



Electrochemical Corrosion Behavior, Microstructure and Soldering Properties of Tin Based Alloys

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Abstract

Microstructure, thermal behavior, wettability and electrochemical corrosion parameters of $\text{Sn}_{82}\text{Bi}_{15}\text{Zn}_3$, $\text{Sn}_{77}\text{Bi}_{15}\text{Zn}_3\text{Sb}_5$, $\text{Sn}_{79}\text{Bi}_{15}\text{Zn}_3\text{Ag}_3$ and $\text{Sn}_{81.3}\text{Bi}_{15}\text{Zn}_3\text{Cu}_{0.7}$ alloys have been studied and analyzed. The contact angles of used alloys varied from 21° to 31° which is less than 90° (low contact angle). That is meant, wetting process of these alloys are very favorable and the fluid will spread over a large area of the surface. Melting temperature values of $\text{Sn}_{82}\text{Bi}_{15}\text{Zn}_3$, $\text{Sn}_{77}\text{Bi}_{15}\text{Zn}_3\text{Sb}_5$, $\text{Sn}_{79}\text{Bi}_{15}\text{Zn}_3\text{Ag}_3$ and $\text{Sn}_{81.3}\text{Bi}_{15}\text{Zn}_3\text{Cu}_{0.7}$ alloys are lower than tin based eutectic solder alloys, (Sn- Zn or Sn- Cu or Sn- Sb), by 11% to 22%. Scanning electron microscope, x-ray analysis and differential scanning calorimetry graphs show that, the used alloys contained different phases. Corrosion rate of $\text{Sn}_{82}\text{Bi}_{15}\text{Zn}_3$ alloy in HCl varied after adding alloying elements. $\text{Sn}_{77}\text{Bi}_{15}\text{Zn}_3\text{Sb}_5$ alloy has lowest corrosion rate value.



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Introduction

Soldering is a low temperature metallurgical joining process. Solder joining is a wetting process followed by a chemical reaction. The chemical reaction following wetting is between the molten solder and the joining metallurgy to form an intermetallic phase region at the interface. Solder alloys are characterized by the melting temperature being a strong function of composition. Microstructure, elastic modulus, thermal behavior and contact angle of $\text{Sn}_{96.5}\text{Ag}_{3.5}$ alloy varied after adding bismuth content¹. Melting temperature, elastic modulus and

contact angle values of $\text{Sn}_{99.3}\text{Cu}_{0.7}$ alloy decreased after adding bismuth or bismuth-indium². The elastic modulus, internal friction, contact angle, melting temperature and specific heat of $\text{Sn}_{91}\text{Zn}_9$ alloy decreased after adding indium³. Also melting temperature, elastic modulus and thermal diffusivity of $\text{Sn}_{96}\text{Zn}_4$ alloy decreased after adding bismuth⁴. Strength of bismuth-tin-zinc alloy increased after adding alloying elements such as silver or indium⁵. Microstructure, thermal parameters, internal friction, wettability and electrochemical corrosion behavior of $\text{Bi}_{30}\text{Sn}_{50}\text{Sb}_{10}\text{Al}_5\text{Zn}_3\text{Cu}_2$, $\text{Bi}_{25}\text{Sn}_{61}\text{Sb}_5\text{Zn}_4\text{Al}_3\text{Ag}_2$, and

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$\text{Bi}_{20}\text{Sn}_{60}\text{Sb}_7\text{Al}_5\text{Zn}_3\text{Cd}_3\text{Cu}_2$, alloys have been studied [6]. Microstructure, wettability behavior, corrosion parameters, thermal properties of quaternary bismuth- tin based alloy have been studied using different experimental techniques⁷. Tin- zinc eutectic alloy has been considered as a candidate for lead free solder materials because of its low melting point, excellent mechanical properties and low cost⁸⁻¹⁰. Also mostly solders are based on Sn-containing binary and ternary alloys as alloying elements such as Zn, Bi, Cu, Ag, Sb and so on¹¹⁻¹³. Tin- zinc eutectic solder alloy is poor wettability, reliability, strength, easy oxidation and microvoid formation. Some researchers¹⁴⁻¹⁷ operated to improve the properties of Sn-Zn alloy by adding small amount of Bi, Cu, In, Ag, Al, Ga, Sb, Cr, Ni, Ge elements to develop Pb free alloys. The aim of our work is to improve soldering properties (melting temperature and contact angle) and corrosion behavior of tin based lead free solder alloys compared to tin eutectic, (Sn- Zn or Sn- Cu or Sn- Sb)alloys or lead- tin commercial solder alloy by changing their composition by adding different alloying elements.

Materials and Methods

Tin, bismuth, zinc, antimony and copper metals with purity more than 99.9 % were used to prepare $\text{Sn}_{82}\text{Bi}_{15}\text{Zn}_3$, $\text{Sn}_{77}\text{Bi}_{15}\text{Zn}_3\text{Sb}_5$, $\text{Sn}_{79}\text{Bi}_{15}\text{Zn}_3\text{Ag}_3$ and $\text{Sn}_{81.3}\text{Bi}_{15}\text{Zn}_3\text{Cu}_{0.7}$ alloys. Each alloy composition has a weight of 20 gram by using the digital milligram scale and the metals were added according to their weight per cent (wt. %) in the specimen. These alloys were melted and then normal casted on graphite substrate. The samples were prepared in convenient shape for all tests such as microstructure, thermal parameters and corrosion behavior. Microstructure of used alloys was performed using Shimadzu X-ray Diffractometer, (Dx-30, Japan) Cu-K α radiation

