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Effect of Terbium Additions on Microstructural, Thermal and Mechanical Properties of Eutectic Sn-3.5Ag Pb- Free Solder for Low Cost Electronic Assembly

Rizk Mostafa Shalaby*, Fatma Elzahraa Ibrahim, Mostafa Kamal

Metal Physics Laboratory, Physics Department, Faculty of Science, Mansoura University, Mansoura, Egypt, B.O.Box: 35516

*Corresponding author: rmibrahim99@gmail.com, rizk2002@mans.edu.eg

Abstract

This work methodically concentrated on the effect of a trace amount of rare earth element terbium, Tb (0.1, 0.2, 0.3, 0.4 and 0.5 wt. %) on the properties of eutectic Sn-3.5 wt. %Ag were studied. The results indicated that addition of Tb rare earth can be refined the microstructure of the solder and intermetallic compound (IMC) Ag₃Sn phase appeared in the solder matrix. Add a few quantity of rare earth Tb enhances the hardness and strength of eutectic Sn-Ag lead free solder joint. Also, results indicate that adding Tb to the eutectic Sn-3.5Ag remarkably enhances solderability, reliability, thermal and mechanical properties. It is also found that increasing in mechanical strength can depend on crystalline size refining in addition to some regular precipitates from IMC, Ag₃Sn.

Keywords: eutectic Sn-Ag; lead free solder; Terbium additions; microstructures; mechanical properties; thermal properties.

1. Introduction

Sn-Pb solders has been used before as interconnect materials extensively because of reliable, low cost ,the ease of processing , good solderability , good strength and forming of small intermetallic products . Nevertheless, the present industry going bound to the green products industry because of the health hazard and environmental regards standing on Pb solder alloys. At 2006, the EU has ordered the environmental standard called the ROHS demand that permitted the existence of substances in final products by less of 1,000 ppm (Ref 1). It is worth mentioning that the utilization of solders became an imperative for the interconnection, packing of practically all electronic devices and circuit present. Lead free solder alloy have been used widely as a part of electronic circuit for its excellent properties (Ref 2, 3).The type of lead free Sn-Ag solder is considered as a good alternative to Sn-Pb solder (Ref 4). It gives a fine wettability, high strength in addition to better resistance to thermal fatigue and creep (Ref 5, 6, 7). The development of new lead free solders at least with the same mechanical properties in addition to microstructures stability as eutectic Pb-Sn solder is an urgent errand. In response to this, many candidate Pb-free solder alloys as Sn-Ag, Sn-Zn, Sn-Sb, Sn-Bi, and Sn-Cu been studied (Ref 8, 9, 10, 11). In general, lead free solder may be arranged into three temperature groups. These days, more consideration tends to research in to solder in the moderate temperature range like; Sn-Ag-Cu, Sn-Zn, Sn-Cu and Sn-Ag alloys (Ref 12, 13, 14). Sn-Ag alloys have been proposed as the good promising alternative for Sn-Pb solders (Ref 15). In the electronics industry, as the packing size decreased as well as service -environment required increase .It is necessary to search and provide new higher mechanical lead free solders.

To improve the effect of solder joints reliability, a group of ,Sn-Cu, Sn-Ag and Sn-Ag-Cu solders alloys doped with added substances was developed. Rare earth elements [REE] may significantly enhanced the properties of metal .Even all the rare earth element have fundamentally the same physical-chemical properties. REE are surface active element which has a vital part in metallurgy of materials like wettability and refinement microstructure of solder (Ref 16,17,18,19) .It is common knowledge that microstructure change will influence

the mechanical property, particularly in solder materials so RE doping can cause a change in mechanical property in solder alloys (Ref 20,21,22,23). With refined microstructure from RE doping make the distance for dislocations to pile up making the doped solders have longer creep rupture time, better creep resistance and higher strength in tension with less elongation (Ref 24). Adding a little amount of rare earth element refines the microstructure in addition to IMC particles as well as makes the thickness of the interfacial IML thicker (Ref 25, 26). Terbium is member of the rare earth lanthanide group. It is a soft silver-gray metal, malleable, ductile. It is stable relatively in air compared to the earlier reactive lanthanides in the first half of the lanthanide series. It is found that literature survey revealed that lack studies on lead-free Sn-3.5Ag solder joints containing small trace of Tb rare earth element. So, the present work is devoted for investigating the effect of addition of trace amount of Tb rare earth element on microstructure, thermal, electrical and mechanical properties of Sn-3.5 wt. % Ag lead free solder for trying to improve its microstructure physical properties.

