ISSN : 0974 - 7486 Volume 14 Issue 6

Materials Science

An Indian Journal

Full Paper

MSAIJ, 14(6), 2016 [227-234]

Effects of Y_2O_3 contents on microstructure and hardness properties of Sn-Ag-Cu-Y_2O_3 composite solders

Emad M.Ahmed

Material Science and Engineering group, Physics Department, Faculty of science, Taif University, 21974 Taif, P.O.Box 888, (KINGDOM OF SAUDI ARABIA)

Solid State Physics Department, National Research Center, Dokki, Cairo, (EGYPT)

E-mail: makoul67@yahoo.com

ABSTRACT

In this study, varyingof Y_2O_3particles from 0 to 1 wt.% were incorporated intoSn-1Ag-0.5Cu (SAC-0) plain solder matrix to form composite solders of 0.2 wt.%Y_2O_3 (SAC-0.2), 0.5wt.%Y_2O_3(SAC-0.5) and 1wt.%Y_2O_3 (SAC-1). Optical (OM) and scanning electron microscopy (SEM) photographs show that Y_2O_3 particles depressed clearly the growth dendrite β-Sn grains. Since the Y_2O_3 particles serve as additionalnucleation sites for the formation of primary Sn-rich phase,the size of β-Sn grains were decreasedgradually from 10-15 toμm 2-5 μm range, while the eutectic areas around the β-Sn grain were shrunk with higher density of intermetallic particles and Y_2O_3 particles. XRD results confirmed mainly the presence of β-Sn, Ag_3Snphases in all composite solders and the presence of Y_2O_3 clearly in SAC-1 composite solder. Moreover, lattice parameter of β-Sn was decreased by increasing of Y_2O_3content in the composite solders referring toenhancement ofCu solubilityin β-Sn for composite solders of higher Y_2O_3content. This microstructure improvement of composite solders leads to microhardness enhancement of SAC-0.2, SAC-0.5 and SAC-1 composite solders with 10.6%, 16.3% and 29% compared to the plain solders SAC-0, respectively.

KEYWORDS

Sn-1Ag-0.5Cu;
Microhardness;
Microstructure;
Lead-free solder.

INTRODUCTION

Low temperature Pb-containingSn based solder alloys have been widely used in electronic package due to their excellent properties. However, due to toxicity of Pb, there has been substantial concerns about the further use of that solders. Therefore, there has been an increase interest in developing alternative Pb-free solders in recent years. There are various alloys as candidates, such as Sn-Cu, Sn-Ag, Sn-Bi system. Although the free Pb-containingSn based solder alloys have received much attention, their mechanical properties and reliabilities are still not better. One of the alternative approaches is develop composite solders. The composite solders consist of a solder matrix and reinforced particles such as intermetallics, metallic powders, carbon fibers or fine oxide particles[1-4]. Among the lead-free solder candidates, the eutectic tin–silver-copper alloy has received much attention as a low meltingpoint solder to replace Pb-containing solder alloys ininterconnects of surface mount microelectronic assemblies[5-7]. However, the intermetallic compound (IMC) growth in Sn-Ag-Cu solder joints is faster than that in eutectic Sn-Pb solder joints[8,9]. It is well known that in Sn-containing solder joint, solder bonds with a Cu substrate through the formation of a dual Cu-Sn IMC layer consisting of Cu and Cu_3Sn that exist between the solder and Cu substrate. Because the IMC layers are more brittle than that of the solder matrix, they can be a site of mechanical weakness, causing failure of the joint with fractures in the IMC layer itself or along the interface between solder and IMC layer[10]. In this study, an innovative method for producing thecomposite solders which consist of a Sn-1Ag-0.5Cu soldermatrix and Y_2O_3 particles is introduced. The purpose ofthis work is to investigate the effect of the addition ofY_2O_3particles on microstructures and microhardness of Sn-1Ag-0.5Cu solder.