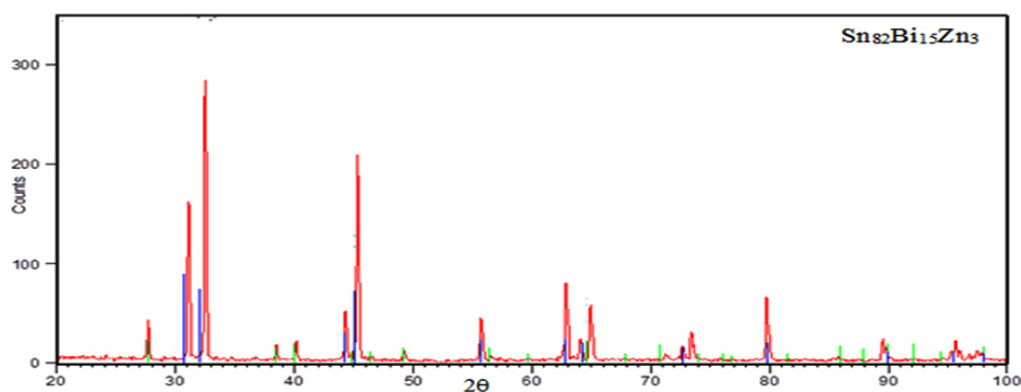
with $\lambda=1.54056 \text{ \AA}$ at 45 kV and 35 mA and Ni-filter, in the angular range 2θ ranging from 0 to 100° in continuous mode with a scan speed 5 deg/min and scanning electron microscope (JEOL JSM-6510 LV, Japan). The polarization studies were performed using Gamry Potentiostat/Galvanostat with a Gamry framework system based on ESA 300. Gamry applications include software DC105 for corrosion measurements, and Echem Analyst version 5.5 software packages for data fitting. The differential scanning calorimetry (DSC) thermographs were obtained by Universal V4. 5A TA Instrument [Q20 (V24. 11 Build 124)] with heating rate 10 k/min in the temperature range 0-400 °C.

Results and Discussion

Microstructure

X-ray analysis

Figure 1 shows x-ray diffraction patterns of $\text{Sn}_{82}\text{Bi}_{15}\text{Zn}_3$, $\text{Sn}_{77}\text{Bi}_{15}\text{Zn}_3\text{Sb}_5$, $\text{Sn}_{79}\text{Bi}_{15}\text{Zn}_3\text{Ag}_3$ and $\text{Sn}_{81.3}\text{Bi}_{15}\text{Zn}_3\text{Cu}_{0.7}$ alloys which have sharp lines corresponding to tetragonal β -Sn phase, hexagonal Bi phase, Zn phase and small lines represented undetected intermetallic compounds with dissolved atoms (Cu or Sb or Ag) in matrix formed a solid solution. The details of x-ray analysis of used alloys which comes from the x-ray device (2θ , Int. %, d $\text{\AA}$, FWHM and area) and x-ray cards (phase and Miller indices) are listed in Table 1. The x-ray analysis show that, the feature of formed phases such as crystallinity (peak intensity), crystal size (peak broadness) and orientations (peak position, 2θ) for $\text{Sn}_{82}\text{Bi}_{15}\text{Zn}_3$ alloy changed after adding antimony, silver and copper. That is because these elements atoms dissolved in matrix alloy formed a solid solution with undetected phases that agree with pervious work^{6,7} and other⁹⁻¹⁰.