2. Experimental procedures

2.1 Materials

Six solders alloys of compositions Sn 96.5-x Ag 3.5 Tb x (x=0, 0.1, 0.2, 0.3, 0.4, 0.5) were formed by using a single copper roller melt-spinning technique discussed elsewhere (Ref 27, 28). The metals used with required quantities of were weighed out and melted in a porcelain crucible. After the alloys melted the melt was completely agitated to have a homogenization effect. At 350°C melt temperature the casting was done in air. The copper wheel speed fixed at 2900 rpm; which agree with a linear speed of 30.4 m/s. By differential scanning calorimetry (DSC) which has a heating rate 10 K/min, the melting temperature of prepared alloys was obtained. The dynamic young's modulus and internal friction of melt-spun alloys was calculated by a dynamic resonance method. The Vickers hardness measurements were examined by using a Vickers micro hardness tester. More than 15 indents to every value were made on every sample to determine any hardness variation caused by having more phases, with one phase soft, ductile and harder phase.

2.2 Characterization

The JEOL JSM-6510LV low vacuum scanning electron microscope (SEM) has a higher performance SEM for fast characterization and fine structures imaging (high resolution of 3.0 nm at 30 kV). It provides observation of specimens up to 150 mm in diameter. Identification of the as quenched melt-spun alloys was obtained by a Shimadzu x-ray diffractometer (Dx-30) (XRD). A Cu - K α radiation with wavelength, $\lambda = 1.54056 \text{ \AA}$ at 4.5KV and 35mA with N Filter in the angular range 2θ ranging from 100° to 800° in continuous mode, which involves the strongest diffraction signals. When Bragg law is used to measure the interplanar spacing d' , since $2 d' \sin \theta = n \lambda$

3. Results and discussion

3.1 Crystal Structures & Lattice Parameters

Lattice parameter data and crystal structure of stable phases observed in the Sn-3.5 Ag system are given in Table 1. Fig. 1 displays the patterns of x-ray diffraction for the melt-spun alloys. The pattern of the eutectic Sn-3.5Ag shows two phases, a tetragonal β -Sn matrix and orthorhombic Ag₃Sn. For the alloys including terbium, the results indicate changes in main matrix microstructure (unit cell volume, lattice parameter and crystal size) and same phases appeared but with increasing the number of IMC phase. It is well known that the β -Sn tetragonal crystal has two axes a and c and when the ratio of c/a rich to unity, a tetragonal cell becomes cubic. And therefore if the cubic cell is distorted along one axis, then it changes to tetragonal, its symmetry decreases, and forms more diffraction lines. The number of lines increases due to the new plane spacing introduction, resulted from non-uniform distortion. So the positions of all atoms found in the unit cell and specification of the lattice parameters is enough for characterize all essential aspects of a crystal structure. The

lattice parameters a , c and the axial variation ratio (c/a) are calculated for β -Sn matrix with the variation of Tb compositions and listed in the table 1. The lattice particle size is estimated from x-ray pattern by using Scherer's equation where $Dhkl=0.891\lambda/\beta hkl\cos\theta$. Lattice distortion $\langle \Sigma \rangle$ is calculated by $B = (1/Deff) + 5 \langle \Sigma \rangle > \frac{1}{2} \sin\theta / \lambda Deff$ where B is full width high maximum (FWHM), $Deff$ is the particle size, λ is the wave length of Cu $K\alpha=1.54056\text{\AA}$ and θ is the reflection angle and listed in table 2. The results display the lowest value of particle size was noticed for Sn-3.5Ag-0.5Tb prepared alloy is 199 nm and display the maximum axial ratio c/a variation compared with other compositions.

