JPEDAV (2004) 25:115-121
DOI: 10.1361/15477030418497
1547-7037/$19.00 ©ASM International

$(Sn\text{-}Ag)_{eut}$ + Cu Soldering Materials, Part I: Wettability Studies

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(Submitted 25 September 2003)

The maximum bubble pressure, dilatometric, and meniscographic methods were used in investigations of the surface tension, density, wetting time, wetting force, contact angles, and interfacial tension of liquid $(Sn\text{-}Ag)_{eut}$ and two $(Sn\text{-}Ag)_{eut}$ + Cu alloys (Cu at.% = 0.46 and 0.74). The density and surface tension measurements were conducted in the temperature range from 230 to 950 °C, and the meniscographic investigations were carried out at 252 °C. The resultant values of surface tension were compared with those calculated from Butler's model based on optimized thermodynamic parameters and our data from earlier investigations. In an earlier study, experimental data for all investigated compositions (Cu at. % = 1.08 to 6.5) exhibit an increase in the surface tension with interesting temperature, while both ternary alloys of this study show a slight lowering tendency in comparison to $(Sn\text{-}Ag)_{eut}$. A more evident decreasing tendency of surface tension and interfacial tension was noted in meniscographic measurements, noting that data of interfacial tension are always lower than surface tension due to the role of the flux. Eight different fluxes were tested to select the lowest interfacial tension for the $(Sn\text{-}Ag)_{eut}$. ROLI (3% solids), which is the alcoholic solution of organic compounds and rosin activated by halogens, was recommended. In $(Sn\text{-}Ag)_{eut}$ + Cu Soldering Materials, Part II: Electrical and Mechanical Studies, for the same $(Sn\text{-}Ag)_{eut}$ and $(Sn\text{-}Ag)_{eut}$ + Cu alloys (Cu at. % = 0.46 and 0.74), the electrical resistance and strength measurements will be presented in parallel with printed-circuit boards in wave soldering at 260 °C.

1. Introduction

In extensive studies undertaken all over the world in the last decade to replace traditional Sn-Pb solders by Pb-free materials, various properties of the candidate alloy systems have been considered; these include physical, chemical, and mechanical properties as well as cost and manufacturability. Among these, the physical properties of surface tension and interfacial tension are important for the evaluation of the wettability. In 1997 at the Institute of Metallurgy and Materials Science in Kraków systematic measurements were initiated for surface tension and density of Pb-free soldering materials by the maximum bubble pressure and dilatometric methods on pure metals, binary low-melting eutectics, and ternary alloys based on $(Sn\text{-}Ag)_{eut}$ intended for practical application. Experimental studies of the surface tension were combined with modeling by Butler's method [1932But] based on optimized thermodynamic parameters obtained in cooperation with Tohoku University, Sendai, Japan [2002Liu, 2002Mos1, 2001Mos1]. It was shown that the experimental temperature dependence of the surface tension is linear, while in Butler's method [1932But] this dependence is curvilinear and when two components have the similar surface tensions (e.g., Sn and In) there is no influence on the surface tension of Sn. In addition, this equivalency continues in ternary alloys [$(Sn\text{-}Ag)_{eut}$ + In] [2002Liu]. In such a case, Butler's modeling is particularly useful, and generally speaking, this method may be used for the design of new Pb-free alloys, but with limitations discussed in a recent publication on Ag-Bi liquid alloys [2003Gas]. In 2002, the Institute of Metallurgy and Materials Science initiated a project with two industrial institutes, Tele and Radio Research Institute in Warszawa and Institute of Non-Ferrous Metals in Gliwice (Poland), that was aimed at the production of Pb-free solders, starting from binary Sn-Ag and ternary Sn-Ag-Cu eutectics with additions of Bi and Sb. The possibility of using eutectic or close to eutectic Sn-Ag-Cu alloys as a substitute for Sn-Pb solders was suggested by Miller et al. [1994Mil]. Experimental and thermodynamic assessment of the Sn-Ag-Cu solder alloys were presented by [2000Ohn] and by [2000Moo].

It is the main aim of this presentation to proceed in the direction of practical application by combining the previously published studies [2002Mos1] of the surface tension and density measurements on eutectic Sn-Ag and on ternary alloys with Cu additions (Cu at.% = 1.08, 2.00, 3.75 and 6.50 molar fractions) with meniscographic studies of interfacial tension using several fluxes for liquid binary eutectic Sn-Ag and on two new alloys close to ternary eutectic Sn-Ag-Cu (Cu at.% = 0.46 and 0.74).

