Thermo-mechanical and wetting behavior of modified SnAg3.5eutectic solder alloy

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Abstract

Effects of adding bismuth content on structure, thermo-mechanical and wetting behavior of SnAg3.5 eutectic alloy have been investigated. Matrix structure of SnAg3.5 eutectic alloy, such as crystallinity, crystal size and lattice parameters, changed after adding bismuth content which effect on all measured properties. Melting temperature of SnAg3.5 eutectic alloy decreased after adding bismuth content. Elastic modulus and contact angle of SnAg3.5 eutectic alloy varied after adding bismuth content. The Sn66.5Ag33.5Bi3 alloy has the best solder properties for electronic applications such as lower melting temperature, contact angle and elastic modulus.

Key words

elastic modulus; contact angle; thermal properties; microstructure; eutectic alloy
1. Introduction

The solder alloys are binary, ternary and some are even quaternary alloys. Since the properties of the binary Pb-free solders cannot fully meet the requirements for applications in electronic packaging, additional alloying elements were added to improve the performance of these alloys. The melting point of a solder should be low enough to avoid thermal damage to the assembly being soldered and high enough for the solder joint to bear the operating temperatures. Tin-silver based solders have been considered as the first choice for a lead free solder due to its excellent mechanical properties. The eutectic composition for this solder is Sn-3.5wt%Ag and the eutectic temperature is 221 °C. Its microstructural studies have confirmed the presence of the fine Ag3Sn needles and β-Sn matrix [1]. Addition of Bi into Sn-Ag eutectic alloy reduces its melting temperature effectively and also improves the wettability [2-4]. Addition of small amount of Cu has been found to be advantageous for this binary Sn-3.5Ag solder. The eutectic Sn-Ag-Cu solder properly wets the substrate. Now a day it is widely used in aircraft and automotive industries, where the solder joints are subjected to thermal stresses. Its mechanical properties have been found to be better than that of Sn-Pb solders. Researchers have conducted many experiments to find the exact eutectic composition for this ternary alloy, but still there is a little controversy. The eutectic temperature for this composition has been found to be 2170C [5]. The creep rupture properties of Sn–3.5Ag based ternary alloys with varying amounts of Cu or Bi were investigated using rolled and heat-treated bulk specimens. The results show that, The 0.75% Cu specimen has lowest creep rate, while the 10% Bi specimen have highest creep rate [6]. In the present study, we have examined three typical Sn–Ag–Cu near-eutectic alloys, Sn–3.0Ag–0.5Cu,Sn–3.5Ag–0.7Cu and Sn–3.9Ag–0.6Cu, as standard lead-free solders. The effects of strain rates and cooling speeds on various properties of the alloys were investigated [7]. The effects of rare earth Ce doping on the properties of SnAgCu solder alloys were studied [8]. The addition of 0.03% (mass fraction) rare earth Ce into SnAgCu solder may improve its mechanical properties, but slightly lower its melting temperature. It is found that SnAgCuCe solders show higher creep resistance than SnAgCu alloys. The effect of cadmium content on structure, elastic modulus, electrical resistivity, thermal diffusivity and internal friction of SnAg eutectic alloy have been investigated [9]. The aim of this work was to investigate the effects of adding bismuth content on structure, elastic modulus, thermal diffusivity, internal friction, melting temperature and wetting behavior of SnAg3.5 eutectic alloy.

2. Experimental work

The tin-silver-bismuth alloy was molten in the muffle furnace using high purity, more than 99.95%, bismuth, tin and silver. The resulting ingots were turned and re-melted several times to increase the homogeneity of the ingots. From these ingots, long ribbons of about 3.5 mm width and ~ 70 μm thickness were prepared as the test samples by directing a stream of molten alloy onto the outer surface of rapidly revolving copper roller with surface velocity 31 m/s giving a cooling rate of 3.7 × 10^4 K/s. The samples then cut into convenient shape for the measurements using double knife cutter. Structure of used alloys was performed using an Shimadzu X-ray Diffractometer (DX-30, Japan) of Cu-Kα radiation with λ=1.54056 Å at 45 kV and 35 mA and Ni-filter in the angular range 20 ranging from 0 to 100° in continuous mode with a scan speed 5 deg/min. Electrical resistivity of used alloys was measured by double bridge method. The melting endotherms of used alloys were obtained using a SDT Q600 V20.9 Build 20 instrument. A digital Vickers micro-hardness tester, (Model-FM-7, Japan), was used to measure Vickers hardness values of used alloys. Internal friction Q’ and the elastic constants of used alloys were determined using the dynamic resonance method [10-12].

