Metallographic study of Sn-Bi-Sb-In alloy prepared by rapid solidification

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Abstract

Sn_{70-X} at.% -Bi_{15} at.% -Sb_{15} at.%- In_x at.% alloy ribbons were produced using melt-spinning technique. The surfaces were characterized with Optical Microscopy. Also, scanning electron microscopy combined with energy dispersive X-ray analysis (SEM-EDX). The results contribute to the understanding of the microstructure evolution in alloys of this type prepared by melt spinning technique. Microscopy can give information concerning a material's composition, previous treatment and properties. Particular features of interest are grain size, phases present, Chemical homogeneity, distribution of phases and, elongated structures formed by melt spinning technique.

Keywords

Rapid Solidification (RS); Lead Free Solder Alloys; Sn-Bi-Sb, optical microscope, Scanning electron microscope (SEM), Energy dispersive X-ray analysis (EDX)

Academic Discipline And Sub-Disciplines

Physics; material science

SUBJECT CLASSIFICATION

Solder alloy

TYPE (METHOD/APPROACH)

Experimental

Introduction

Optical metallography, one of three general categories of metallography, entails examination of materials using visible light to provide a magnified image of the micro- and macrostructure. In scanning electron microscopy (SEM), the second category, the surface of the specimen is bombarded with a beam of electrons to provide information for producing an image.

Optical microscopy and, occasionally, SEM are used to characterize structure by revealing grain boundaries, phase boundaries, inclusion distribution, and evidence of mechanical deformation. Scanning electron microscopy is also used to characterize fracture surfaces, integrated circuits, corrosion products, and other rough surfaces, especially when elemental microanalysis of small features is desired.

The melt-spinning technique is very important rapid solidification processing exhibit unusual properties due to their structure evolution. Rapid solidification of metallic alloys results in refined microstructures with reducing micro segregation and improves mechanical properties of the final products as compared to normal castings. The rapidly solidified Sn-based solders by melt spinning have proved to be suitable for soldering at low temperature and short soldering duration [1-3]. It has been established that rapid solidification can produce high strength structural materials for use in tools and bearing components, high temperature materials, corrosion-resistant materials, catalytic and storage materials and, finally, electrical and magnetic materials [4-5]. These properties actually depend on the structural changes, intermetallic compound formation produced in each particular case [6]. Rapid solidification can produce different type of phases [7-9]. Metallic ribbons have many applications namely in stretchable electronics and sensing where their surfaces may play an important role because they will bind to other systems (bio-molecules in bio-sensors, as an example) and their surface properties (surface Plasmon resonance, for instance) are important.

In the present investigation, rapidly quenched alloys Sn_{70-x}-Bi_{15}-Sb_{15}-In_x (x= 1 , 3, 5 and 7 in at. %) were examined by optical microscope, scanning electron microscope and energy dispersive spectroscopy x-ray. Another work of these alloy had been established by [10]. Knowledge of the microstructure of a material, which is the number of phases present, their distribution, volume fraction, shape, and size, are essential since many properties are structure sensitive, e.g. mechanical and electrical properties. Thus this section is concerned with the study of the microstructure of the alloys which have been prepared to explain their properties.

Experimental procedures

The materials used in the present work are Sn, Sb, Bi, and In fragments, with purity was better than 99.99% produced by a single copper roller (200 mm in diameter) melt-spinning technique [11-13]. The process parameters such as the ejection temperature, and the linear speed of the wheel were fixed at 873 K and 30.4 ms^{-1} respectively The polished and etched samples were observed using an optical microscope (Olympus Model: PMG 3) with the objective of determining the microstructural evolution. The microstructure analysis was carried out on a scanning electron microscope (SEM) of type (FEI Inspect-S50) operate at 30Kv with high resolution 3nm. Quantx Bruker EDS spectrometer System is a full-featured X-Ray Microanalysis System (EDX/EDS) for SEM result.

Sample preparation

Specimens were mounted at room temperature using acrylics castable mounting materials (cold resin). Then, the specimens were mechanically ground using emery papers up to 1200 grade. Scratches from the abrasive papers are removed by polishing specimens for several minutes on a wheel covered with a short-nap cloth impregnated with 0.25 µm diamond paste. The samples rinsed and degreased with acetone, and then etched using 5 ml nitric acid + 2ml HCL in 100 ml methanol for 45s.