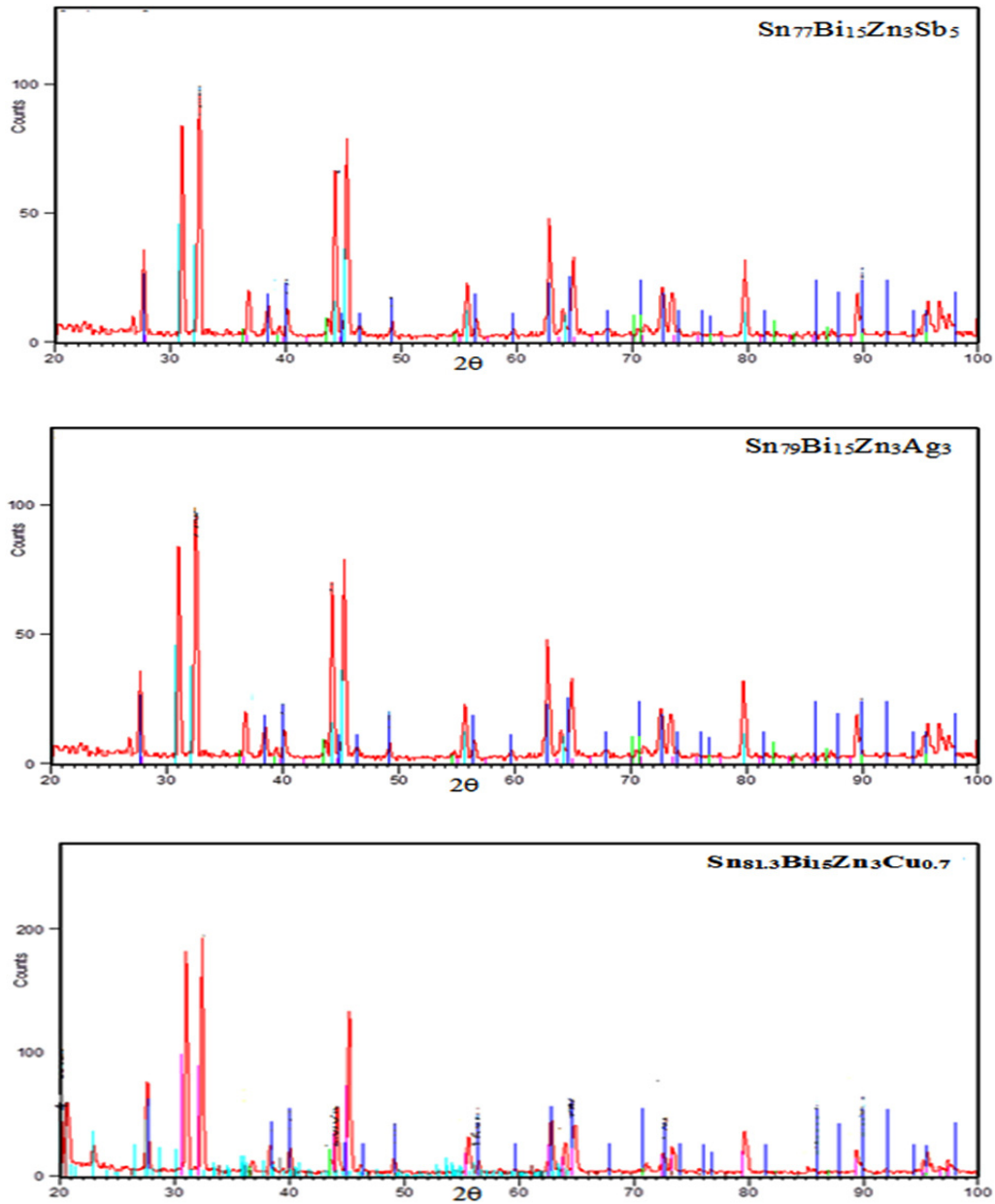


Fig. 1: x-ray diffraction patterns of used alloys

Table 1: x-ray diffraction analysis of used alloys

X- ray analysis of $\text{Sn}_{82}\text{Bi}_{15}\text{Zn}_3$ alloy						
2θ	d Å	Phase	hkl	Int. %	FWHM	Area
27.7026	3.22025	Bi	012	14.14	0.1968	7.77
31.1119	2.87471	Sn	101	55.11	0.1968	30.27
32.5097	2.75423	Sn	101	100	0.2165	60.42
38.5038	2.33814	Bi	104	5.82	0.1968	3.2
40.1776	2.24451	Bi	110	6.68	0.2165	4.04
44.2828	2.0455	Bi	015	16.9	0.1968	9.28
45.2954	2.0021	Sn	211	67.56	0.2165	40.81
49.2336	1.85078	Bi	202	3.6	0.2755	2.77
55.7043	1.65015	Sn	301	14.33	0.1771	7.09
62.8549	1.47854	Sn	112	25.84	0.1771	12.77
64.059	1.45362	Sn	400	6.84	0.1771	3.38
64.8801	1.4372	Sn	321	17.48	0.1771	8.64
72.6214	1.3019	Sn	420	4.32	0.1968	2.37
73.3889	1.29017	Sn	411	9.72	0.1574	4.27
79.7631	1.20233	Sn	312	22.21	0.1771	10.98
89.6001	1.09409	Bi	306	7.06	0.1771	3.49
95.6897	1.03908	Sn	332	6.29	0.192	4.55
X- ray analysis of $\text{Sn}_{77}\text{Bi}_{15}\text{Zn}_3\text{Sb}_5$ alloy						
2θ	d Å	Phase	hkl	Int. %	FWHM	Area
27.7042	3.22007	Bi	012	17.16	0.2558	11.31
31.1312	2.87297	Sn	101	57.74	0.1968	29.29
32.4757	2.75703	Sn	101	100	0.2362	60.88
38.4274	2.34261	Bi	104	9.83	0.2558	6.48
40.1071	2.2483	Bi	110	7.81	0.1968	3.96
44.2389	2.04743	Bi	015	22.19	0.1968	11.26
45.2974	2.00202	Sn	211	66.53	0.2362	40.5
46.432	1.95571	Bi	021	2.28	0.4723	2.78
49.2009	1.85193	Bi	202	6.04	0.1968	3.07
55.6878	1.6506	Sn	301	17.09	0.2755	12.14
56.5179	1.62831	Bi	024	3.33	0.2362	2.03
62.8382	1.47889	Sn	112	22.52	0.1771	10.28
64.1038	1.45272	Sn	400	5.66	0.2755	4.02
64.869	1.43742	Sn	321	18.33	0.2362	11.16
72.6852	1.30091	Sn	321	6.48	0.3149	5.26
73.3804	1.2903	Sn	411	10.83	0.1574	4.4
79.7308	1.20274	Sn	312	20.74	0.1771	9.47
89.5697	1.09439	Bi	306	6.33	0.1771	2.89
95.7368	1.03955	Sn	431	5.62	0.1771	2.57
X- ray analysis of $\text{Sn}_{79}\text{Bi}_{15}\text{Zn}_3\text{Ag}_3$ alloy						
2θ	d Å	Int. %	Phase	hkl	FWHM	Area
27.6551	3.22567	38.81	Bi	012	0.2755	8.52
30.9967	2.88513	100	Sn	200	0.2952	23.53