2. Experimental

The physics of wetting is governed by the Young-Dupré relation combining wettability σ_{SV} or σ_{SF} with the tendency

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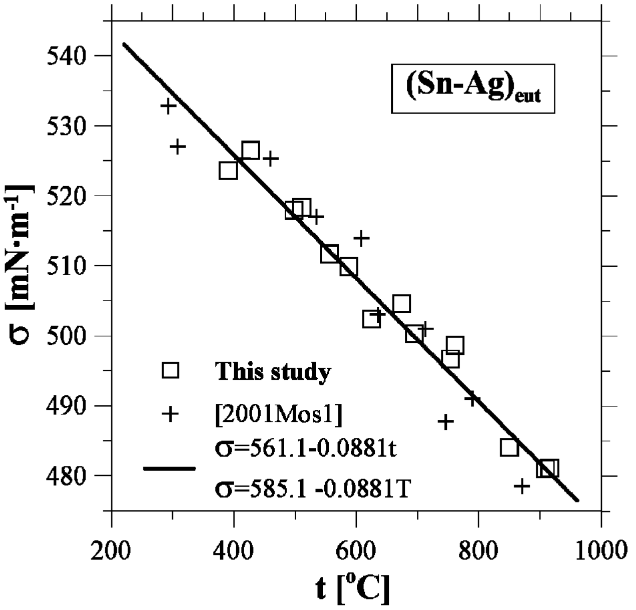

Fig. 1 Temperature dependence of the surface tension of the Sn-Ag eutectic alloy. Continuous line indicates the average value from this study combined with [2001Mos1] (Table 1).

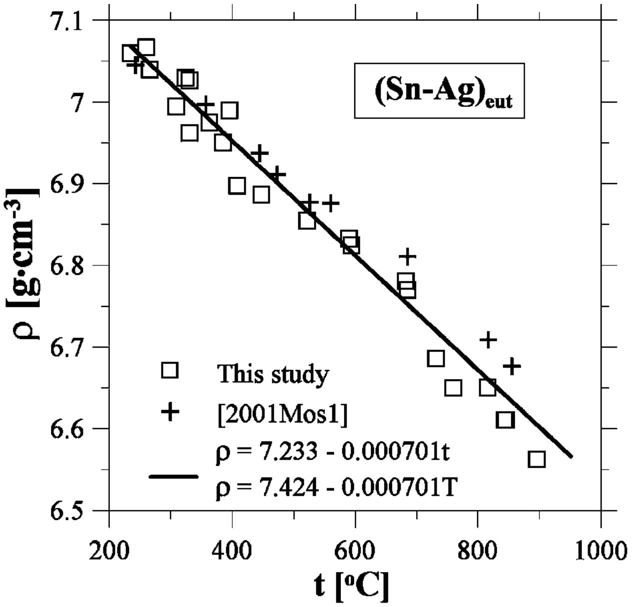

Fig. 2 Temperature dependence of the density of the Sn-Ag eutectic alloy. Continuous line indicates the average value from this study combined with [2001Mos1] (Table 2).

Table 1 Temperature Dependencies of the Surface Tension of Sn-Ag-Cn Alloys Together With the Values of σ Calculated at 250 °C and the Estimated Errors

Cu, at.%	$\sigma = a + bT$, mN m^{-1}	$\sigma_{(250\,°C)}$, mN m^{-1}	Err[a], mN m^{-1}	Err[b], mN m^{-1} K^{-1}
1.08 (a)	586.5-0.0819T	543.6 ± 6.4	±8.2	±0.0095
2.00 (a)	600.5-0.0879T	554.6 ± 10.5	±14.2	±0.0160
3.75 (a)	597.1-0.0763T	557.2 ± 11.8	±17.3	±0.0202
6.50 (a)	612.5-0.0887T	566.2 ± 8.2	±12.3	±0.0155
0.00 (a)	586.4-0.0904T	539.1 ± 12.5	±15.6	±0.0179
0.46 (b)	587.0-0.0964T	536.6 ± 7.8	±7.4	±0.0086
0.74 (b)	582.1-0.0867T	536.7 ± 6.9	±5.8	±0.0068
0.00 (b)	585.0-0.0873T	539.1 ± 6.3	±8.1	±0.0086
0.00 (c)	585.1-0.0881T	539.0 ± 8.2	±7.8	±0.0085

(a) [2002Mos1]
(b) Binary eutectic Sn-Ag and ternary alloys Sn-Ag-Cu, this study
(c) Average values of [2001Mos1] and of this study for binary eutectic Sn-Ag indicated in Fig. 2 and 3

Table 2 Temperature Dependencies of the Density of Sn-Ag-Cu Alloys Together With the Values of ρ Calculated at 250 °C and the Estimated Errors

X_{Cu}	$\rho = a + bT$ g cm^{-3}	$\rho_{(250\,°C)}$ g cm^{-3}	Err[a] g cm^{-3}	Err[b] g cm^{-3} K^{-1}
1.08 (a)	7.4529-0.000662T	7.107 ± 0.034	±0.047	±0.000051
2.00 (a)	7.5605-0.000722T	7.183 ± 0.045	±0.056	±0.000067
3.75 (a)	7.6958-0.000809T	7.273 ± 0.024	±0.031	±0.000035
6.50 (a)	7.7631-0.000776T	7.357 ± 0.061	±0.096	±0.000125
0.00 (a)	7.3645-0.000599T	7.051 ± 0.031	±0.040	±0.000047
0.46 (b)	7.4577-0.000691T	7.096 ± 0.037	±0.050	±0.000058
0.74 (b)	7.4615-0.000703T	7.094 ± 0.027	±0.028	±0.000035
0.00 (b)	7.4450-0.000739T	7.059 ± 0.054	±0.042	±0.000051
0.00 (c)	7.4241-0.000701T	7.058 ± 0.063	±0.043	±0.000052