EXPERIMENTAL PROCEDURES

Sn-1Ag-0.5Cu-xY_2O_3 lead free solder alloys were made by using pure Sn, Ag, Cu and Y_2O_3 (purity of 99.9%) with *x* of 0, 0.2, 0.5,and 1 wt.%, respectively. These solder alloys with Y_2O_3 contents of 0, 0.2, 0.5, and 1 wt% were labeled as SAC-0, SAC-0.2, SAC-0.5, and SAC-1, respectively. The process of melting was carried out in a muffle furnace to produce rod-like specimen with diameter of 10 mm. The melt was held at 500 °C for 100 minto complete the dissolution of Sn, Ag and Cu. The Sn-1Ag-0.5Cu lead free base composite solders were prepared by mechanically dispersing 0.2, 0.5 and 1wt% of micro-Y_2O_3 particles into the melt. The melt then poured in a graphite mold to prepare the chillcast ingot. TABLE 1 lists the actual chemical composition of the experimental alloys used in the present investigation. The microstructure was examined by optical microscopy (OM) and scanning electron microscopy (SEM) with an energy dispersive X-ray spectrometer (EDS) after etching. A solution of 2%HCL, 3%HNO_3 and 95% (vol.%) ethyl alcohol was prepared and used to etch the samples. Thermal analysis has been performed using Differential Thermal Analysis (DTA) to study the effect of Y_2O_3 contents on the melting point of the studied composite solder through heating the samples in DTA under argon with heating rate of 10 °C/min. Phase identification of the alloys samples was carried out by X-raydiffractometry at 40 KV and 20 mA using Cu K_α radiation with diffraction angles (2θ) from 20° to 90° and a constant scanning speed of 1°/min. Individualphases and their crystal structures were identified by matching the characteristic XRD peaks against JCPDS data. Hardness was measured at room temperature using the Vickers hardness Leitz Wetzlar Germany instrument with loads of 250 g. A total of 10 measurements were performed on the longitudinal section of each sample and the average is taken as the microhardness value.

RESULTS AND DISCUSSION

OM, SEM and EDX of the plain and composite solders

The addition of Y_2O_3micro-particles into the SAC-0 solder influences final microstructures significantly. Figure 1 shows a cross-section images of solders as a function of Y_2O_3 concentration. OM bright field and cross-polarized images are employed to analyze the microstructure of β-Sn grains, β-Sn dendrites and different phases. OM bright ûeld images can represent both grain boundaries and dendrite cell boundaries, but cannot distinguish differ-

TABLE 1 : Chemical composition of SAC-0, SAC-0.2, SAC-0.5 and SAC-1 composite solders

Alloy	Cu	Ag	Y_2O_3	Sn
SAC0	0.5	1	-	Bal.
SAC1	0.5	1	0.2	Bal.
SAC2	0.5	1	0.5	Bal.
SAC3	0.5	1	1	Bal