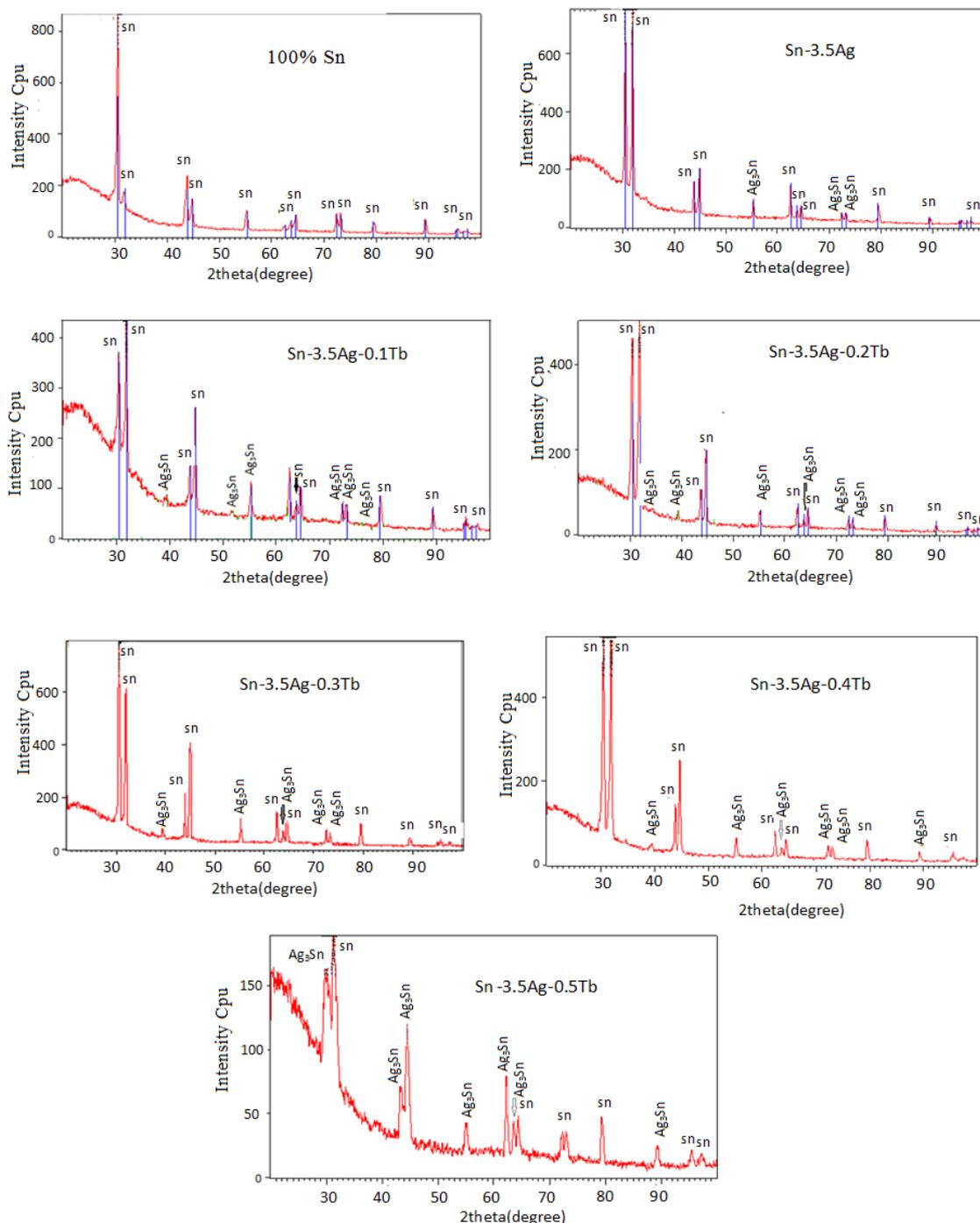


Fig. 1: The x-ray diffraction patterns of all Sn-3.5Ag-Tb lead free solders

Table 1: Lattice and cell parameters of Sn phase for Sn-3.5Ag-Tbx (x = 0.0, 0.1, 0.3, 0.4 and 0.5 by wt. %) alloys.