(a) [2002Mos1]
(b) Binary eutectic Sn-Ag and ternary alloys Sn-Ag-Cu, this study
(c) Average values of [2001Mos1] and of this study for binary eutectic Sn-Ag indicated in Fig. 2 and 3

to form an intermetallic compound (IMC) denoted by σ_{SL}, surface tension by σ_{LV}, or interfacial tension by σ_{LF} and the contact angle by ϕ. L indicates liquid solder, S the solid substrate-usually Cu, V protective gas, and F the flux. This is illustrated by two schemes:

First, one for surface tension measurements performed under a protective gas:

$$\sigma_{SV} = \sigma_{SL} + \sigma_{LV} \cos\phi$$

Second, for meniscographic measurements of interfacial tension using flux:

$$\sigma_{SF} = \sigma_{SL} + \sigma_{LF} \cos\phi$$

Wettability, on the interphase Cu/flux or Cu/gas correlated with contact angle is impossible to quantify, but for practical applications for Sn-Pb and Pb-free solders in meniscographic tests, the wetting force as a function of time is monitored to evaluate interfacial tension and contact angles. The shortest wetting time, the highest wetting force, and the lowest contact angle are the indications of good wettability.

In the surface tension measurements, for instance by means of the maximum bubble pressure method, a decrease of surface tension also shows an improvement of wettabil-

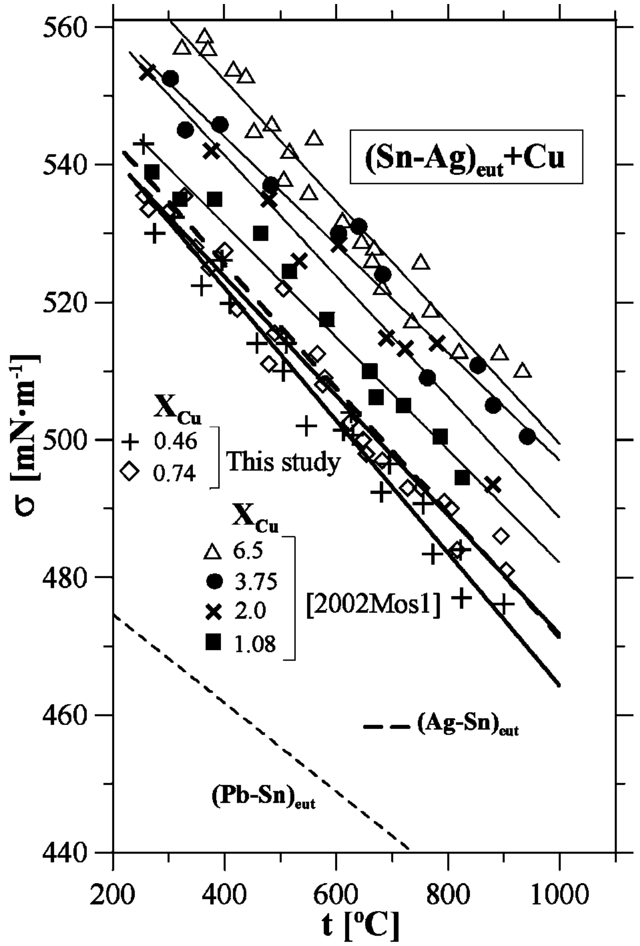

Fig. 3 Temperature dependencies of the surface tension of binary eutectic Sn-Ag (average from Table 1) and of Sn-Ag-Cu alloys. Plot for Sn-Pb eutectic from [2001Gas] illustrates the distance from investigated Pb-free alloys.

ity. The values of contact angle indicate wettability: 0° < ϕ < 30° very good wetting, 30° < ϕ < 40° good wetting, 40° < ϕ < 55° acceptable, 55° < ϕ < 70° poor wetting, and ϕ > 70° very poor wetting.

In both schemes of the Young-Dupré relation, the same term σ_{SL} corresponds to the reaction on the interface Cu/solder. It was shown by [1998Lee] that the composition at the Cu substrate/solder interface can be estimated by thermodynamic calculations of metastable equilibria between the substrate and the liquid solder. The fast formation of an IMC on this interface contribute to lower σ_{SL} and the driving force at the substrate/solder interface reflects wettability of liquid solder to substrate. Generally speaking, data of interfacial tension are lower than the surface tension because the fluxes decrease the surface energy.