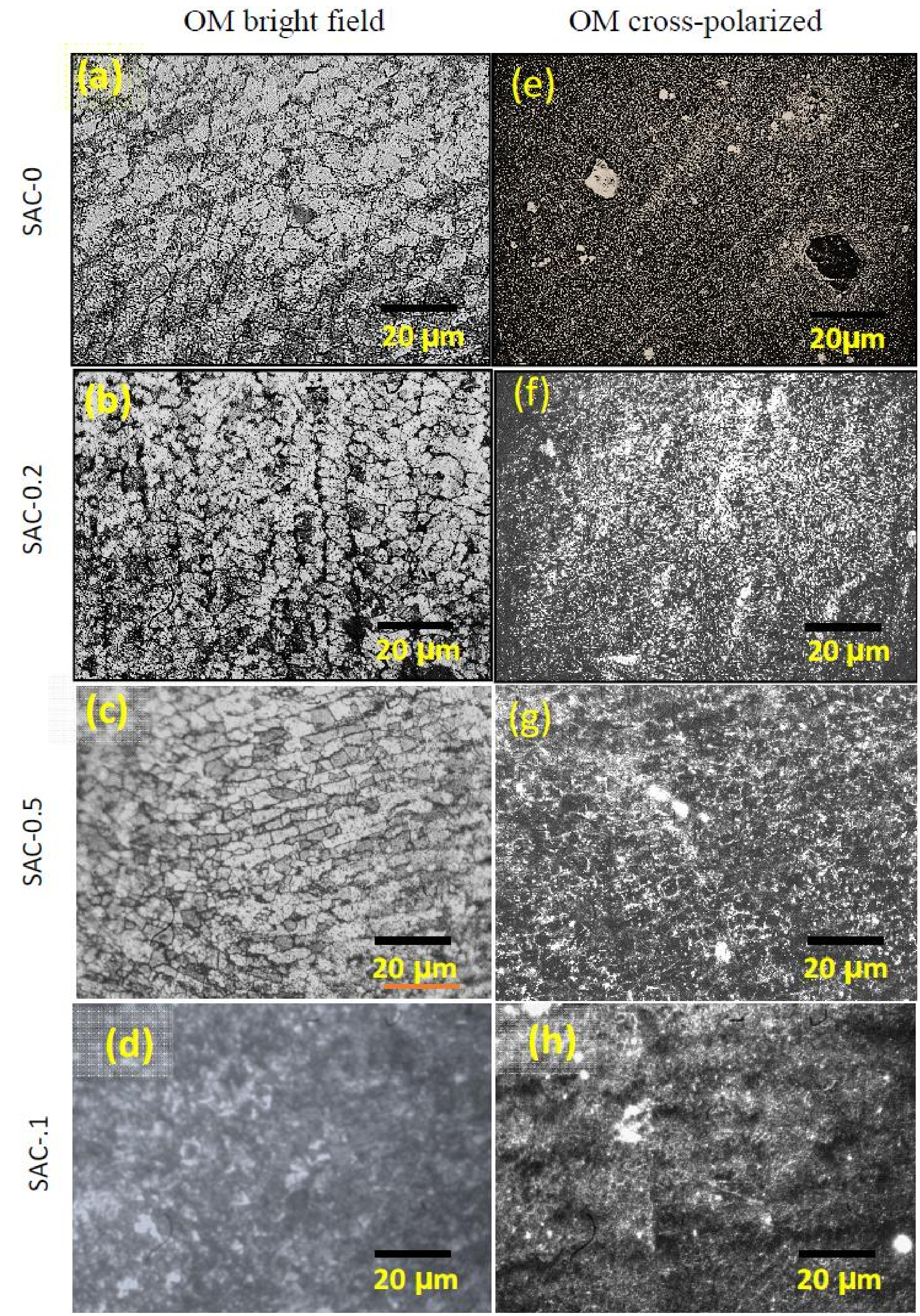

Figure 1 : Optical micrographs (bright field and cross-polarized) of SAC-0, SAC-0.2, SAC-0.5 and SAC-1 composite solders

Full Paper

ent phases as shown in Figure 1 (a-d). However, cross-polarized images can tell different phases and deûne grain boundaries as shown in Figure 1 (e-h). Figure 1 (a-d) show the bright field images of SAC-0, SAC-0.2, SAC-0.5 and SAC-1 solders cooled in air. It is obvious that, β-Sn grains became ûner as well as diverse in orientations as increasing of Y_2O_3 content. In addition, the grain boundaries become sharperas shown in Figure 1 (c). Moreover, the darker contrast precipitations and the dense phase in between β-Sn are attributed to Y_2O_3 and Sn-Ag-Cu eutectic respectively. Figure 1 (e-h) represent the cross-polarized images of the same composite solders. The microstructure of the intermetallic Ag_3Sn (white) is clearly observed as shown in Figure 1 (e) and become finer as the increasing of Y_2O_3 as shown in Figure 1 (f), (g) and (h). Moreover, homogeneous distribution of Y_2O_3 particles through the β-phase in SAC-1 composite solder was confirmed as shown in Figure 2.

As the plain SAC-0sample is not an eutectic solders but near the eutectic composition, the volume of ratio of the dendrite β-Sn phase was not very high as usually obtained for eutectic solder (average grain size = 26 μm), and its average grain size was relatively smaller (10-15 μm). Addition of a small percentage of Y_2O_3 particles to the plain solder was observed to alter the as solidified condition SEM microstructure. A huge effect of Y_2O_3 on the β-Sn grain size and the eutectic areas was clearly observed in the SEM photographs of SAC-0, SAC-0.2, SAC-0.5 and SAC-1 compositesolders as shown in Figure 3. These revealed significant improvements in the refinement of dendrite β-Sn grains, Ag_3Sn grains and Ag_3Sn phase. β-Sn grain size has been decreased from 10-15 to 2-5 μm range while the eutectic areas around the β-Sn grain were shrunk with higher density of intermetallic particles and Y_2O_3 particles. The size and spacing between Ag_3Sn grains in the composite solder matrix decrease with increase of Y_2O_3 particles. According to the EDS analysisshown in Figure 4, the eutectic areas were found to contain Sn, Ag, Cu and O. As the solders used were Sn-1Ag-0.5C solders intermixing micro-Y_2O_3 particles, Cu element is solution in the matrix and Ag_3Sn was the only precipitate phase containing Ag element in the solder, the network eutectic areas can be considered to consist of submicro-Ag_3Sn and micro-Y_2O_3particles.