Lead Free Solders Wt. %	Lattice parameters			Cell Volume (\AA^3)	Number of atoms per unit cell
	a(\AA)	c(\AA)	c/a		
100Sn	5.83	3.183	0.545	108.18	3.98
Sn-3.5Ag	5.832	3.19	0.546	109	3.54
Sn-3.5Ag-0.1Tb	5.837	3.197	0.5477	108.9	3.9
Sn-3.5Ag-0.2Tb	5.85	3.183	0.544	108.9	4
Sn-3.5Ag-0.3Tb	5.841	3.1842	0.545	108.6	4.09
Sn-3.5Ag-0.4Tb	5.8494	3.182	0.54	108	3.68
Sn-3.5Ag-0.5Tb	5.8	3.3	0.568	111	4.5

Table 2: Particle size and Lattice distortion for Sn-3.5Ag-Tbx (x = 0.0, 0.1, 0.2, 0.4, and 0.5 by wt. %) alloys.

Lead free solder Wt.%	phase designation	Phase no.	crystal system	crystal size (nm)	Lattice Distortion
100Sn	β -Sn	13	Tetragonal	364.296	1.1E-03
Sn-3.5Ag	β -Sn	10	Tetragonal	369	1.32E-03
	Ag ₃ Sn	3	Orthorhombic	317.45	1.18E-03
Sn-3.5Ag-0.1Tb	β -Sn	11	Tetragonal	328	1.63E-03
	Ag ₃ Sn	6	Orthorhombic	507.465	1.27E-03
Sn-3.5Ag-0.2Tb	β -Sn	9	Tetragonal	249.478	2.07E-03
	Ag ₃ Sn	5	Orthorhombic	295.28	1.42E-03
Sn-3.5Ag-0.3Tb	β -Sn	10	Tetragonal	426	1.2E-03
	Ag ₃ Sn	5	Orthorhombic	326	1.16E-03
Sn-3.5Ag-0.4Tb	β -Sn	8	Tetragonal	346	1.63E-03
	Ag ₃ Sn	6	Orthorhombic	326	1.52E-03
Sn-3.5Ag-0.5Tb	β -Sn	6	Tetragonal	222	2.02E-03
	Ag ₃ Sn	7	Orthorhombic	199	2.34E-03

3.2 Microstructures characterization change with the addition of rare earth, Tb.

A scanning electron microscope (SEM) was used for examining the studied alloys microstructure. A typical microstructure of eutectic Sn-3.5Ag lead-free solder consists of a two mixture phases; a Sn-rich as dark contrast and eutectic structure which consists of β -Sn and Ag₃Sn as bright contrast phase, as noted in Fig.2b. After added small amount from rare earth Tb to binary eutectic alloy, the ternary Sn-3.5Ag-Tb microstructure composed of fine and uniformly particles of intermetallic compound Ag₃Sn distributed in β -Sn matrix as noted in Fig.2d-g. The finer particles were identified as being Ag₃Sn IMC according to XRD analysis. The volume fraction of every phase is related to the compositions of alloys. The morphology of the IMC particles is superfine as indicated in structure. This effect could be attributed to the rapidly solidified and high cooling rate during the production of solders. It is found that rapid cooling during solidification processing by melt spun technique produces a finer microstructure as compared conventional methods Fig.2c-g. The

microstructural features of a eutectic solder, like distribution, volume fraction, and spacing of every phase, give the physical and mechanical properties.