3. Results and discussions

Structure

X-ray diffraction patterns of Sn96.5—Ag3.5—Bi (x = 0, 6, 12, 18, 24 and 30 wt. %) rapidly solidified alloys have lines corresponding to β-Sn, Ag3Sn and hexagonal Bi phases as shown in Figure (1a). The analysis of x-ray patterns show that, adding different ratio of bismuth content to Sn96.5Ag3.5 alloy caused a change in its matrix microstructure such as lattice parameters and formed crystal structure (crystallinity, crystal size and the orientation) as seen in Table (1a). Lattice parameters (a and c), and unit volume cell of β-Sn phase in Sn96.5Ag3.5Bi alloys were determined and then listed in Table (1b). Also lattice parameter, a, and unit volume of β-Sn in Sn96.5Ag3.5 increased after adding bismuth content. That is because some Bi atoms dissolved in matrix alloy forming solid solution and other accumulated forming Bi phase.

Scanning electron micrographs, SEM, of Sn96.5—Ag3.5—Bi (x = 0, 6, 18 and 30 wt. %) alloys show heterogeneity structure as shown in Figure (1b). Microstructure of Sn96.5—Ag3.5—Bi alloys show β-Sn matrix, needle Ag3Sn and spherical Bi atoms and that agree with x-ray results.

Thermal properties

Thermal analysis is often used to study solid state transformations as well as solid-liquid reactions. DSC thermographs were obtained by SDT Q600 (V20.9 Build 20) with heating rate 10 °C/min in the temperature range 0-400 °C. Figure (2a) shows DSC thermographs for Sn96.5—Ag3.5—Bi (x = 0, 6, 12, 18, 24 and 30 wt. %) alloys. A little variation in the exo-thermal peaks shape which related to a change in matrix alloy after adding Bi content. The melting temperature and other thermal properties of Sn96.5—Ag3.5—Bi alloys are listed in Table 2(a). Melting temperature of Sn96.5Ag3.5 alloy decreased after adding Bi content.
Wettability

Wetting is the ability of a liquid to maintain contact with a solid surface, resulting from intermolecular interactions when the two are brought together. Low contact angle, less than 90°, usually indicates that wetting of the surface is very favorable and the fluid will spread over a large area of the surface but high contact angle, greater than 90°, generally means that wetting of the surface is unfavorable so the fluid will minimize contact with the surface and form a compact liquid droplet. Table (2b) shows the contact angles of Sn$_{96.5}$Ag$_{3.5}$Bi$_x$ alloys on Cu substrate. The contact angle of Sn$_{96.5}$Ag$_{3.5}$Bi$_x$ alloy increased after adding Bi content up to 24% and then decreased. The photographs of spreading Sn$_{96.5}$Ag$_{3.5}$Bi$_x$ molten alloys on Cu substrate in air are shown in Figure (2b).

Mechanical properties

The elastic constants are directly related to atomic bonding and structure. Elastic modulus of Sn$_{96.5}$Ag$_{3.5}$Bi$_x$ alloys are listed in Table (3). Elastic modulus of Sn$_{96.5}$Ag$_{3.5}$Bi$_x$ alloy decreased after adding Bi content except 12 and 18% it's increased. That is because adding Bi content to Sn$_{96.5}$Ag$_{3.5}$ alloy changed its matrix microstructure which effects on atomic bonding. The resonance curves Sn$_{96.5}$Ag$_{3.5}$Bi$_x$ alloys are shown in Figure (3). Calculated internal friction and thermal diffusivity values of Sn$_{96.5}$Ag$_{3.5}$Bi$_x$ alloys are seen in Table (3).