2.1 Surface Tension and Density Measurements

Similar to previous work [2002Liu, 2002Mos1, 2001Mos1] this study also used the maximum bubble pressure method and dilatometric techniques to measure surface tension and density in the temperature range 230-950 °C. Details of the experimental set up and the calculations were described previously [2001Mos1]. Results are presented in Table 1 for surface tension and in Table 2 for density. Data on surface tension and density of binary Sn-Ag eutectic that were obtained in this study (plotted in Fig. 1 and 2) were averaged together with the data from [2001Mos1], and this

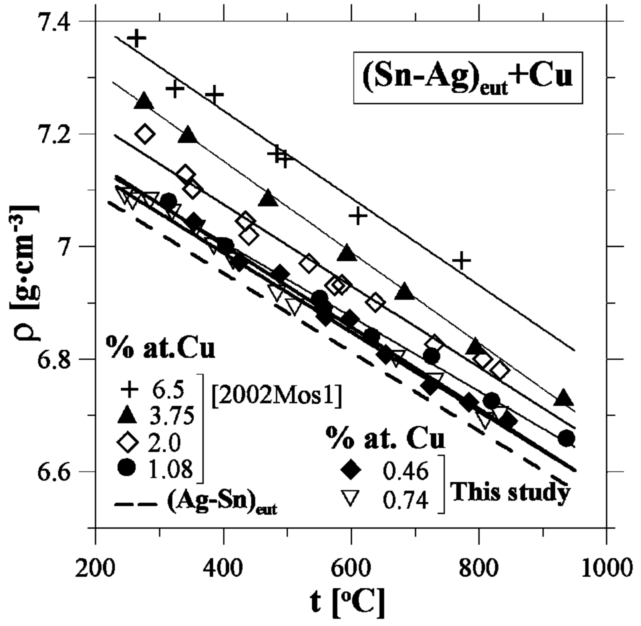

Fig. 4 Temperature dependencies of the density of Sn-Ag-Cu liquid alloys.

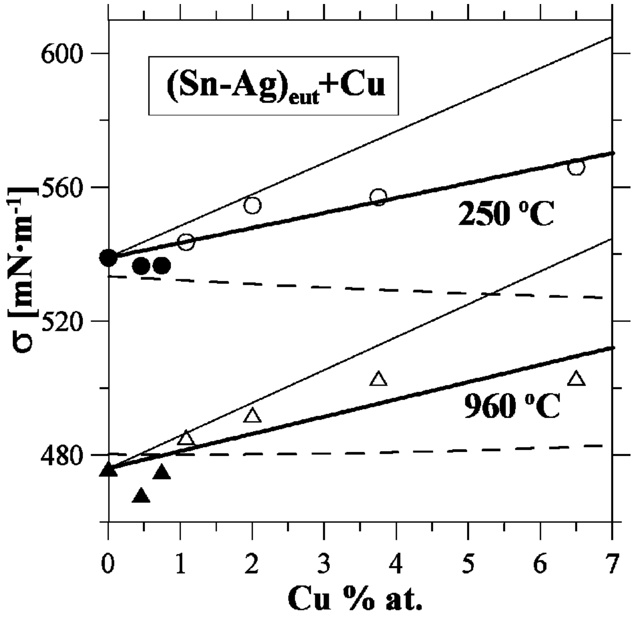

Fig. 5 Isotherms of the surface tension calculated at 250 °C and 960 °C of liquid Sn-Ag-Cu alloys. Points reflect data from Table 1; the filled points are from this study and the open points are from [2002Mos1].

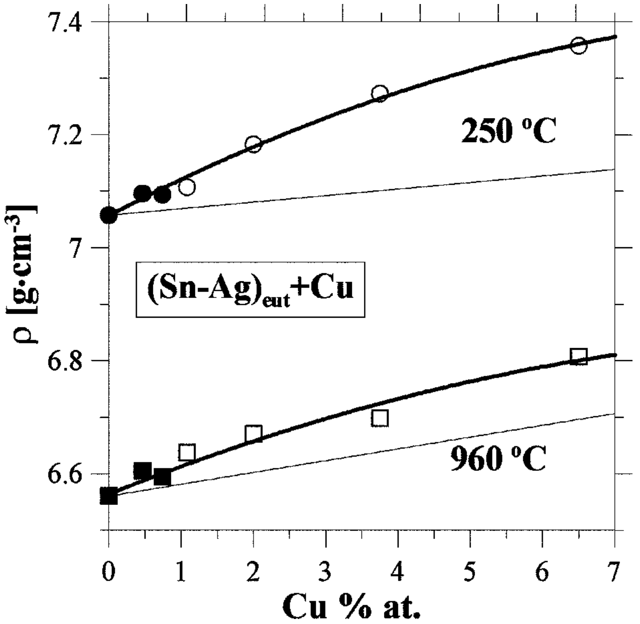

Fig. 6 Isotherms of the density calculated at 250 °C and 960 °C of liquid alloys Sn-Ag-Cu. Points reflect data from Table 2, the filled points are from this study and the open points are from [2002Mos1].

average was used in further plots for ternary alloys. In Fig. 3 and 4 there are presented temperature dependencies of the surface tension and density, Fig. 5 and 6 show their isotherms at 250 and 960 °C, and Fig. 7 is a plot of isotherms of the molar volumes also at 250 and 960 °C.