2- Results and Discussion

2.1 Imaging

Using melt spinning technique there are two main surfaces in contact with the rotating wheel and exposed to the air. The two surface in contact with the rotating wheel (CS) looked, at naked eye, very different from the surface in contact with air (AS): AS was significantly duller than CS as shown by both sides pictures displayed in Figure 1. In our work, we study the surface which contact with the rotating wheel (CS) looked and all images after polishing and etching.

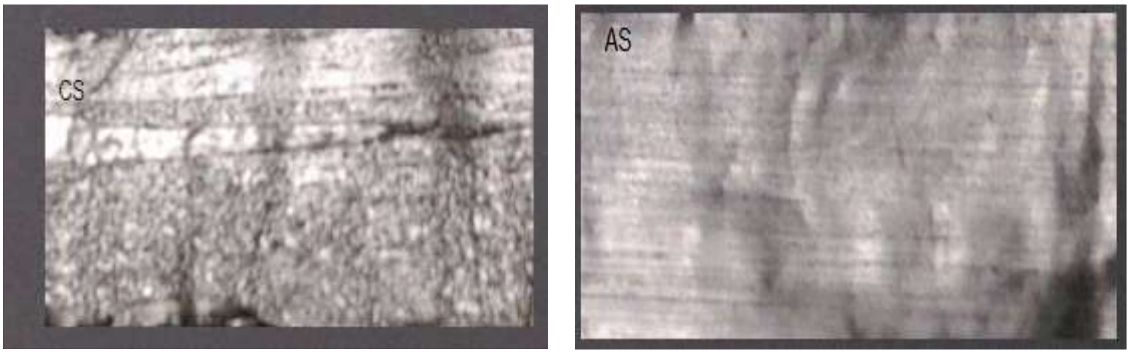
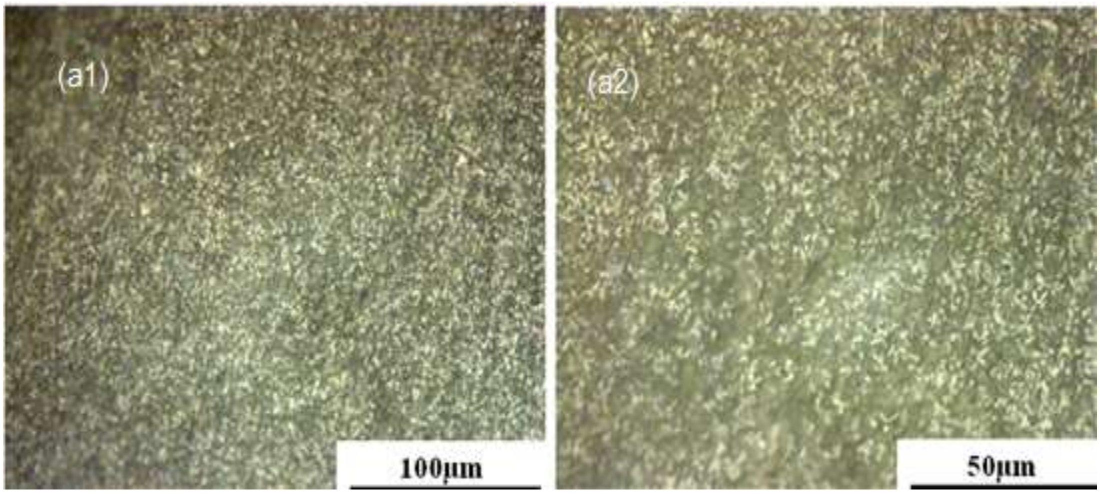


Fig. 1 Photograph of ribbon surfaces CS and AS.

2.2 Optical microscope

A more detailed view of the alloys surfaces is shown in figure (2) where optical microscopy images show that the surfaces have different structures for each prepared alloy.



