sb alloys from this report. This provides an indication of the effects of Bi and Sb additions to solders currently in use.

3. Discussion

The experimental results show that Sb, analogously to results found in the previous report[1] for Bi, decreases the

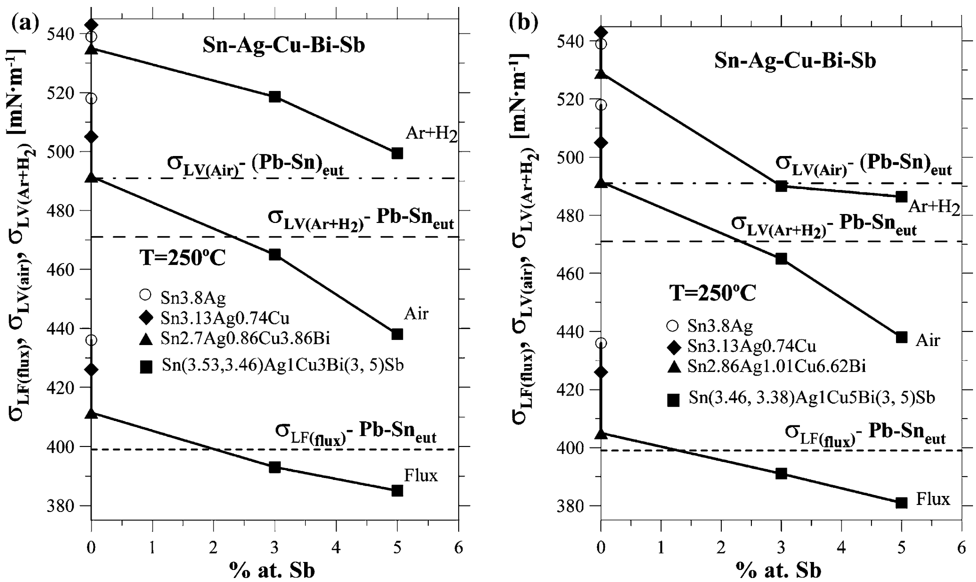

Fig. 5 Comparison of surface tension σ_{LV} from the maximum bubble pressure method (upper plots) under Ar-H_2 protective atmosphere with meniscographic data (middle plot) in air. The lower plots are for interfacial tension σ_{LF} from meniscographic data for the solder/flux interface. (a) and (b) are for the same ternary alloy Sn3.13Ag0.74Cu. According to Table 1, in (a) the following alloys are plotted Sn(3.5, 3.46)Ag1Cu3Bi(3.5)Sb and in (b) Sn(3.46, 3.38)Ag1Cu5Bi(3.5)Sb. Sn-Pb values are shown for comparison

Table 3 Results of the meniscographic studies with the surface tension from maximum bubble method