Tables 1 and 2 list, respectively, calculated values of the surface tension and density at 250 °C to later make a comparison in the next part of this presentation with wetting balance measurements performed at 252 °C.

2.2 Wetting Balance Measurements of Interfacial Tension, Wetting Force and Wetting Angle

Two kinds of the wetting balances (meniscographs) were used: Solderability Tester MENISCO ST 60 produced in France by Metronelec, and Solderability Tester Mk6^A produced in USA by General Electric. The wetting balance test can be used to observe the dynamic process of wetting by measuring the force that acts between the immersing specimen and molten solder. In this study, the interfacial tension (solder/flux) and surface tension (solder/air) were measured by a method presented by Miyazaki et al. [1997Miy]. In this method, a non-wetting specimen was immersed into a molten solder bath at a fixed speed, and the interfacial tension σ_{LF} [mN/m] was calculated by analyzing the measured force (F_r)-immersion depth relationship:

$$F_r = \sigma_{LF} L \cos\varphi - \rho V g \qquad \text{(Eq 1)}$$

In Eq. 1, L [m] denotes circumference of the immersed sample, ρ is the density of the solder in g/cm^3 (data from Table 2 were used), V is the immersion volume of specimen, g [m/s^2] is the acceleration of the gravity, and ϕ is the contact angle. The first part of the meniscographic procedure by the method proposed by Miyazaki et al. [1997Miy] was aimed at the choice of the proper flux. The eight candidates differing in composition were tested by measurements of the interfacial tension using Teflon plates of the following dimensions: 6 × 25 × 1.5 mm at 252 °C immersed at the depth of 9 mm into the liquid binary eutectic Sn-Ag with the speed of immersing and pulling out 1 mm/s. In these measurements the Solderability Tester Mk6^A (Metronelec, France) was used. The results for all tested fluxes are presented in Table 3.

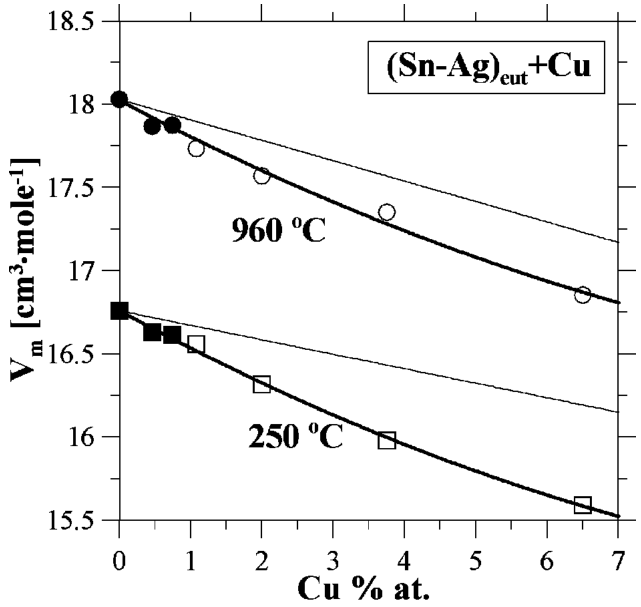

Fig. 7 Isotherms of the molar volume of the Sn-Ag-Cu liquid alloys calculated at 250 °C and 960 °C.

Table 3 Interfacial Tension Measurements of the Investigated Fluxes versus the Eutectic Sn-Ag Alloy

No.	Type of Flux According to J-STD 004 Standard	Average Value of Interfacial Tension, mN/m
1	ROL0 (25% solids)	403 ± 9
2	ROL1 (25% solids)	383 ± 17
3	ROM1 (25% solids)	372 ± 6
4	ROM0 (24% solids)	392 ± 15
5	ROL1 (3% solids)	390 ± 5
6	ORL0 (1% solids)	376 ± 22
7	ORL0 (0.5% solids)	410 ± 13
8	VOC-free (4% solids) (a)	414 ± 12

(a) Type not compatible with the J-STD 004 standard

With the information from Table 3, it was decided to use flux ROL1 (3% solids) for further measurements of the interfacial tension of the three alloys of this study from Table 1 and for the Sn-Pb euetectic due to a moderately low value of interfacial tension and the low scatter during the tests. Besides, the washing is not necessary after the soldering operation. Results are summarized in Table 4.

The wetting balance measurements were made by means of Solderability Tester Menisco ST 60. The tests conditions were as follows:

Cu samples of the dimensions: 6 × 25 × 1.5 mm covered by ROL1 flux (during 5 s), at 252 °C, for10 s immersion time at the depth of the 4 mm. The speed of immersion and removing the samples was 21 mm/s. The meniscograph yielded results for the wetting time, wetting force, and the contact angle in both graphical and tabular form due to the special programs for computer control.

A typical plot from wetting balance is shown in Fig. 8.

An example of plot obtained from wetting balance for binary eutectic Sn-Ag is presented in Fig. 9, indicating the wetting time 0.38 ± 0.03 s and maximum wetting force 5.42 mN. The contact angle 46° was calculated for this experiment from Eq 1.