27.6551	3.22567	38.81	Bi	012	0.2755	8.52
30.9967	2.88513	100	Sn	200	0.2952	23.53
32.5771	2.74868	96.75	Sn	101	0.3936	30.35
36.834	2.44021	19.43	Zn	002	0.2952	4.57
38.4397	2.34189	14.96	Bi	104	0.3149	3.75
40.1119	2.24804	12.6	Bi	110	0.3149	3.16
44.1699	2.05047	71.65	Bi	015	0.3149	17.98
45.32	2.00107	93.86	Sn	211	0.3542	26.5
49.2037	1.85183	6.58	Bi	202	0.4723	2.48
55.7294	1.64947	25.4	Sn	301	0.3149	6.38
62.8183	1.47931	55.04	Sn	112	0.2165	9.5
64.92	1.43641	37.82	Sn	321	0.2952	8.9
72.5841	1.30248	23.23	Sn	321	0.3936	7.29
73.5035	1.28844	20.42	Sn	411	0.3936	6.41
79.72	1.20287	36.48	Sn	312	0.2558	7.44
89.5297	1.09477	19.27	Bi	306	0.2362	3.63
95.5592	1.04015	9.52	Sn	431	0.576	5.91

X-ray analysis of $\text{Sn}_{81.3}\text{Bi}_{15}\text{Zn}_3\text{Cu}_{0.7}$ alloy						
2θ	d Å	Int. %	Phase	hkl	FWHM	Area
20.1708	4.40243	18.7	Bi	003	0.1378	4.5
20.5734	4.31719	27.91	Bi	003	0.2362	11.5
23.008	3.86558	10.11	Bi	101	0.3149	5.56
27.5571	3.23692	38.35	Bi	012	0.2558	17.12
30.9694	2.88761	98.21	Sn	200	0.2952	50.59
32.3271	2.76937	100	Sn	101	0.3149	54.95
36.834	2.44021	4.07	Zn	002	0.3542	2.52
38.3222	2.3488	11.32	Bi	104	0.1968	3.89
40.0254	2.2527	11.27	Bi	110	0.2755	5.42
43.5884	2.07646	5.37	Sn	220	0.3149	2.95
44.1079	2.05321	29.44	Bi	015	0.1968	10.11
45.1593	2.00782	71.92	Sn	211	0.2952	37.05
49.0546	1.85711	6.03	Bi	202	0.3149	3.32
55.5567	1.65418	15.86	Sn	301	0.2165	5.99
56.4327	1.63057	4.81	Bi	024	0.3149	2.64
62.7327	1.48112	24.21	Sn	112	0.3149	13.3
64.1149	1.45249	11.48	Sn	400	0.2755	5.52
64.7323	1.44012	17.05	Sn	321	0.3542	10.54
72.5751	1.30261	7.75	Sn	321	0.3936	5.32
73.3377	1.29094	11.46	Sn	411	0.3936	7.87
79.6088	1.20427	19.58	Sn	312	0.1771	6.05
89.4527	1.09551	9.83	Bi	306	0.2362	4.05
95.5354	1.04035	6.66	Sn	431	0.576	9.05

Scanning Electron Microscope Analysis (SEM)

Fig. 2 shows the scanning electron micrographs of $\text{Sn}_{82}\text{Bi}_{15}\text{Zn}_3$, $\text{Sn}_{77}\text{Bi}_{15}\text{Zn}_3\text{Sb}_5$, $\text{Sn}_{79}\text{Bi}_{15}\text{Zn}_3\text{Ag}_3$ and $\text{Sn}_{81.3}\text{Bi}_{15}\text{Zn}_3\text{Cu}_{0.7}$ alloys. $\text{Sn}_{82}\text{Bi}_{15}\text{Zn}_3$ alloy has lamellar structure (tin phase as gray color and

bismuth phase as black color) contained white color (zinc or bismuth-tin) with different shape and size. $\text{Sn}_{77}\text{Bi}_{15}\text{Zn}_3\text{Sb}_5$ alloy has a good symmetric lamellar structure (gray broad lamellar structure and black small width lamellar) contained white small crystals

with different shape. $\text{Sn}_{79}\text{Bi}_{15}\text{Zn}_3\text{Ag}_3$ alloy has a mixed lamellar structure and white color along at the lamellar boundary. $\text{Sn}_{81.3}\text{Bi}_{15}\text{Zn}_3\text{Cu}_{0.7}$ alloy has a heterogeneous structure and white color as slabs

with different shape, size and orientation. From SEM graphs analysis, it obvious that, these alloys have heterogeneous structure which confirmed from x-ray and DSC results.