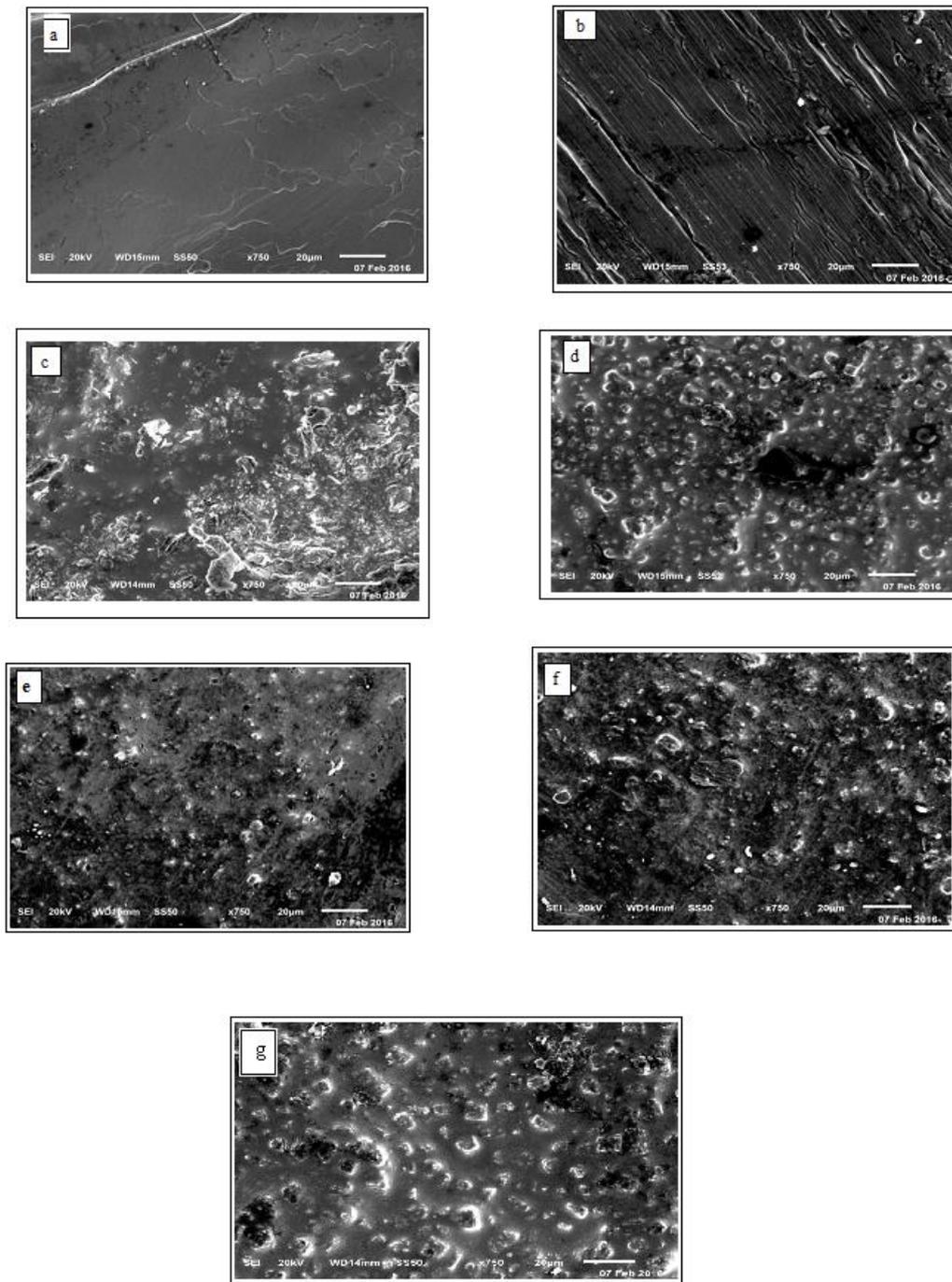


Fig. 2 SEM for (a)Sn , (b) $\text{Sn}_{96.5}\text{-Ag}_{3.5}$, (c) $\text{Sn}_{96.4}\text{-Ag}_{3.5}\text{-Tb}_{0.1}$, (d) $\text{Sn}_{96.3}\text{-Ag}_{3.5}\text{-Tb}_{0.2}$, (e) $\text{Sn}_{96.2}\text{-Ag}_{3.5}\text{-Tb}_{0.3}$, (f) $\text{Sn}_{96.1}\text{-Ag}_{3.5}\text{-Tb}_{0.4}$ and (g) $\text{Sn}_{96}\text{-Ag}_{3.5}\text{-Tb}_{0.5}$.