Thermal analysis

Melting temperature is one of the most vital considerations for development of new solder alloys. Regarding solder composites, the melting point should not increase much more than that of the plain solder alloy otherwise the solder composites loses their advantages. The results of DTA analysis for solder composites are presentedin Figure (5) and summarized in TABLE 2. The onset (T_{onset}) and melting temperatures (T_m) of SAC-0, SAC-0.2, SAC-

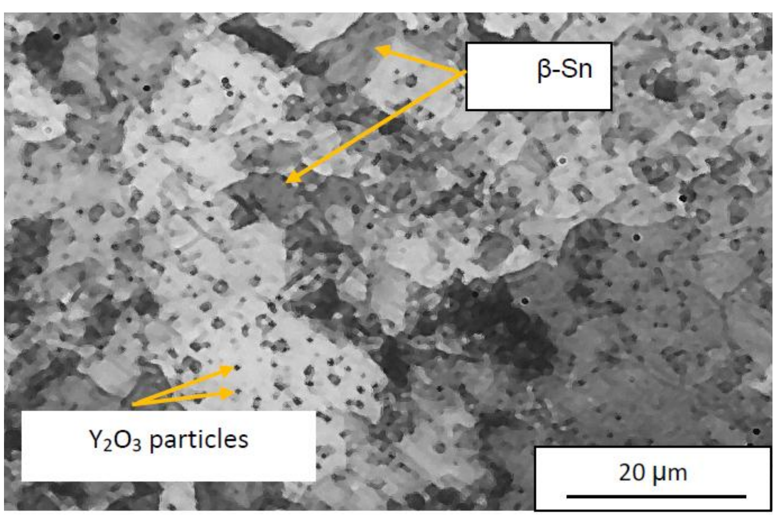

Figure 2 : Optical micrograph (bright field) of SAC-1 composite solder showing Y_2O3 particles distribution in addition to different phases; β-Sn andAg_3Sn