Conclusions

Matrix microstructure such as unit cell and formed crystal of Sn$_{96.5}$Ag$_{3.5}$Bi$_x$ alloy changed after adding Bi content. Melting point and internal friction of Sn$_{96.5}$Ag$_{3.5}$Bi$_x$ alloy decreased after adding Bi content. Elastic modulus, thermal parameters and contact angle of Sn$_{96.5}$Ag$_{3.5}$Bi$_x$ alloy varied after adding Bi content. The Sn$_{96.5}$Ag$_{3.5}$Bi$_x$ alloy has the best solder properties for electronic applications such as lower melting temperature, contact angle and elastic modulus.

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Figure 1a: X-ray diffraction patterns of Sn$_{96.5-x}$Ag$_{3.5}$Bi$_x$ alloys
Figure 1b: SEM of Sn$_{96.5}$Ag$_{3.5}$Bi$_x$ alloys
Figure 2a: DSC thermographs of Sn$_{96.5}$Ag$_{3.5}$Bi$_x$ alloys
Figure 2b: Photographs of Sn$_{96.5}$Ag$_{3.5}$Bi$_x$ molten alloys on Cu substrate in air
**Figure 3:** - Resonance curves of Sn$_{96.5-x}$Ag$_{3.5}$Bi$_x$ alloys