The comparisons of contact angles, wetting force, and wetting time for the tested alloys are shown in Fig. 10-12. It is evident in comparison to binary eutectic, that the wetting times for ternary alloys are comparable, the contact angles for ternary alloys are lower, and the highest wetting force was obtained for sample with 0.46 at.% Cu.

3. Conclusions

Our previous systematic experimental studies of physical properties of Pb-free solders were limited to surface tension and density, allowing only the observation of the trend toward improving wettability when an addition of a component decreased the surface tension. However, in [2002Mos2], comparison of the influence of Bi and In additions were made for the surface tension of binary eutectic Sn-Ag by the maximum bubble pressure method and of the interfacial tension from the meniscographic studies of Takemoto et al. [2001Tak] and Vianco et al. [1999Via] and showed the same trend as indicated in Fig. 13. In this study, for the first time we combined surface tension with wetting time, wetting force, contact angle, and interfacial tension. In this manner it was possible to verify the trend observed previously in [2002Mos2]. This is documented in Fig. 14 presenting a comparison of the surface tension at 250 °C from the maximum bubble pressure method (solder/protective atmosphere), with interfacial tension (solder/flux) and surface tension (solder/air) at 252 °C from wetting balance measurements.

The slight decrease of surface tension at 0.46 at.% of Cu (the upper part of Fig. 14) is compatible with interfacial tension and surface tension from wetting balance data. The results from wetting balance for both ternary alloys are lower in comparison to the binary eutectic. In addition, more evident changes are indicated by interfacial tension and surface tension from meniscographic measurements. The graphical results show a slight decreasing tendency of surface tension from the maximum bubble pressure method and interfacial tension at low Cu additions contrary to the tendency observed for higher Cu concentrations. It should be noted that the difference in numerical values of the surface tension from the maximum bubble pressure method and from meniscographic technique may result from the use of protective argon + hydrogen atmosphere in the first case, while in the second, ordinary air was used. When a protective atmosphere is used, the values of surface tension are higher than they are in air, as indicated in [1994Gla] for binary eutectic Sn-Ag at 271 °C, with the value in air was 431 mN/m, and that in nitrogen was 493 mN/m.

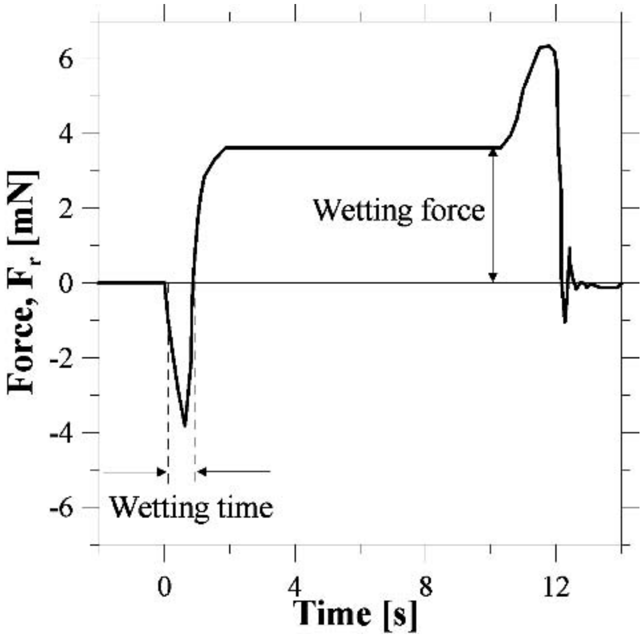

Fig. 8 Graphical output for a typical measurement of the wetting force and the wetting time by the meniscographic method.

Table 4 Interfacial Tension [flux ROL1 (3% solids)] and Surface Tension Determined by Miyazaki et al. [1997Miy] Method versus Investigated Alloys With Sn-Pb Eutectic Data for Comparison

No	Composition, at.%	Average Value of Interfacial Tension, mN/m	Average Value of Surface Tension, mN/m
1	Sn3.8Ag	390 ± 5	464 ± 10
2	(Sn-Au) eut + 0.46Cu	384 ± 11	408 ± 3
3	(Sn-Ag) eut + 0.74Cu	371 ± 14	415 ± 6
4	Sn60Pb40	318 ± 5	356 ± 11

From Fig. 14, the differences of the Sn-Ag eutectic in relation to the Sn-Pb eutectic are also visible. From the differences between surface tension plotted in argon + hydrogen and in air, one can draw the conclusion that it is not reasonable to use the protective atmosphere, as in this case the higher values of surface tension are obtained. It should be taken into account that in Fig. 14 we are only analyzing the term σ_{LV} in the Young-Dupré relation.