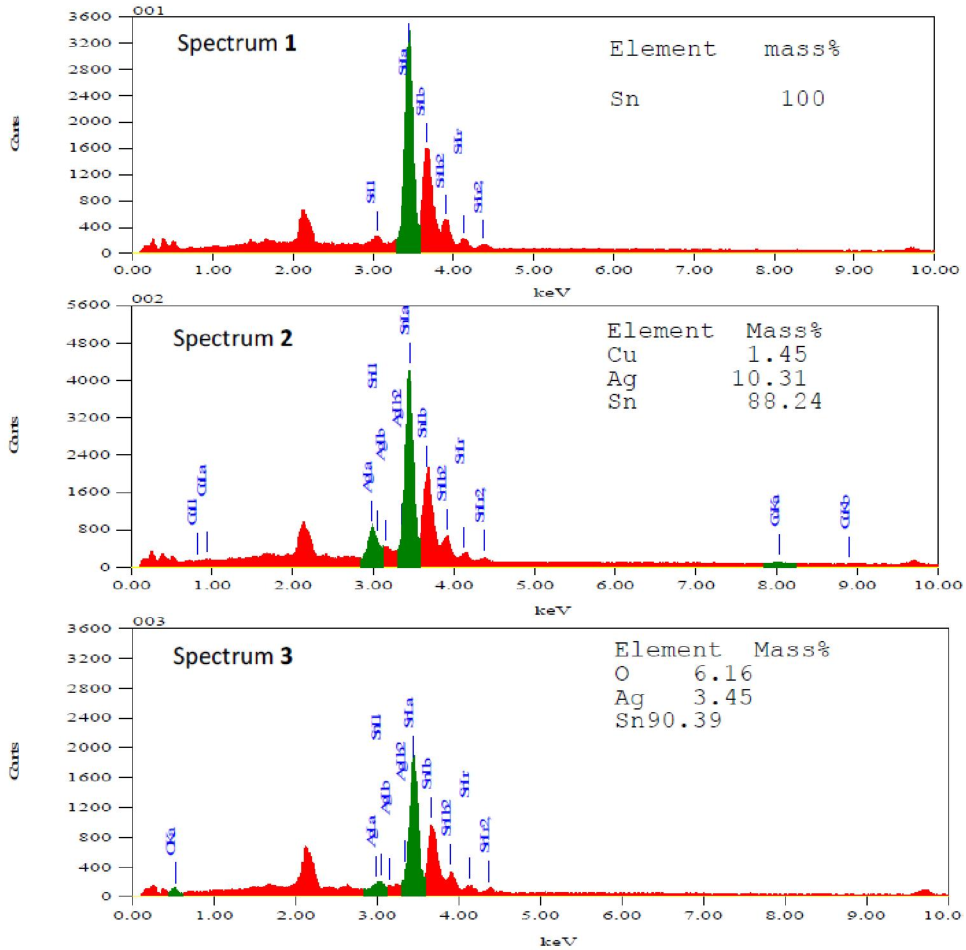

Figure 3 : Secondary electron micrographs of SAC-0, SAC-0.2, SAC-0.5 and SAC-1 composite solders

0.5 and SAC-1 solder composites are, respectively, 219.6 °C, 223.4 °C, 223.6 °C, 224 and 226.3 °C[11], 228.9 °C, 227.8 °C, 228.7. These results confirm that addition of Y_2O_3 has a little effect on the T_{onset} and T_m. This slightincreasein the melting point of the composites older couldbe possibly attributed to the change in the surface in stability and the variation in physical properties of grainb oundary/interfacial characteristics.

XRD analysis

To identify the phase structures of SAC-0, SAC-0.2 andSAC-1, XRD was conducted and the corresponding pattern sare presented in Figure 6. Only large peaks intensity of β-Sn and smallpeaks of Ag_3Snphases have been detected. XRD lines corresponding to Y_2O_3 could not be observed at low content of Y_2O_3sample (x=0.2), while they appear clearly for higher content of Y_2O_3sample (x=1). This indicated that the Y_2O_3 micro-particles have been successfully blended with the SAC solders. XRD analysis shows a clear decrease of lattice constant for SAC-1 comparing to the SAC-0 as shown in TABLE 3, indicating to the increase of the content of solute Cu atoms, of lower atomic radius, in β-Sn matrix. As a result, microstrain of β-Sn has been observed to increase clearly by increasing of Y_2O_3 content.

MICROHARDNESS

Vickers microhardness values represented average hardness values of ten intents performed at different regions of SAC-0, SAC-0.2, SAC-0.5 and SAC-1composite solders. The influence of Y_2O_3micro-particles on microhardness of composite solders is summarized in TABLE 4 and compared in the bar graph of Figure 7. It can be seen that the microhardness of the original SAC-0 composite solder is as low as 14.3HV, while those of SAC-0.2,