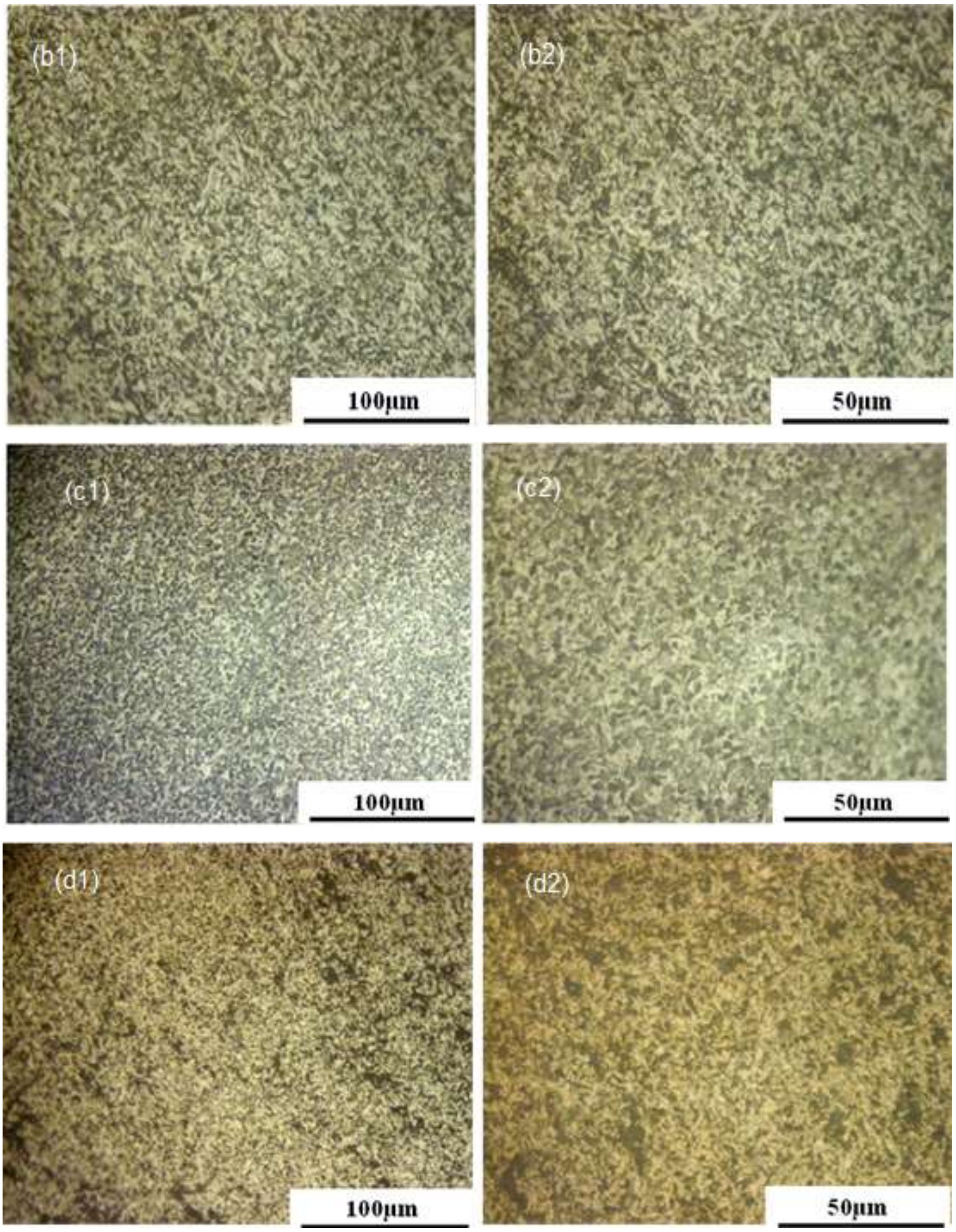


Fig. 2 Optical Microscopy images for (a) Sn_{69}-Bi_{15}-Sb_{15}-In_1; (b) Sn_{67}-Bi_{15}-Sb_{15}-In_3; (c) Sn_{65}-Bi_{15}-Sb_{15}-In_5; and (d) Sn_{63}-Bi_{15}-Sb_{15}-In_7.

2.3 SEM and EDX

Figure (3) shows the SEM image of alloy Sn_{69}-Bi_{15}-Sb_{15}-In_1, EDS Spectra of the points 1 and 6 of EDS spot analysis and EDS map analysis of the alloying elements shows a homogenous distributions of the alloying elements in alloy $Sn_{70}Bi_{15}Sb_{15}$. Figures (3a1, 3a2) shows the formation of the soft phase (Sn) nearly continuous network in the Sn_{69}-Bi_{15}-Sb_{15}-In_1 alloy. The grains in the middle of ribbon, which looks like a rod in shape, are extended along the length direction (the direction of the linear momentum of the wheel). The number of grains per mm2 (Mav) was found to be 716; the total projected area is 115 μm2; mean grain area is 160*10-15 m2; mean grain size is 0.36 μm; total grain volume is 65*10-21 m3.