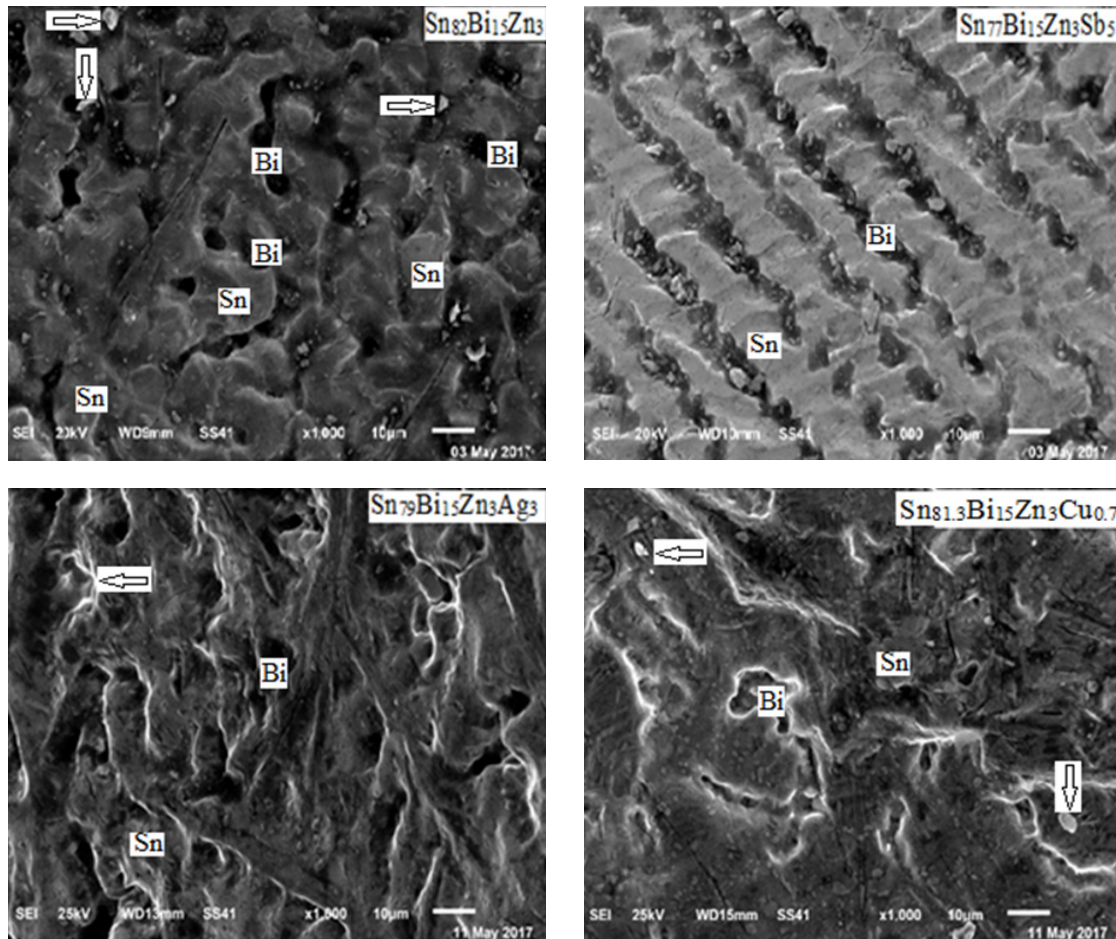


Fig. 2: scanning electron micrographs of used alloys

Soldering Properties

Thermal Behavior

Thermal analysis depend on the nature of solid phase and on its temperature. They used to study solid state transformations as well as solid liquid reactions. The DSC thermographs were obtained with heating rate $10\text{ }^{\circ}\text{C}/\text{min}$ in the temperature range $0\text{--}400\text{ }^{\circ}\text{C}$. DSC thermographs of $\text{Sn}_{82}\text{Bi}_{15}\text{Zn}_3$, $\text{Sn}_{77}\text{Bi}_{15}\text{Zn}_3\text{Sb}_5$, $\text{Sn}_{79}\text{Bi}_{15}\text{Zn}_3\text{Ag}_3$ and $\text{Sn}_{81.3}\text{Bi}_{15}\text{Zn}_3\text{Cu}_{0.7}$ alloys are shown in Figure 3 (a-d). DSC graphs show many peaks with different intensity, broadness and position. That is meant, these alloys contained many phases with different crystallinity, crystal

size and orientation. The melting temperature of used alloys as shown in Table 2 dependent on alloys composition. Also it is less than the tin eutectic based alloys and very closed with commercial lead-tin solder alloy ($183\text{ }^{\circ}\text{C}$).

Wetting Behavior

Wett ability is quantitatively assessed by the contact angle formed at the solder substrate's flux triple point. The contact angles of $\text{Sn}_{82}\text{Bi}_{15}\text{Zn}_3$, $\text{Sn}_{77}\text{Bi}_{15}\text{Zn}_3\text{Sb}_5$, $\text{Sn}_{79}\text{Bi}_{15}\text{Zn}_3\text{Ag}_3$ and $\text{Sn}_{81.3}\text{Bi}_{15}\text{Zn}_3\text{Cu}_{0.7}$ alloys on pure Cu substrate are listed in Table 3. The contact angle of $\text{Sn}_{82}\text{Bi}_{15}\text{Zn}_3$ alloy increased after adding Sb

or Ag or Cu. That is meant the spreading of used alloys molten on pure Cu at room temperature in air dependent on alloy composition (cohesive force and

adhesive force). Also these alloys have good wetting process (contact angles less than 50 °C) compared to its eutectic alloys^{1,2,4}.

Table 2: melting temperature of used alloys

Alloys	Melting temperature °C	Based alloys	Melting temperature °C
$\text{Sn}_{82}\text{Bi}_{15}\text{Zn}_3$	178.6	$\text{Sn}_{91}\text{Zn}_9$	199
$\text{Sn}_{77}\text{Bi}_{15}\text{Zn}_3\text{Sb}_5$	189.7	$\text{Sn}_{95}\text{Sb}_5$	242.06
$\text{Sn}_{79}\text{Bi}_{15}\text{Zn}_3\text{Ag}_3$	186.48	$\text{Sn}_{96.5}\text{Ag}_{3.5}$	223.81
$\text{Sn}_{81.3}\text{Bi}_{15}\text{Zn}_3\text{Cu}_{0.7}$	185.24	$\text{Sn}_{99.3}\text{Cu}_{0.7}$	231.55