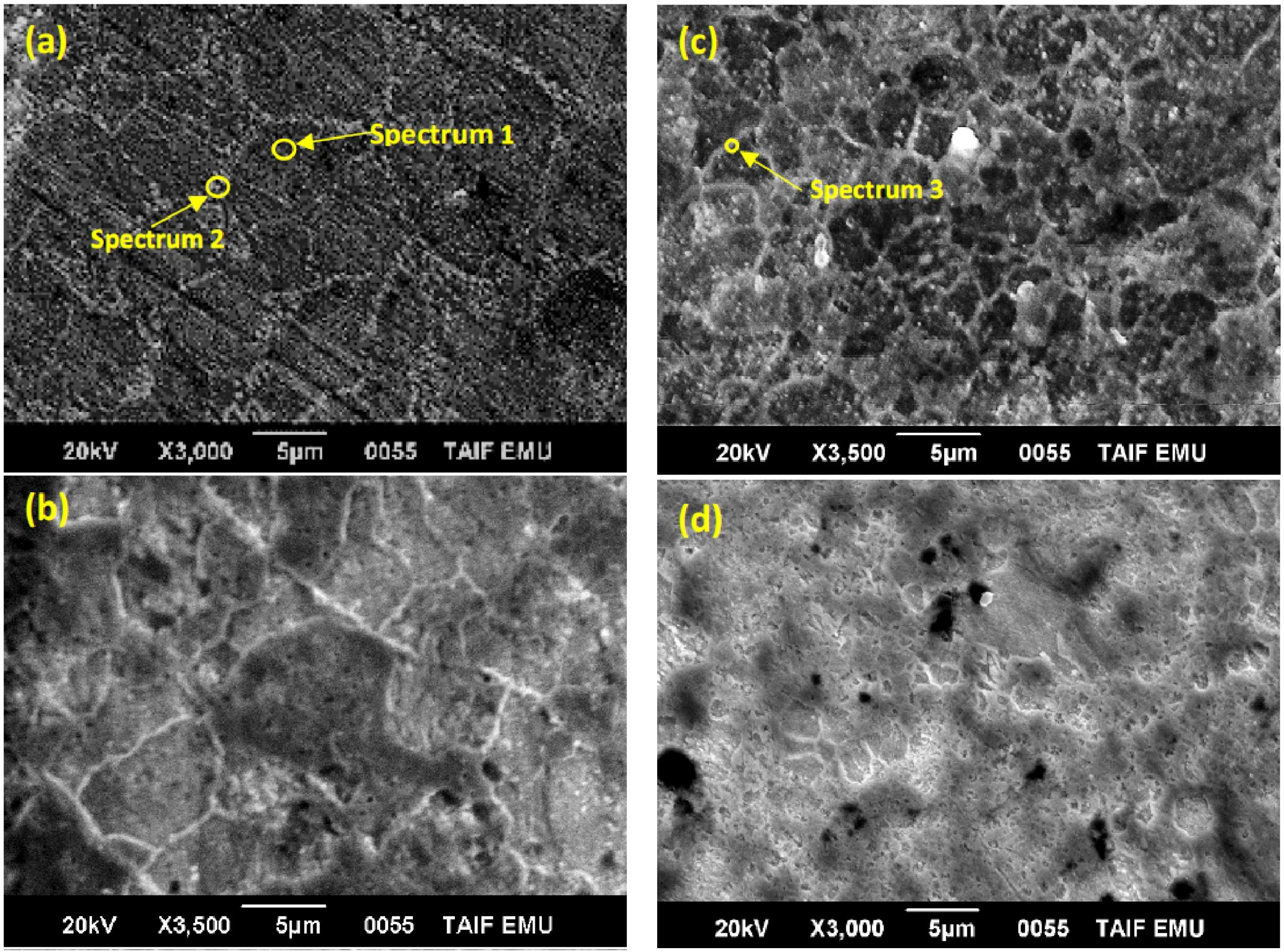

Figure 4 : EDX signals of points 1, 2 and 3 represented in Figure 3

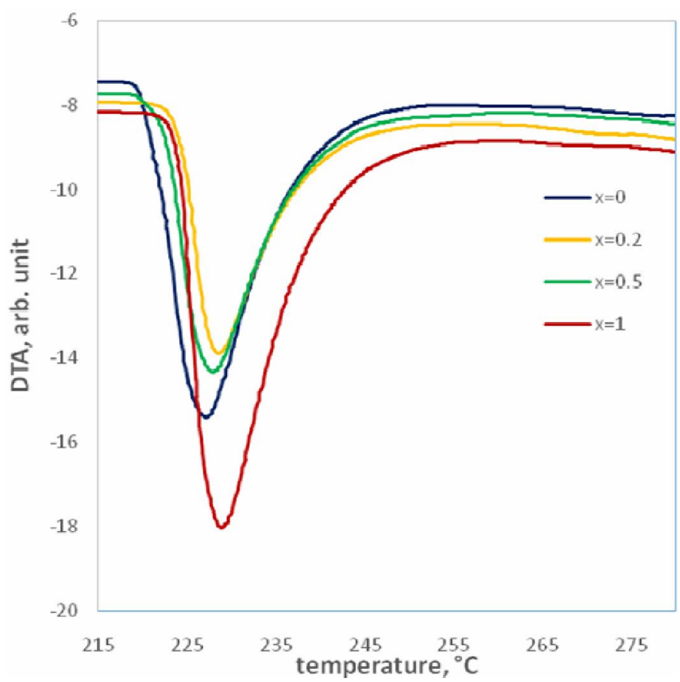

Figure 5 : DTA signalsduringheating (endothermal) of SAC-0, SAC-0.2, SAC-0.5 and SAC-1 composite solders