**Table 1a:** X-ray analysis of Sn$_{96.5-x}$Ag$_{3.5}$Bi$_x$ alloys

| SnAg$_{3.5}$ |  |  |  |  | SnAg$_{3.5}$Bi$_6$ |  |  |  |  |
|-------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| 2θ          | Area            | FWHM            | τA              |                  | 2θ              | Area            | FWHM            | τA              |
| 30.5625     | 419.32          | 0.2165          | 366.937         |                  | 30.5526         | 90.34           | 0.2362          | 336.333         |
| 31.9859     | 514.29          | 0.2362          | 336.333         |                  | 31.9449         | 100             | 0.216           | 367.786         |
| 37.5561     | 24.68           | 0.6298          | 126.138         |                  | 32.0629         | 49.5            | 0.096           | 827.519         |
| 39.5036     | 23.01           | 0.2362          | 336.335         |                  | 39.5382         | 2.53            | 0.384           | 206.881         |
| 43.8454     | 125.4           | 0.2362          | 336.336         |                  | 43.7456         | 21.81           | 0.288           | 275.843         |
| 44.8673     | 194.23          | 0.1771          | 448.576         |                  | 44.7675         | 51.79           | 0.168           | 472.873         |
| 55.2738     | 76.27           | 0.2755          | 288.362         |                  | 55.1846         | 10.83           | 0.288           | 275.846         |
| 62.4617     | 84.1            | 0.2362          | 336.344         |                  | 62.3667         | 16.51           | 0.192           | 413.773         |
| 63.6882     | 33.06           | 0.1968          | 403.682         |                  | 63.6115         | 6.19            | 0.288           | 275.849         |
| 64.5709     | 44.27           | 0.1378          | 576.522         |                  | 64.404           | 11.65           | 0.168          | 472.885         |
| 72.359      | 37.51           | 0.1968          | 403.688         |                  | 72.1615         | 5.62            | 0.288           | 275.853         |
| 73.1213     | 32.46           | 0.1574          | 504.738         |                  | 72.9011         | 6.24            | 0.192           | 413.780         |
| 79.4521     | 41.2            | 0.1574          | 504.744         |                  | 79.2624         | 9.46            | 0.24            | 331.028         |
| 89.3488     | 20.72           | 0.1574          | 504.754         |                  | 89.1117         | 4.63            | 0.192           | 413.793         |
| 95.5001     | 16.9            | 0.144           | 551.731         |                  | 95.2757         | 3.32            | 0.192           | 413.798         |
| 97.3837     | 19.05           | 0.192           | 413.800         |                  | 97.0716         | 1.91            | 0.384           | 206.899         |
| 2θ  | Area | FWHM | Å  | 2θ  | Area | FWHM | Å  |
|-----|------|------|----|-----|------|------|----|
| 27.1481 | 51.74 | 0.2362 | 336.332 | 27.2011 | 155.75 | 0.2165 | 366.936 |
| 30.5083 | 525.64 | 0.2558 | 310.562 | 30.5292 | 194.38 | 0.1181 | 672.665 |
| 31.9147 | 635.89 | 0.2558 | 310.562 | 31.9513 | 450.13 | 0.1771 | 448.570 |
| 34.5328 | 17.61 | 0.4723 | 168.202 | 37.9422 | 51.51 | 0.1968 | 403.669 |
| 37.9169 | 16.84 | 0.2362 | 336.334 | 39.6415 | 64.99 | 0.2362 | 336.335 |
| 39.5025 | 47.25 | 0.3149 | 252.278 | 43.7802 | 108.64 | 0.1771 | 448.575 |
| 43.6951 | 78.76 | 0.1181 | 672.673 | 44.7452 | 175.45 | 0.1771 | 448.576 |
| 44.783 | 270.82 | 0.2362 | 336.337 | 45.867 | 19.97 | 0.3149 | 252.279 |
| 55.1854 | 53.5 | 0.2362 | 336.341 | 48.6967 | 22.98 | 0.2755 | 288.359 |
| 62.3394 | 70.02 | 0.1968 | 403.681 | 55.2282 | 76.28 | 0.2755 | 288.362 |
| 63.5442 | 20.85 | 0.1968 | 403.682 | 62.3763 | 98.16 | 0.2362 | 336.344 |
| 64.3591 | 64.21 | 0.2755 | 288.366 | 63.5815 | 31.08 | 0.2362 | 336.345 |
| 72.1601 | 32.33 | 0.2362 | 336.349 | 64.4166 | 102.74 | 0.3542 | 224.293 |
| 72.9502 | 23.69 | 0.1968 | 403.688 | 70.8912 | 16.35 | 0.3149 | 252.288 |
| 79.2561 | 43.04 | 0.2362 | 336.353 | 72.1711 | 21.27 | 0.1574 | 504.738 |
| 89.0869 | 13.04 | 0.1574 | 504.754 | 72.9924 | 36.53 | 0.2362 | 336.349 |
| 95.2694 | 14.47 | 0.2362 | 336.364 | 79.3238 | 47.97 | 0.1968 | 403.693 |
| 97.1882 | 25.62 | 0.576 | 137.933 | 85.2119 | 18.75 | 0.9446 | 84.107 |
| 97.2974 | 26 | 0.576 | 137.933 | 89.1624 | 16.92 | 0.1574 | 504.754 |
|       |      |      |     | 95.3219 | 18.5 | 0.2362 | 336.364 |
|       |      |      |     | 97.2974 | 26 | 0.576 | 137.933 |
| 2θ   | Area  | FWHM | τÅ  |
|------|-------|------|-----|
| 22.3746 | 28.23 | 0.2952 | 269.110 |
| 27.1518 | 209.81 | 0.2362 | 336.332 |
| 30.5285 | 432.25 | 0.2558 | 310.562 |
| 31.9257 | 425.87 | 0.2558 | 310.562 |
| 34.4912 | 9.53  | 0.4723 | 168.202 |
| 37.8821 | 83.04  | 0.3149 | 252.278 |
| 39.6098 | 70.36  | 0.2755 | 288.357 |
| 43.6942 | 116.27 | 0.2362 | 336.336 |
| 44.7481 | 167.83 | 0.2362 | 336.337 |
| 45.7569 | 17.7   | 0.2362 | 336.337 |
| 48.7718 | 21.96  | 0.3542 | 224.289 |
| 51.927  | 5.73   | 0.4723 | 168.205 |
| 55.1892 | 36.27  | 0.2755 | 288.362 |
| 56.0325 | 11.65  | 0.2755 | 288.362 |
| 59.2132 | 7.97   | 0.2755 | 288.363 |
| 62.3198 | 45.58  | 0.2362 | 336.344 |
| 63.6265 | 11.44  | 0.1968 | 403.682 |
| 64.2929 | 35.66  | 0.2362 | 336.345 |
| 67.3153 | 4.73   | 0.3149 | 252.287 |
| 70.7134 | 10.21  | 0.433  | 183.477 |
| 72.1151 | 20.32  | 0.2362 | 336.349 |
| 72.8539 | 14.01  | 0.1968 | 403.688 |
| 79.2192 | 23.52  | 0.2165 | 366.959 |
| 85.0131 | 10.18  | 0.7872 | 100.924 |
| 89.0318 | 10.94  | 0.2165 | 366.966 |
| 91.7617 | 5.22   | 0.4723 | 168.217 |
| 95.273 | 7.75  | 0.2362 | 336.364 |
| 97.2544 | 16.17  | 0.768  | 103.450 |