Figures (4a1, 4a2) show the SEM image for alloy $Sn_{67}Bi_{15}Sb_{15}In_3$. Figures (4b1, 4b2) are shown EDS Spectra of the points 1 and 5 of EDS spot analysis and figure 4c is the EDS map analysis of the alloying elements.

Figures (5a1, 5a2) show the SEM image for alloy $Sn_{65}Bi_{15}Sb_{15}In_5$, EDS Spectra of the points 1 and 4 of EDS spot analysis shown in figures (5b1, 5b2) and EDS map analysis of the alloying elements showing the homogenous distributions of the alloying elements for alloy $Sn_{65}Bi_{15}Sb_{15}In_5$ shown in figure 5c.

Figures (6a1,6a2) show the SEM image for alloy $Sn_{63}Bi_{15}Sb_{15}In_7$, figures (6b1, 6b2) show EDS Spectra of the points 1 and 4 of EDS spot analysis. Figure 6c show EDS map analysis of the alloying elements showing the homogenous distributions of the alloying elements for alloy $Sn_{66}Bi_{15}Sb_{15}In_4$.

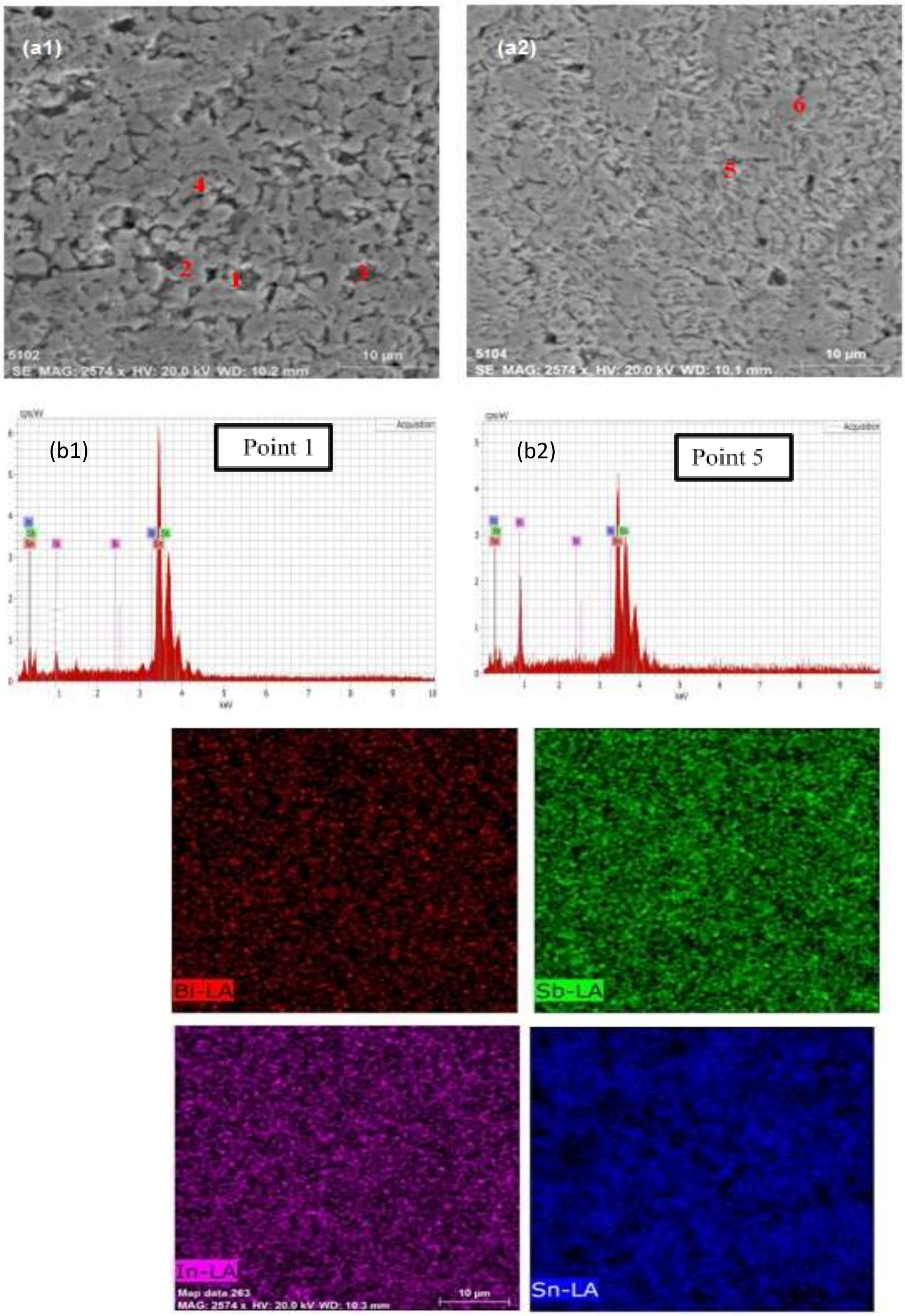


Fig. 3: (a) SEM images (b) EDS spectra of the points 1 and 5 and (c) EDS map analysis for $Sn_{69}Bi_{15}Sb_{15}In_1$ alloy.

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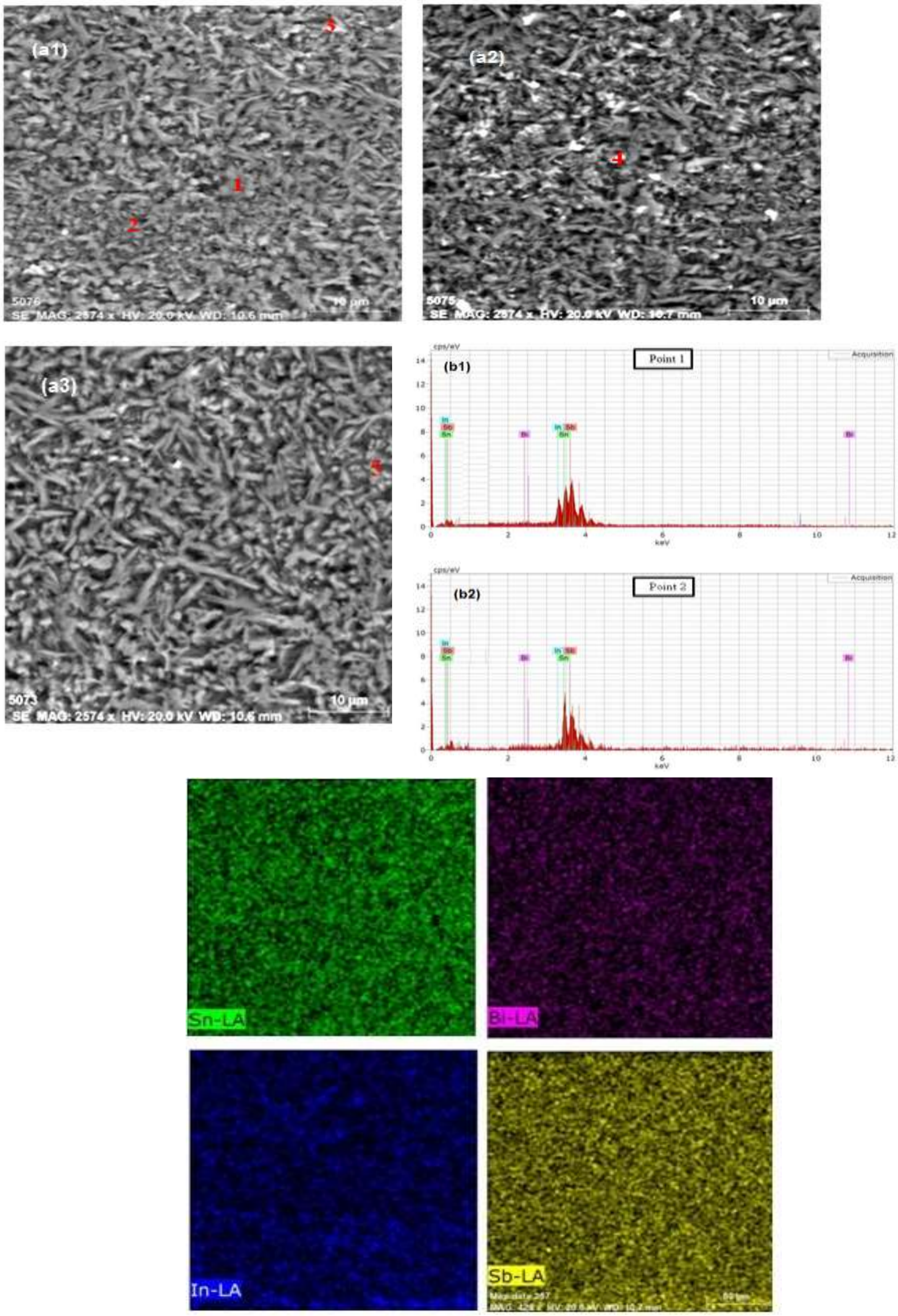