TABLE 2 : Solidus temperature (T_{onset})and peak temperature for solder composites alloys during heating curve

Composite Solder	T_{onset}	Peaktemperature
SAC-0	219.6	226.3
SAC-0.2	223.4	228.9
SAC-0.5	223.6	227.8
SAC-1	224.0	228.7

TABLE 3 : Lattice constant, crystallite size and microstrain% of pure β-Snas a reference, and β-Sn in SAC-0, SAC-0.2 and SAC-1 composite solders

Lattice constant	Crystallite size (nm)	microstrain% (nm)	
β-Sn	a= 0.58316	--------	-------
	c= 0.31813		
SAC-0	a=0.58275	89	0.1374
	c=0.31770		
SAC-0.2	a=0.58298	91	0.1356
	c=0.31798		
SAC-1	a=0.58190	86	0.1556
	c=0.31708		

TABLE 4 : Microhardness results of SAC-0, SAC-0.2, SAC-0.5 and SAC-1composite solders

Specimens	Y_2O_3	Microhardness (Hv)
SAC-0	Nil	14.3
SAC-0.2	0.2	15.6
SAC-0.5	0.5	16.4
SAC-1	1	18.2

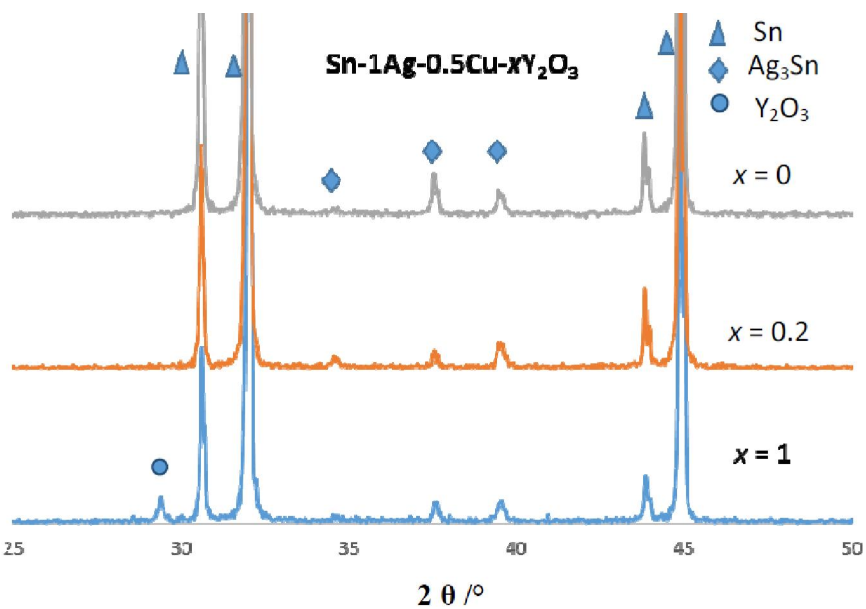

Figure 6 : XRD patterns of SAC-0, SAC-0.2 and SAC-1 composite solders

SAC-0.5 and SAC-1 composite soldersincrease to15.6, 16.4,and18.2 HV, respectively. It is also found that the microhardness enhancement of these Y_2O_3-containing composite solders are10.6%, 16.3% and 29% compared with Y_2O_3-free non-composite solders. This enhancement might be attributed to the reduction in the grain size of the β-Sn and spacing between Ag_3Sn grains in solder matrix in