| 2θ   | Area  | FWHM | τÅ  |
|------|-------|------|-----|
| 22.4755 | 15.33 | 0.3149 | 252.275 |
| 27.1264 | 187.57 | 0.2755 | 288.354 |
| 30.5419 | 287.06 | 0.2952 | 269.112 |
| 31.9653 | 249.82 | 0.3149 | 252.276 |
| 34.5284 | 10.69  | 0.4723 | 168.202 |
| 37.9173 | 126.29 | 0.3346 | 237.424 |
| 39.6347 | 160.21 | 0.3542 | 224.287 |
| 43.7275 | 235.34 | 0.3149 | 252.278 |
| 44.7852 | 277.47 | 0.3346 | 237.426 |
| 46.885 | 32.34  | 0.3542 | 224.288 |
| 48.6666 | 48.82  | 0.2362 | 336.338 |
| 51.9243 | 5.62   | 0.3149 | 252.281 |
| 55.1519 | 45.07  | 0.2362 | 336.341 |
| 56.0054 | 22.98  | 0.2362 | 336.341 |
| 59.183  | 18.48  | 0.3542 | 224.292 |
| 61.1063 | 4.24   | 0.2362 | 336.344 |
| 62.3444 | 66.94  | 0.2362 | 336.344 |
| 63.5945 | 23.05  | 0.2558 | 310.573 |
| 64.4052 | 132.76 | 0.3936 | 201.841 |
| 67.3111 | 11.3   | 0.3149 | 252.287 |
| 69.0701 | 6.8    | 0.3149 | 252.287 |
| 70.7474 | 19.95  | 0.2362 | 336.348 |
| 72.1306 | 28.5   | 0.1771 | 448.592 |
| 72.9167 | 28.59  | 0.2362 | 336.349 |
| 74.8997 | 7.54   | 0.6298 | 126.145 |
| 79.2855 | 44.82  | 0.2755 | 288.373 |
| 84.8872 | 11.13  | 0.3149 | 252.294 |
| 87.0822 | 6.27   | 0.3149 | 252.296 |
| 89.1293 | 21.68  | 0.2165 | 366.966 |
| 91.7125 | 10.24  | 0.3149 | 252.298 |
| 95.2868 | 12.32  | 0.1771 | 448.612 |
| 96.4002 | 4.36   | 0.1968 | 403.707 |
| 97.3277 | 37.14  | 0.768  | 103.450 |
### Table 1b: Lattice parameters, unit cell volume and crystal size of Sn<sub>96.5-x</sub>Ag<sub>x</sub>Bi<sub>x</sub> alloys

| Alloys            | ave. particle size (Å<sup>0</sup>) | a (Å)  | c (Å)  | c/a   | Unit cell volume (Å<sup>3</sup>) |
|-------------------|-----------------------------------|--------|--------|-------|-----------------------------------|
| Sn<sub>96.5</sub>Ag<sub>3.5</sub> | 402.438                           | 5.84   | 3.186  | 0.546 | 10.707                            |
| Sn<sub>90.5</sub>Ag<sub>3.5</sub>Bi<sub>6</sub> | 373.796                           | 5.849  | 3.039  | 0.519 | 11.259                            |
| Sn<sub>84.5</sub>Ag<sub>3.5</sub>Bi<sub>12</sub> | 345.044                           | 5.854  | 3.192  | 0.545 | 10.738                            |
| Sn<sub>78.5</sub>Ag<sub>3.5</sub>Bi<sub>18</sub> | 352.93                            | 5.853  | 3.111  | 0.532 | 11.0137                           |
| Sn<sub>72.5</sub>Ag<sub>3.5</sub>Bi<sub>24</sub> | 282.037                           | 5.857  | 3.2014 | 0.547 | 10.714                            |
| Sn<sub>66.5</sub>Ag<sub>3.5</sub>Bi<sub>30</sub> | 278.686                           | 5.854  | 3.19   | 0.545 | 10.743                            |