Table 3: contact angles of used alloys

Alloys	Contact angle (θ°)		(θ°)
$\text{Sn}_{82}\text{Bi}_{15}\text{Zn}_3$	21.25	$\text{Sn}_{91}\text{Zn}_9$	46
$\text{Sn}_{77}\text{Bi}_{15}\text{Zn}_3\text{Sb}_5$	26	$\text{Sn}_{95}\text{Sb}_5$	27
$\text{Sn}_{79}\text{Bi}_{15}\text{Zn}_3\text{Ag}_3$	27.25	$\text{Sn}_{96.5}\text{Ag}_{3.5}$	36
$\text{Sn}_{81.3}\text{Bi}_{15}\text{Zn}_3\text{Cu}_{0.7}$	31	$\text{Sn}_{99.3}\text{Cu}_{0.7}$	43

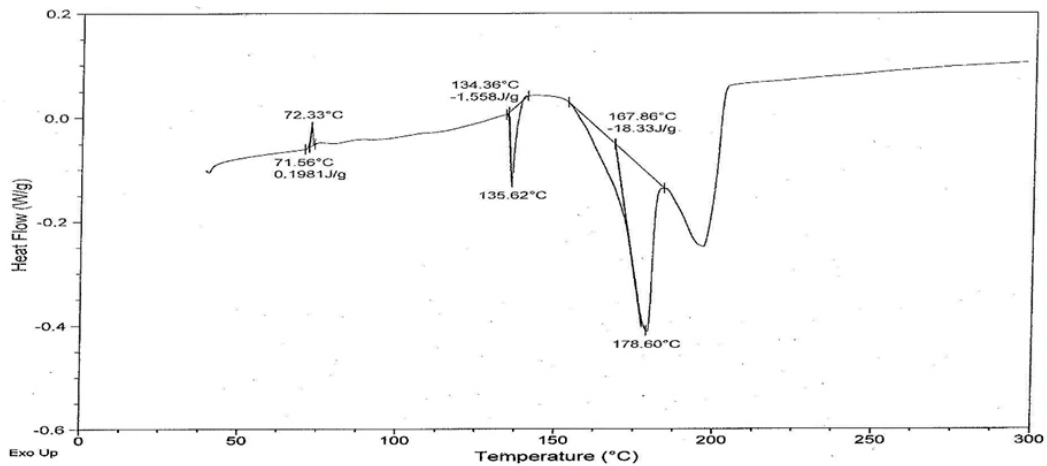


Fig. 3a: DSC graph of $\text{Sn}_{82}\text{Bi}_{15}\text{Zn}_3$ alloy