Full Paper

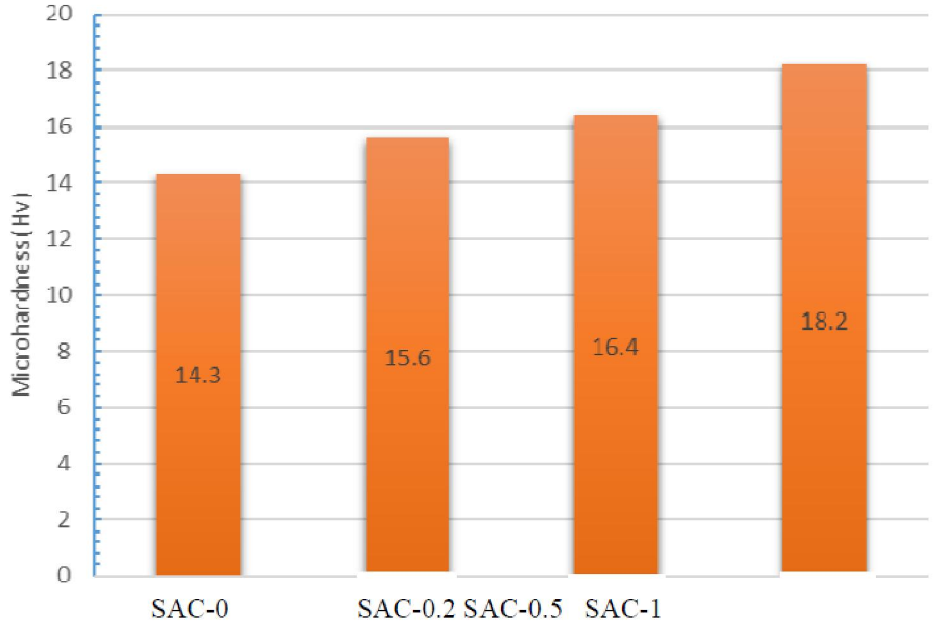

Figure 7 : Microhardness comparison on bar graph ofSAC-0, SAC-0.2, SAC-0.5 and SAC-1 composite solders

addition to the pinning of linear dislocations. Moreover, the presence of homogeneous distribution of Y_2O_3particles in the solder matrix, and the refinement of the intermetallic compounds could act as reinforcement.

CONCLUSION

In this work, the influence of Y_2O_3 micro-particles addition on themicrostructure, and microhardness of Sn-1Ag-0.5Cu-xY_2O_3 composite solderswereinvestigated. Significant microstructural changes were observed in composite solder specimens. β-Sn grain size was decreased from 10-15 to 2-5 μm range. In addition, the eutectic areas around the β-Sn grain were shrunk with higher density of intermetallic particles and Y_2O_3 particles. Thermal analysis confirmed that addition of Y_2O_3 has a little effect on the melting point of the studied composite solders. OM and XRD analysis show that Y_2O_3particles have been successfully blended with the SAC-0 solder and Cu solubility in β-Sn and microstarin% of β-Sn increase by increasing of Y_2O_3 contents. Microhardness of the studied composite solders has been improved owing to the reduction in the grain size of the β-Sn and spacing between Ag_3Sn grains, homogeneous distribution of Y_2O_3 micro-particles in the solder matrix and refinement of the intermetallic compounds.

REFERENCES

[1] S.H.J.F.LiMannan, M.P.Clode *et al.*; Acta Mater., **55**, 737 **(2007)**.
[2] W.Dong, Y.Shi*et al.*; J.Electron.Mater., **37**, 982 **(2008)**.
[3] S.M.L.Nai, J.Wei, M.Gupta; J.Alloy.Compd., **473**, 100 **(2009)**.
[4] Z.X.Li, M.Gupta; Adv.Eng.Mater., **7**, 1049 **(2005)**.
[5] E.P.Wood, K.L.Nimmo; Journal of Electronic Materials, **23**, 709 **(1994)**.
[6] M.R.Hassison, J.H.Vincent, H.A.H.Steen; Soldering and Surface Mount Technology, **13**, 21 **(2001)**.
[7] S.Gadag, S.Patra; Journal of Electronic Materials, 29, 1392 **(2000)**.
[8] T.Y.lee, W.J.Choi, K.N.Tu; J.W.Jang, S.M.Kuo, J.K.Lin; Journal of Materials Research, **17**, 291 **(2002)**.
[9] D.R.Frear, J.W.Jang, J.K.Lin, C.Zhang; Journal of Metals, **53**, 28 **(2001)**.
[10] Y.G.Lee, J.G.Duh; Journal of MaterialsScience: Materials in Electronics, **11**, 33 **(2000)**.
[11] A.A.El-Daly , G.S.Al-Ganainy , A.Fawzy , M.J.Younis; Materials and Design, **55**, 837 **(2014)**.