Fig. 4 (a) SEM images (b) EDS spectra of the points 1 and 2 and (c) EDS map analysis for $Sn_{67}Bi_{15}Sb_{15}In_{3}$ alloy.

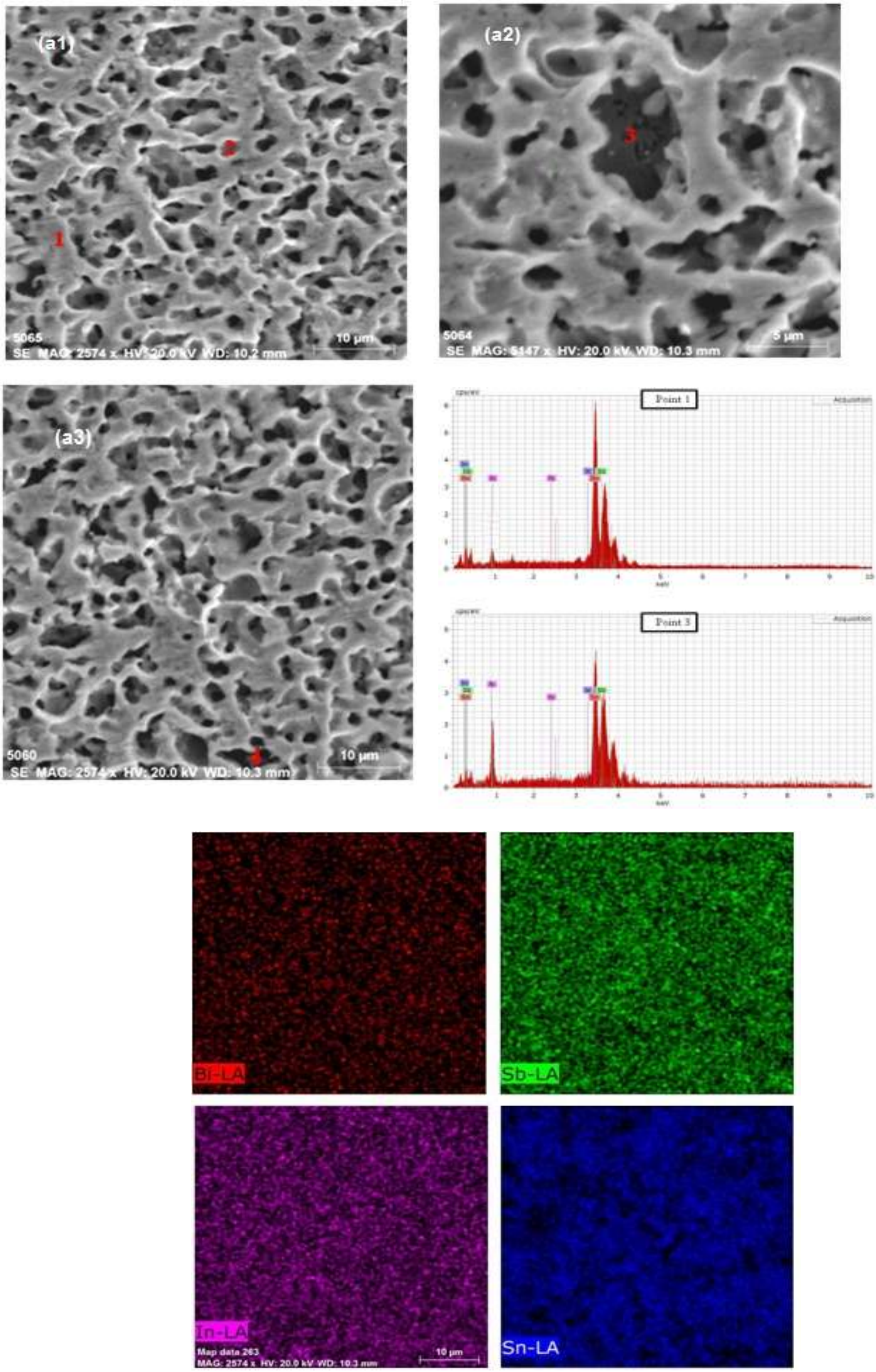

Fig. 5: (a) SEM images (b) EDS spectra of the points 1 and 3 and (c) EDS map analysis for $Sn_{65}Bi_{15}Sb_{15}In_5$ alloy.