	Wetting time (τ_z), s	Wetting force after 2 s F2, mN	Contact angle after 3 s (θ_3), degrees	Interfacial tension solder/flux(a), mN/m	Surface tension air(a), mN/m	Surface tension Ar + H_2, mN/m
Sn3.65Ag(b)	0.38 ± 0.03	5.38 ± 0.18	47 ± 1	436 ± 5	518 ± 6	539.0 ± 8.2
Sn2.56Ag0.26Cu(b)	0.40 ± 0.03	5.52 ± 0.20	46 ± 3	430 ± 6	514 ± 8	536.6 ± 7.8
Sn2.86Ag0.40Cu(b)	0.37 ± 0.02	5.40 ± 0.17	46 ± 2	426 ± 4	505 ± 4	536.7 ± 6.9
Sn2.77Ag0.25Cu6.91Bi(c)	0.21 ± 0.01	6.96 ± 0.02	22 ± 1	399 ± 5	491 ± 8	522.7 ± 6.2
Sn2.56Ag0.27Cu11.45Bi(c)	0.21 ± 0.04	6.72 ± 0.02	25 ± 0	394 ± 4	473 ± 3	511.9 ± 4.5
Sn2.4Ag0.45Cu6.65Bi(c)	0.27 ± 0.02	6.67 ± 0.04	29 ± 1	408 ± 6	488 ± 4	533.9 ± 8.8
Sn2.49Ag0.52Cu11.17Bi(c)	0.18 ± 0.04	6.95 ± 0.12	21 ± 2	397 ± 4	487 ± 6	520.0 ± 7.9
Sn2.76Ag0.27Cu7.44Bi(d)	0.32 ± 0.02	6.97 ± 0.05	23 ± 1	401 ± 4	480 ± 9	520.7 ± 4.2
Sn3.17Ag0.26Cu5.19Bi3.02Sb	0.35 ± 0.04	6.32 ± 0.19	30 ± 3	389 ± 4	469 ± 3	515.5 ± 5.9
Sn3.10Ag0.26Cu5.19Bi5.04Sb	0.41 ± 0.05	6.11 ± 0.06	34 ± 1	390 ± 7	465 ± 9	495.7 ± 8.8
Sn3.03Ag0.26Cu8.52Bi2.98Sb	0.28 ± 0.03	6.39 ± 0.01	28 ± 1	387 ± 8	453 ± 6	482.3 ± 7.4
Sn2.99Ag0.26Cu8.52Bi4.96Sb	0.33 ± 0	6.15 ± 0.02	28 ± 1	371 ± 6	448 ± 14	495.3 ± 6.3
Sn3.16Ag0.53Cu5.20Bi3.03Sb	0.34 ± 0.04	6.40 ± 0.14	30 ± 1	393 ± 7	465 ± 8	518.6 ± 5.9
Sn3.09Ag0.53Cu5.20Bi5.00Sb	0.35 ± 0.03	6.16 ± 0.06	31 ± 1	385 ± 7	438 ± 10	499.4 ± 3.8
Sn3.05Ag0.52Cu8.54Bi2.99Sb	0.30 ± 0.02	6.34 ± 0.09	30 ± 1	391 ± 6	456 ± 3	490.0 ±± 6.1
Sn2.98Ag0.52Cu8.54Bi4.97Sb	0.36 ± 0.03	6.01 ± 0.02	33 ± 1	381 ± 4	458 ± 4	486.4 ± 4.2
SnPb	0.23 ± 0.04	7.66 ± 0.04	0	399 ± 5	491 ± 11	471.4 ± 6.3

(a) Ref 9. (b) Ref 7. (c) Ref 1. (d) Second quaternary alloy with 7.4 mass% Bi to check reproducibility (Ref 1)

surface tension of alloys both in air and under a protective atmosphere. In the earlier report,[1] the influence of Bi additions to the near-ternary eutectic Sn-Ag-Cu alloys was studied. A beneficial influence on wettability and on decreasing the melting temperature seems evident, but a desirable and suitable additive concentration is a compromise of various factors, especially in view of the tendency for "lifting-off" failure as discussed by Ohnuma et al.[2] In the present report, the combined influence of both Bi and Sb are shown in Fig. 4 and 5. The tendency for surface tension and interfacial tension to drop below the binary eutectic level is greatest at a low concentration of Sb, and no lowering at all occurs for surface tension under Ar-H_2 atmosphere. Recently published work concerns Sb additions to Sn-Ag-Cu alloys without the presence of Bi; this work can be found in a *Festschrift*[10] honoring Prof. F. Sommer. The work indicates that an addition of about 12 at.% Sb lowers the surface tension of a Sn-3.3Ag-0.76Sn near-eutectic alloy to the level of the surface tension of the commonly used Sn-Pb eutectic alloy solder.

Examination of the summarized results in Table 3 makes clear that, in agreement with the suggestions of Lopez et al.,[11] surface tension under air, surface tension under protective atmosphere, and interfacial tension together with contact angle correlate with the compositions of Sn-Ag-Cu-Bi alloys. This is also true for the calculated contact angle from interfacial tension,[5] for wetting time, and for wetting force, but only for Bi additions. This correlation is not fulfilled for Sb additions to Sn-Ag-Cu-Bi alloys in the case of wetting force, wetting time, nor for calculated contact angles from wetting force and interfacial tension. It is expected that the conclusions of Lopez et al.[11] would be valid for direct determinations of contact angles by the sessile drop method. Future plans include an analysis, in a manner previously used,[2] of the observation of the crossing of the surface tension of the Sn-Pb eutectic value by the reduction of surface tension of Sn-Ag-Cu-Sb alloys containing ~12 at.% Sb to determine if this reduction is applicable to quaternary alloys. This might include a calculation of the Sn-Ag-Cu-Bi-Sb quinary equilibrium diagram to propose a possible range of Sb additions to quaternary Sn-Ag-Cu-Bi alloys for practical applications. Also the experimental surface tension measured in a protective Ar-H_2 atmosphere will be compared with calculated values by the Butler method;[3] such a calculation would be based on the available thermodynamic data for constituent alloys of the Sn-Ag-Cu-Bi system.

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