3.3 Thermal behavior

By using the differential scanning calorimetry (DSC) the solder alloys melting temperatures is determined. The melting temperature (T_m) is considered an important physical property of solder. The DSC curves for the eutectic and ternary solder alloys based on heating at scanning rate of $10\text{ }^\circ\text{C}/\text{min}$ are shown in Fig.3. In the DSC curves, the melting point is typically calculated by first selecting the most steeping part of the low temperature side of well of the heat absorption, obtaining the slope, then extrapolating the slope line to the temperature at the zero differential heat flow axis. Tb substitution for Sn by weight in the Sn-3.5Ag eutectic reduced the melting temperature by few degrees. This decreasing is caused by the decrease of the crystalline size. This reducing is great because it is frequently not possible to reduce melting points of eutectic with ternary additions by $10\text{ }^\circ\text{C}$ without making the phase equilibria complicating. The pasty is defined as the temperature difference between solidus and liquidus temperatures. In this temperature range the solder is a semi solid in addition to it gives different properties from the liquid or solid phases which during solidification lead to the solder left- off For the eutectic alloy Sn96.5-3.5Ag, the pasty range is equal to zero while the other alloys shows a positively increase up to 11.2°C as shown in table 3. Using a solder with narrow pasty rang leads to avoid this problem. The results show, the lowest pasty range was achieved at 0.3wt % addition.