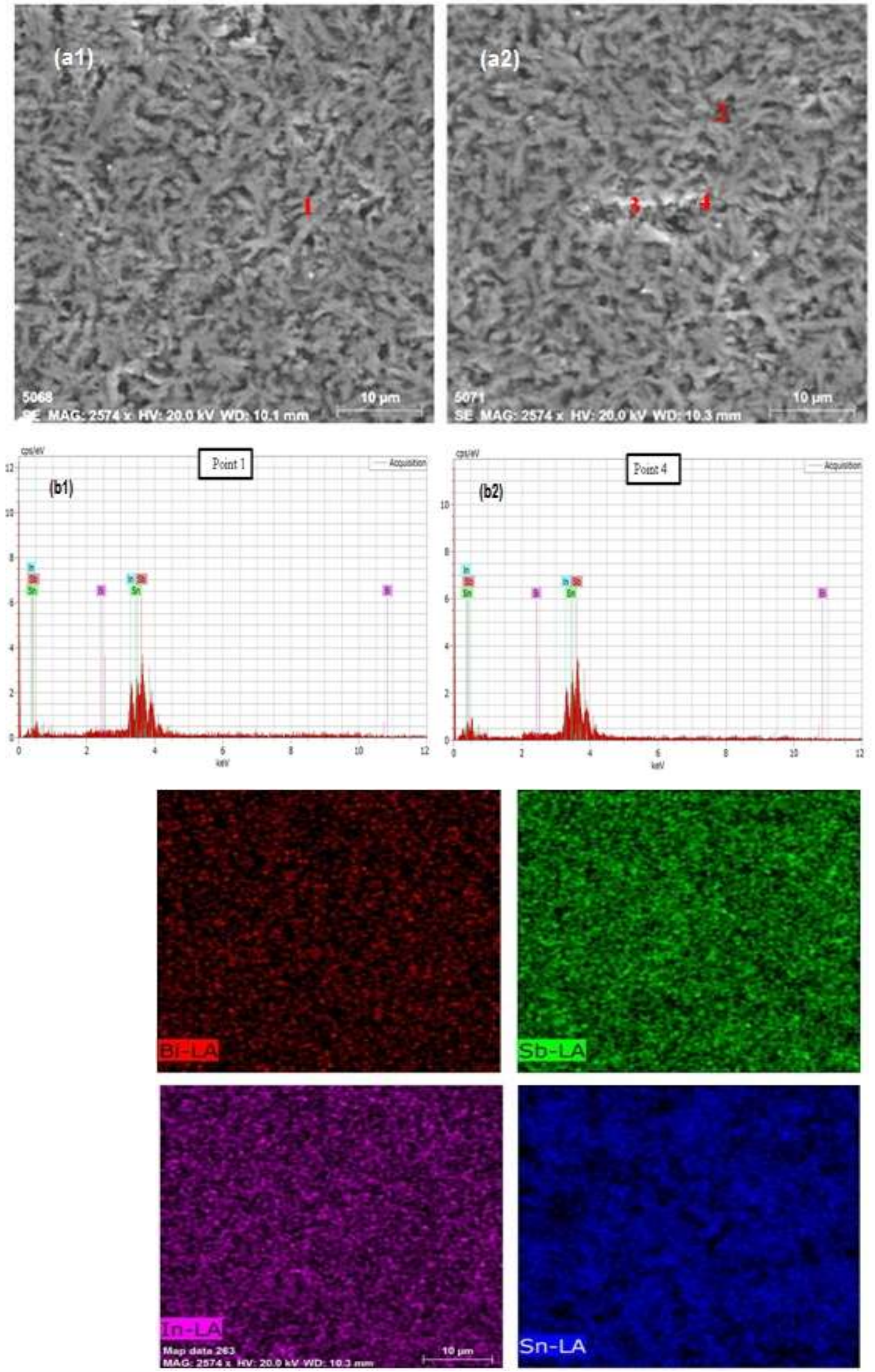

Fig. 5: (a) SEM images (b) EDS spectra of the points 1 and 3 and (c) EDS map analysis for $Sn_{63}Bi_{15}Sb_{15}In_7$ alloy.

Table (2) EDS spot analysis of the points 1, 2, 3, 4, 5 and 6 in the SEM images for all alloys. Table (3) shows the variation of number of grains and the grain size for different alloys. Crystallite and grain are both single crystals; a crystallite is a single crystal in powder form but a grain is a single crystal within a bulk/thin film.

A particle is also thought of as an agglomerate, small enough in size to not consider it as a bulk or thin film, but composed of 2 or more individual crystallites. From the figures, it shows that the microstructures were composed of elongated grains, a few microns in width and several microns in length. Also evident in the micrographs was a highly density of extinction contours which indicated that considerable internal strain was introduced in the material as a result of melt spinning.

Table 2. EDS spot analysis of the points 1, 2, 3, 4, 5 and 6 in the SEM images for all prepared alloys.

Alloy	Element	Wt.%					
		Point 1	Point 2	Point 3	Point 4	Point 5	Point 6
$Sn_{69}Bi_{15}Sb_{15}In_1$	Sn	88.26	52.26	66.86	79.13	72.04	81.41
	Sb	1.27	21.91	17.20	10.15	12.51	7.04
	In	7.73	3.54	7.72	7.28	6.82	7.45
	Bi	2.27	22.29	8.41	3.44	7.56	4.10
$Sn_{67}Bi_{15}Sb_{15}In_3$	Sn	32.39	40.07	35.92	23.90	37.39	
	Sb	43.03	34.28	40.79	51.12	35.39	
	In	16.39	17.22	0.9	7.14	23.10	