On the other hand, in relation to Fig. 14, it should be mentioned that Pb-free solders oxidize more rapidly than Sn-Pb solders, and a new oxide layer forms on a liquid solder immediately after the skin is removed. The Pb-free solders are more evident as they contain as much as 95% Sn compared with only 63% for the eutectic tin-lead solders. The higher tin content results in a more rapid formation of

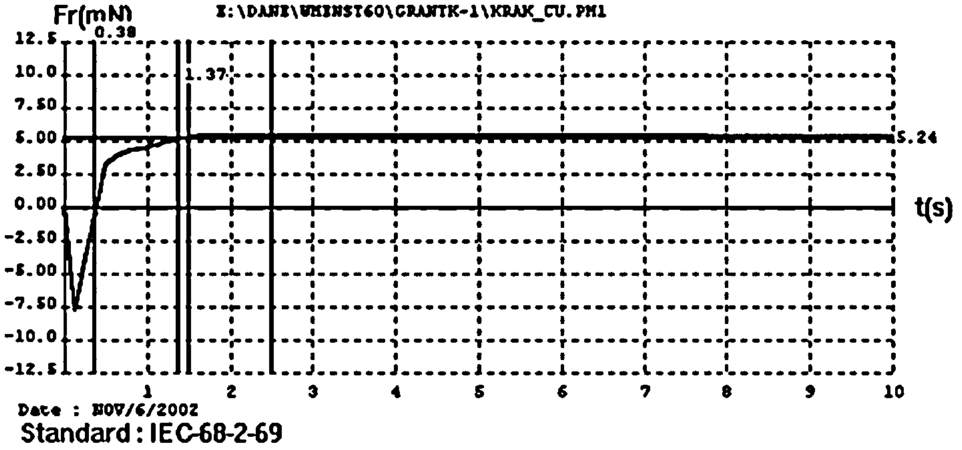

Fig. 9 Plot of the wetting time and wetting force from meniscographic method for the Sn-Ag eutectic.

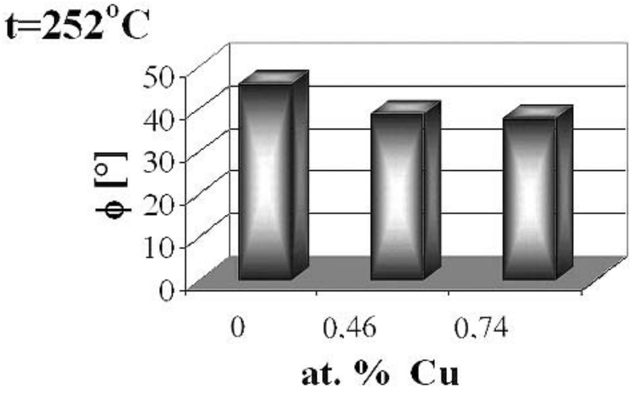

Fig. 10 Values of the contact angles for the Sn-Ag-Cu liquid alloys at 252 °C from meniscographic measurements compared with data for binary eutectic Sn-Ag.

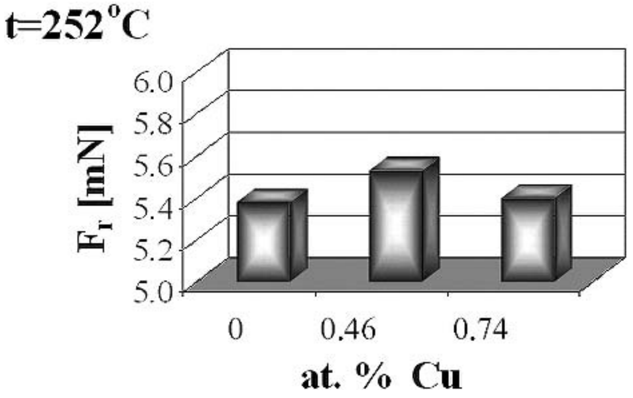

Fig. 11 Values of the wetting forces for the Sn-Ag-Cu liquid alloys at 252 °C from meniscographic measurements compared with data for binary eutectic Sn-Ag.

tin oxides (SnO and SnO_2) in the presence of oxygen, while the higher solder bath temperature also accelerates the oxidation process. The oxide layer formed by a Sn-Ag or Sn-Ag-Cu lead-free solder is more tenacious and does not break

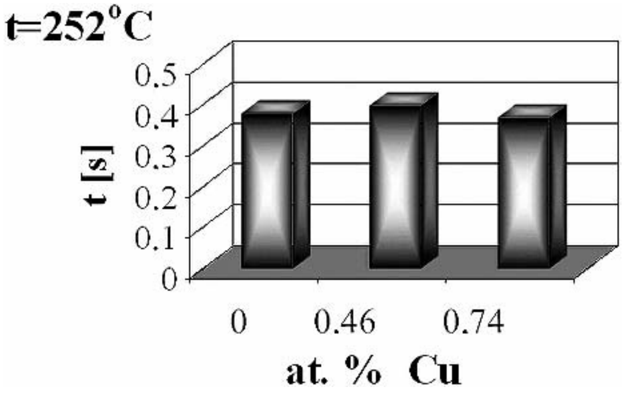

Fig. 12 Values of the wetting time for the Sn-Ag-Cu liquid alloys at 252 °C from meniscographic measurements compared with the data of binary eutectic Sn-Ag.