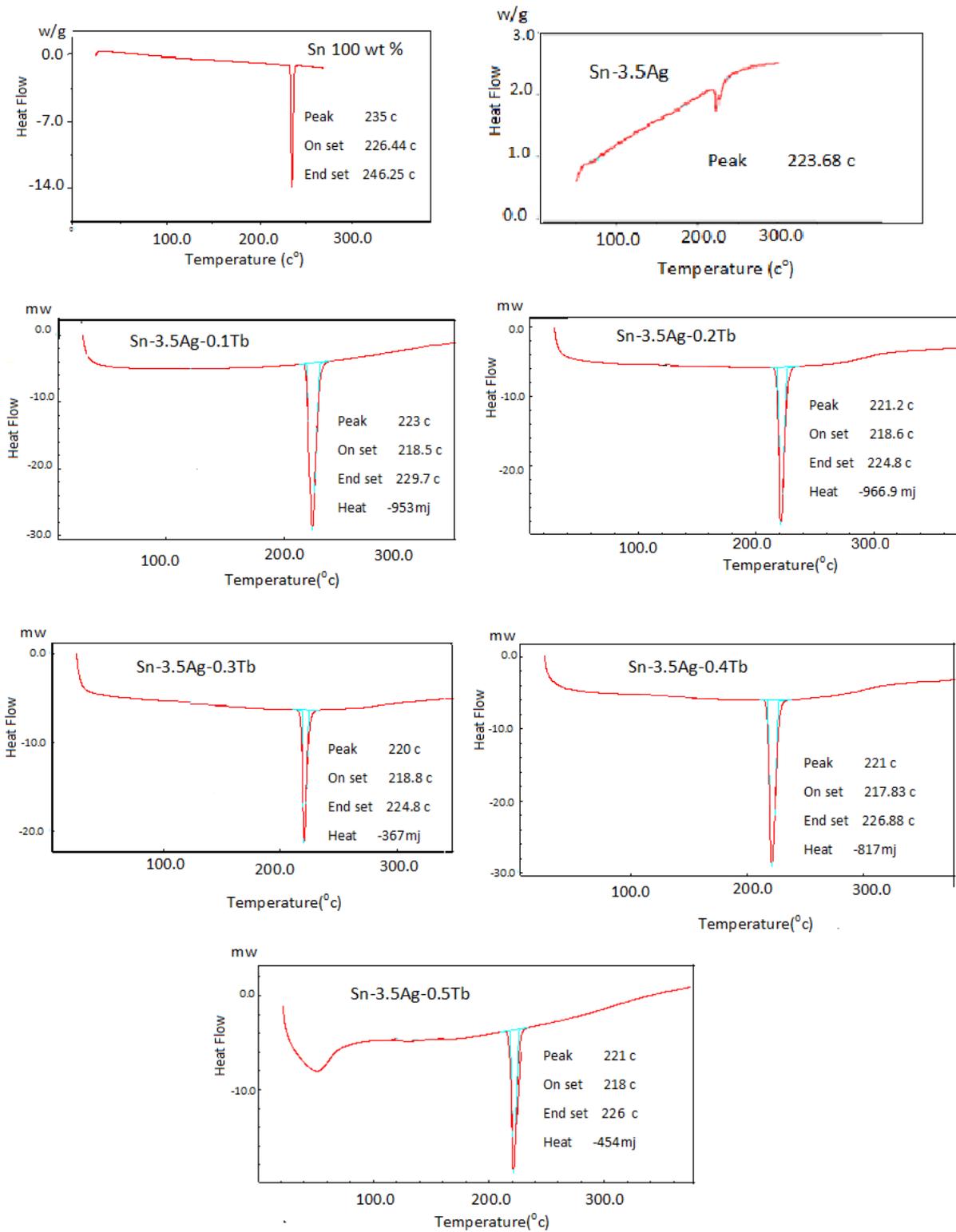


Fig.3 Differential scanning calorimetry (DSC) curves for lead free solder alloys

Table 3: Thermal properties of Sn-3.5Ag-Tb lead free solder

Lead free solder wt.%	Solidus temperature T_s ($^{\circ}\text{C}$)	Liquids temperature T_l ($^{\circ}\text{C}$)	Pasty range ($^{\circ}\text{C}$)	melting point T_m ($^{\circ}\text{C}$)
Sn	226.44	242.25	15.81	235
Sn-3.5Ag	222	228	6	221.68
Sn-3.5Ag- 0.1Tb	218.50	229.70	11.20	223.70
Sn-3.5Ag- 0.2Tb	218.62	226.57	07.95	221.22
Sn-3.5Ag- 0.3Tb	218.80	224.80	06.00	220.20
Sn-3.5Ag- 0.4Tb	217.83	226.88	09.05	221.06
Sn-3.5Ag- 0.5Tb	218.40	226.10	07.70	221.30

3.4 Mechanical testing

3.4.1 Effect of Tb on Young's modulus

The manufacture of modern electronic assemblies is depending on materials characterized by a broad range of thermal expansion coefficients. Normally heat generates within the electronic device operation. When these materials are matched together, during operation the physical restrictions produce internal thermal stresses and strains. The formation of intermetallic compound Ag₃Sn may be responsible for the mechanical properties of solder joints. Increases the Tb contents above 0.1 wt. % caused increase mechanical properties (Young's modulus). The material strength is related to the forces of atomic bonding that are reflected by the elastic modulus macroscopically. The influences of addition of Tb on elastic moduli were investigated in Table 4. The results show that the dynamic elastic modulus reached to the maximum value of 82 MPa at 0.5 wt.% Tb approximately 100 % so that the elastic modulus of Sn-3.5Ag-0.5Tb became two times as large as Sn-3.5Ag as shown in fig.4. This is may be because solid solution hardening effect and refining the micro structure of Ag₃Sn IMC were identified by XRD that act as hard inclusions in main matrix Sn. But for 0.3Tb it be noticed that size of β -Sn particles were estimated from the XRD and found that the Ag₃Sn particle size was increased from about 369 nm for un-doped alloy to about 426 nm for 0.3Tb doped alloy. The increased particles and grain increase the distance for dislocations to pile up, consequently the doped alloy has low elastic modulus. Also according to XRD it is found that 0, 4 Tb was added to melt tend to make formation of Ag₃Sn intermetallic compound easier. Hence, increase the intermetallic compound phases, it led to increasing the elastic modulus of the Sn-Ag melt-spun solder alloys.

Also, the results show that the internal friction has a sensitivity to the ratio of the added Tb component as one of the alloying elements to the binary Sn-3.5Ag as listed in Table 4. In Fig.5 the internal friction Q^{-1} changed with the content of Tb is shown by a reduction in transformable shear stress with biggest free volume. Values of shear modulus (G) and bulk modulus (B) were determined using slandered equation $G=E/2(1+\nu)$ and $B=E/3(1-2\nu)$. It is shown here that when Pb-free solder alloys such that Sn-3.5Ag is doped with a small amount of RE elements, the mechanical properties of them are enhanced. The internal fraction and thermal diffusivity of alloys have been calculated by the resonance curves as shown in Fig.6 and listed in the Table 4.