### Table 2a: Melting point and thermal parameters of Sn<sub>96.5-x</sub>Ag<sub>x</sub>Bi<sub>x</sub> alloys

| Sample        | Melting point °C | T<sub>1</sub>(K) | T<sub>2</sub>(K) | ΔH×10<sup>4</sup> (J/Kg.K) | Cp (J/Kg.K) | ΔS (J/Kg.K) |
|---------------|------------------|-------------------|-------------------|--------------------------|-------------|-------------|
| SnAg<sub>3.5</sub> | 223.81           | 492.94            | 509.85            | 4.5240                   | 2675.34     | 90.25       |
| SnAg<sub>3.5</sub>Bi<sub>6</sub> | 213.29           | 477.75            | 498               | 5.3520                   | 2642.96     | 109.74      |
| SnAg<sub>3.5</sub>Bi<sub>12</sub> | 203.65           | 466.49            | 481.6             | 2.9730                   | 1967.57     | 62.73       |
| SnAg<sub>3.5</sub>Bi<sub>18</sub> | 195.6            | 455.8             | 476.78            | 2.1160                   | 1008.58     | 45.39       |
| SnAg<sub>3.5</sub>Bi<sub>24</sub> | 187.28           | 443.83            | 473               | 1.8250                   | 625.64      | 39.83       |
| SnAg<sub>3.5</sub>Bi<sub>30</sub> | 177.19           | 439.64            | 460.87            | 0.5666                   | 266.89      | 12.59       |
### Table 2b: Contact Angles of Sn$_{96.5-x}$Ag$_{3.5}$Bi$_x$ Alloys

| Alloys              | Contact angles ($^\circ$) |
|---------------------|---------------------------|
| Sn$_{96.5}$Ag$_{3.5}$| 26±2                      |
| Sn$_{90.5}$Ag$_{3.5}$Bi$_6$ | 34±2                      |
| Sn$_{84.5}$Ag$_{3.5}$Bi$_{12}$ | 33.5±2                    |
| Sn$_{78.5}$Ag$_{3.5}$Bi$_{18}$ | 30±2                      |
| Sn$_{72.5}$Ag$_{3.5}$Bi$_{24}$ | 33.5±2                    |
| Sn$_{66.5}$Ag$_{3.5}$Bi$_{30}$ | 23.5±2                    |

### Table 3: Elastic Moduli, Internal Friction and Thermal Diffusivity of Sn$_{96.5-x}$Ag$_{3.5}$Bi$_x$ Alloys

| Sample       | E GPa | B GPa | μ GPa | $Q^{-1}$ | $D_H \times 10^{-4}$ cm$^2$.Sec$^{-1}$ |
|--------------|-------|-------|-------|----------|---------------------------------------|
| SnAg$_{3.5}$ | 49    | 53.7  | 18.2  | 0.0455   | 4.25                                  |
| SnAg$_{3.5}$Bi$_6$ | 46    | 50.4  | 17.1  | 0.0276   | 5.71                                  |
| SnAg$_{3.5}$Bi$_{12}$ | 53.8  | 59    | 20    | 0.0280   | 4.29                                  |
| SnAg$_{3.5}$Bi$_{18}$ | 54    | 59.2  | 20    | 0.0342   | 1.95                                  |
| SnAg$_{3.5}$Bi$_{24}$ | 43    | 47.1  | 15.9  | 0.0305   | 7.92                                  |
| SnAg$_{3.5}$Bi$_{30}$ | 28    | 30.7  | 10.4  | 0.0448   | 2.48                                  |