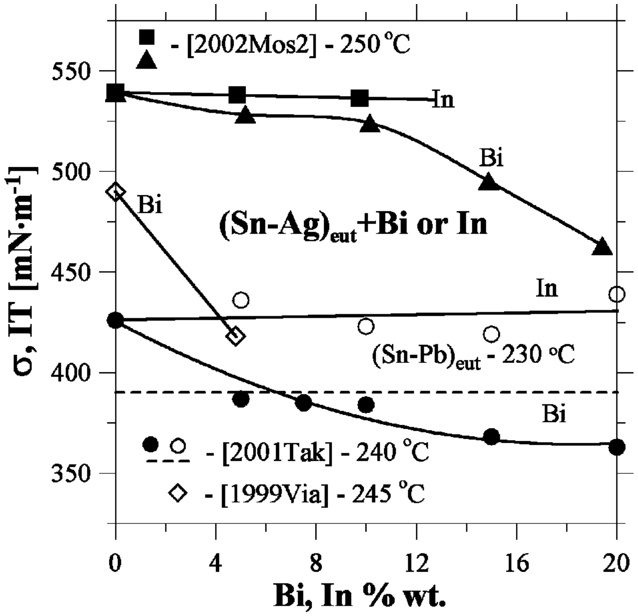

Fig. 13 Comparison of the surface tension and interfacial tension for $(Sn\text{-}Ag)_{eut}$ with In and Bi additions.

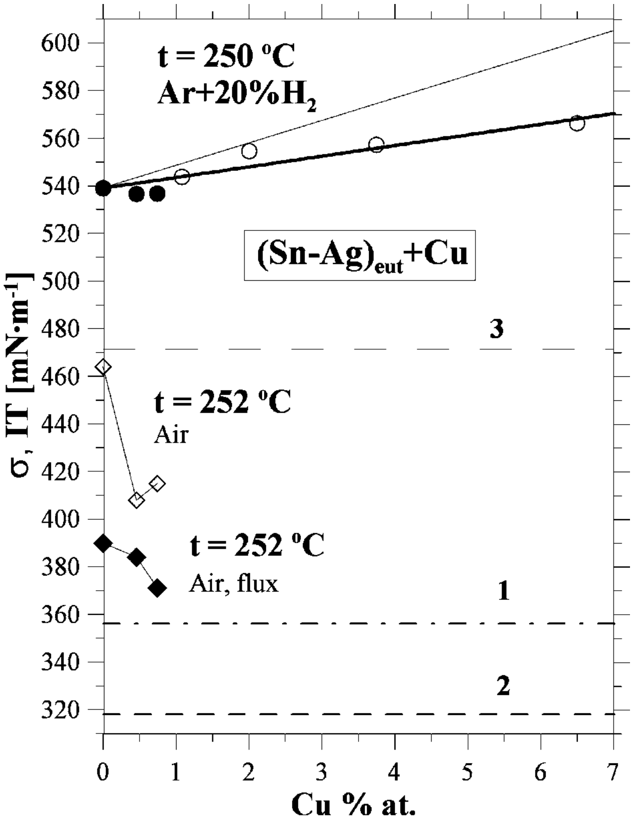

Fig. 14 Comparison of the surface tensions (σ) from maximum bubble pressure method (the upper part in argon with hydrogen) with meniscographic technique (middle data in air) and with interfacial tension (IT) of binary eutectic Sn-Ag and ternary alloys Sn-Ag-Cu versus $(Sn\text{-}Pb)_{eut}$ liquid alloys as indicated by various dotted lines (1,2,3) to illustrate the distance from investigated Pb-free alloys. 1-measurements of the surface tension in air (the last column of Table 4), 2-measurements of the interfacial tension (the middle column of Table 4) and 3-measurements of the surface tension from [2001Gas].

up into smaller particles, making dross removal more difficult. The use of nitrogen is highly recommended when implementing lead-free soldering since it not only minimizes the formation of tin oxides and dross (oxides lower the surface energy of the substrate) but at the same time influences the reactions at the interface Cu/solder (σ_{SL} in the Young-Dupré relation) and finally improves the degree and the rate of wetting.

In Fig. 14 (upper part, open points from [2002Mos1]), for higher additions of Cu to binary eutectic Sn-Ag, an increase of surface tension was observed in agreement with the surface tension of pure Cu, contrary to the results of this paper. A more evident tendency was observed in meniscographic studies, however not confronted by further measurements for higher Cu contents. On the other hand, alloys with higher Cu content are not recommended based on mechanical properties and the increase of melting temperatures so important in soldering processes.

The tendency observed in our studies toward lowering surface tension at low Cu additions in comparison to binary eutectic and the use of such alloys for practical applications seem reasonable in the light of the observation of Anderson et al. [2001And], who, from DTA measurements have shown the similar “near-binary eutectic” melting behavior for several Sn-Ag-Cu alloys and for low Cu concentrations.

It should be stressed that, until now, the properties of new Pb-free solders are far from those for traditional Sn-Pb solders, considering melting temperature, mechanical properties, manufacturability and the moderate cost.

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