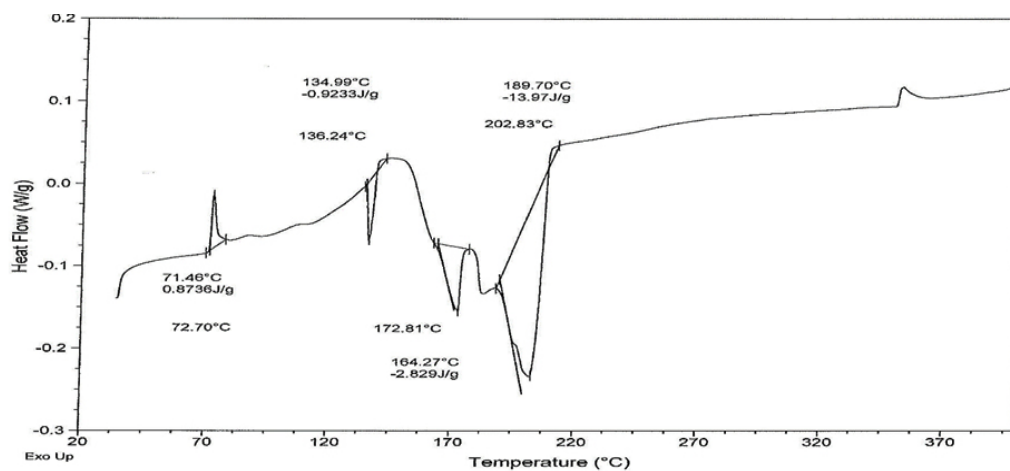


Fig. 3b: DSC graph of $\text{Sn}_{77}\text{Bi}_{15}\text{Zn}_3\text{Sb}_5$ alloy

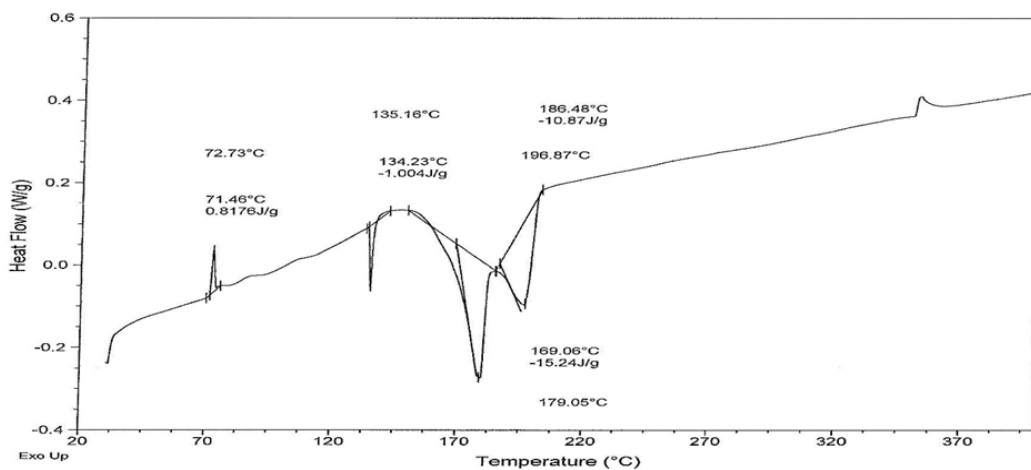


Fig. 3c: DSC graph of $\text{Sn}_{79}\text{Bi}_{15}\text{Zn}_3\text{Ag}_3$ alloy

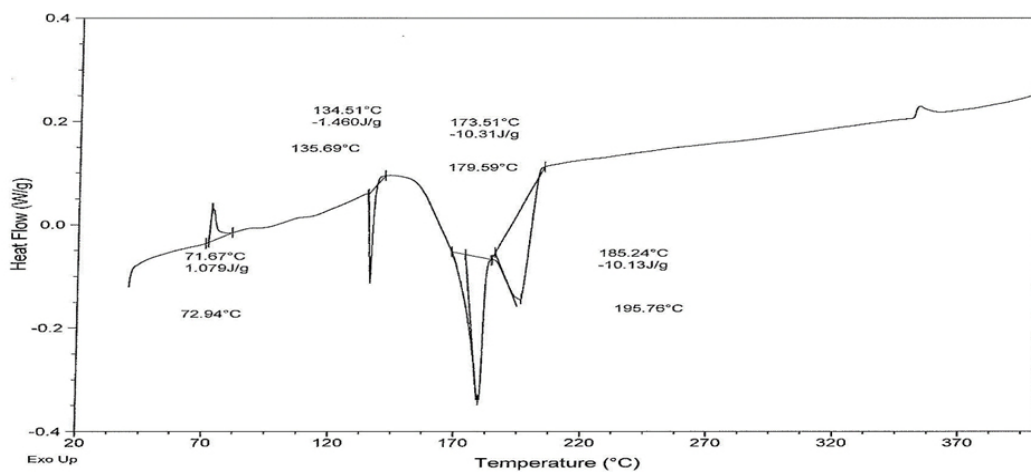


Fig. 3d: DSC graph of $\text{Sn}_{81.3}\text{Bi}_{15}\text{Zn}_3\text{Cu}_{0.7}$ alloy

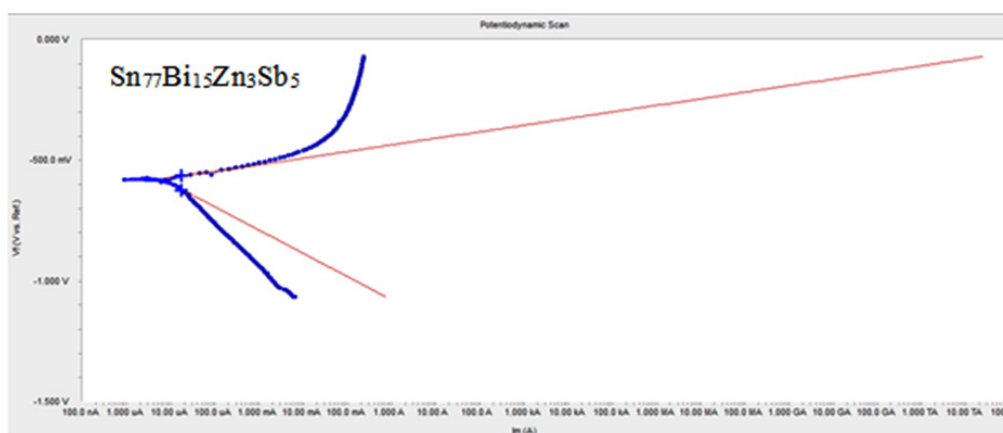
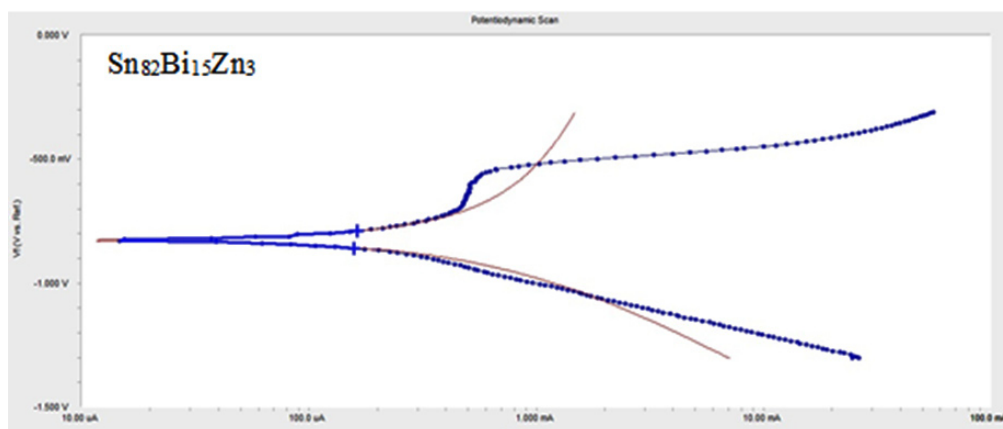
Electrochemical Corrosion Behavior