	Bi	8.08	8.42	22.28	16.17	4.12	
$Sn_{65}Bi_{15}Sb_{15}In_5$	Sn	65.02	29.73	64.57	68.61		
	Sb	0.0	0.91	1.29	0.0		
	In	27.54	14.80	27.93	28.79		
	Bi	7.44	54.55	6.21	2.6		
$Sn_{63}Bi_{15}Sb_{15}In_7$	Sn	38.21	38.78	35.63	40.27		
	Sb	26.12	24.08	20.91	26.51		
	In	31.92	33.06	38.41	25.43		
	Bi	3.75	4.08	5.04	7.78		

Table 3. Grain details for all prepared alloys.

Alloy	Number of grains	Total projected area (μm^2)	Mean grain area (10^{-15} m^2)	Mean grain size (μm)	Total grain volume (10^{-21} m^3)
Sn_{79}-Bi_{15}-Sb_{15}- In_1	716	115	160	0.36	65
Sn_{67}-Bi_{15}-Sb_{15}- In_3	415	82	189	0.41	61
Sn_{65}-Bi_{15}-Sb_{15}- In_5	833	142	171	0.36	91
Sn_{63}-Bi_{15}-Sb_{15}- In_7	859	127	148	0.34	78

3. Conclusion

From the present study the data shows that:

- It's found that, there are homogenous distributions of the alloying elements for all prepared alloys by melt spinning technique.
- The latest number of grains is for Sn_{67}-Bi_{15}-Sb_{15}- In_3 alloy and the largest number is for Sn_{63}-Bi_{15}-Sb_{15}- In_7 alloy.

- Mean grain size is equal for Sn_{79}-Bi_{15}-Sb_{15}- In_1 alloy and Sn_{65}-Bi_{15}-Sb_{15}- In_5 alloy. The latest grain size is for Sn_{63}-Bi_{15}-Sb_{15}- In_7 alloy and the largest grain size is for Sn_{67}-Bi_{15}-Sb_{15}- In_3 alloy.
- The melt spinning technique produce new microstructures and new phases and these play important role for new applications.

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