Figure 4 shows electrochemical polarization curves of $\text{Sn}_{82}\text{Bi}_{15}\text{Zn}_3$, $\text{Sn}_{77}\text{Bi}_{15}\text{Zn}_3\text{Sb}_5$, $\text{Sn}_{79}\text{Bi}_{15}\text{Zn}_3\text{Ag}_3$ and $\text{Sn}_{81.3}\text{Bi}_{15}\text{Zn}_3\text{Cu}_{0.7}$ alloys in 0.25 M HCl. The corrosion potential of used alloys exhibited a negative potential. Also the cathodic and the anodic polarization curves showed similar corrosion trends. The corrosion potential (E_{corr}), corrosion current (I_{corr}) and corrosion rate (C. R) of $\text{Sn}_{82}\text{Bi}_{15}\text{Zn}_3$,

$\text{Sn}_{77}\text{Bi}_{15}\text{Zn}_3\text{Sb}_5$, $\text{Sn}_{79}\text{Bi}_{15}\text{Zn}_3\text{Ag}_3$ and $\text{Sn}_{81.3}\text{Bi}_{15}\text{Zn}_3\text{Cu}_{0.7}$ alloys are presented in Table 4. The corrosion rate of $\text{Sn}_{82}\text{Bi}_{15}\text{Zn}_3$ alloy in 0.25 M HCl is decreased by adding Sb or Cu but it's increased by adding Ag content. That is because adding Sb or Ag or Cu caused a heterogeneous microstructure (more phases) with dissolved atoms in matrix that effected on microsegregation and reactivity of atoms with HCl solution.

Table 4: Corrosion parameters of used alloys

Alloys	E_{corr} mV	I_{corr} μm	C.R mpy
$\text{Sn}_{82}\text{Bi}_{15}\text{Zn}_3$	-862.0	841	348.3
$\text{Sn}_{77}\text{Bi}_{15}\text{Zn}_3\text{Sb}_5$	-578.0	639	292.1
$\text{Sn}_{81.3}\text{Bi}_{15}\text{Zn}_3\text{Cu}_{0.7}$	-792.0	650	297.2
$\text{Sn}_{79}\text{Bi}_{15}\text{Zn}_3\text{Ag}_3$	-949.0	828	378.2



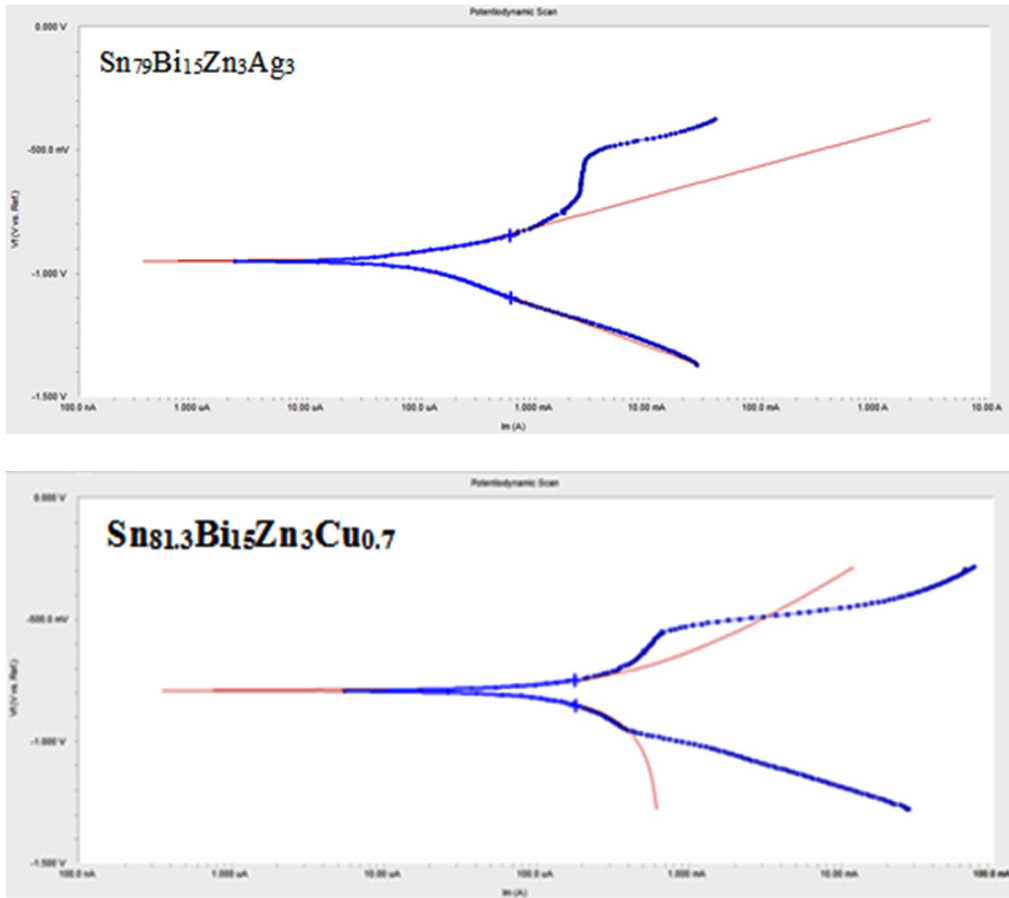


Fig. 4: electrochemical polarization curves of used alloys

Conclusion

In this work, it can produced lead free solder alloys with superior soldering properties (melting temperature and wettability) and corrosion resistance compared to eutectic and non-eutectic tin based solder alloys by adding different alloying elements.

Melting temperature and contact angle of tin- zinc, tin- antimony and tin- copper alloys decreased after adding bismuth content. Corrosion resistance of $\text{Sn}_{82}\text{Bi}_{15}\text{Zn}_3$ alloy in 0.25 M HCl increased after adding Sb or Cu element.

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