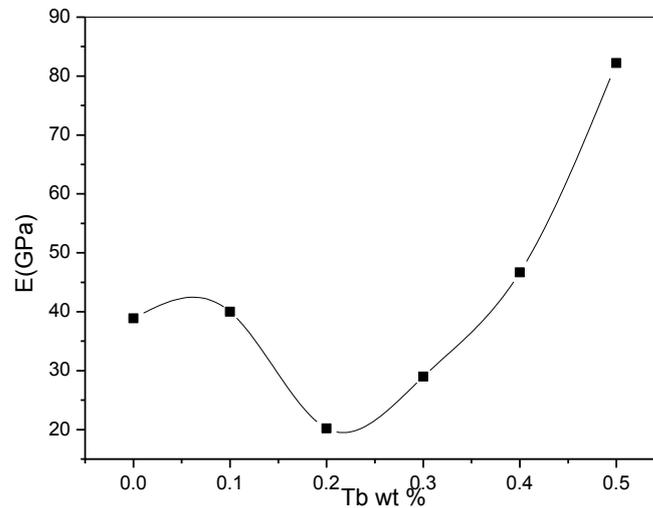


Fig 4: Young Modulus Variations with Tb addition for prepared lead free solder alloys

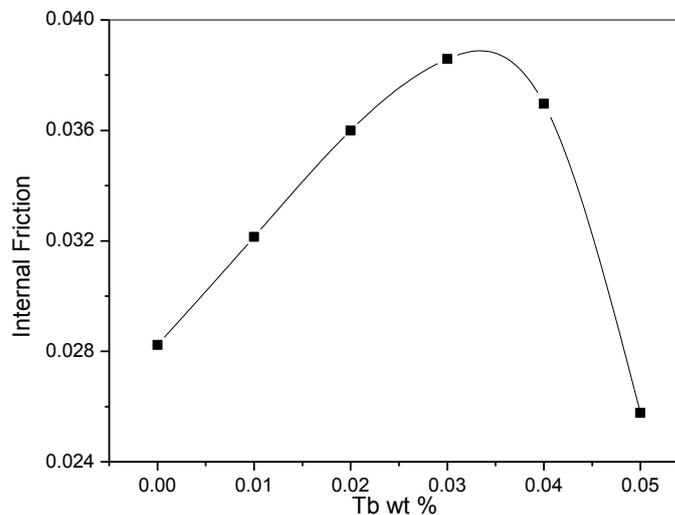


Fig 5: Internal Friction of Sn-3.5Ag Lead Free Solder Alloys

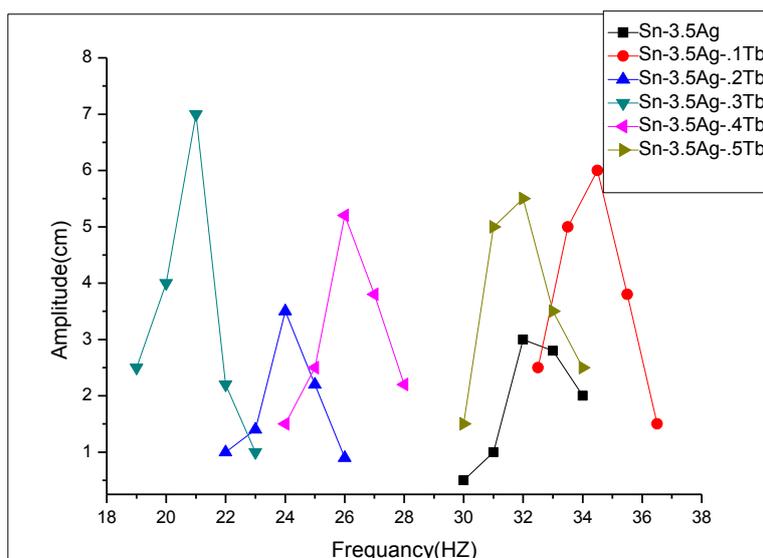


Fig 6: Resonance curves for lead free solder alloys

Table 4: Internal friction (Q^{-1}) values and dynamic Young's modulus of prepared alloys

Lead free solder wt. %	E GPa	G GPa	B GPa	Q^{-1}
Sn	37.4	13.8	43.6	.0252
Sn-3.5Ag	38.9	14	45	0.023
Sn-3.5Ag- 0.1Tb	40	14	46.7	0.032
Sn-3.5Ag- 0.2Tb	20.2	7.4	23.6	0.036
Sn-3.5Ag- 0.3Tb	29	10.7	33.8	0.038
Sn-3.5Ag- 0.4Tb	46.7	17	54.4	0.037
Sn-3.5Ag- 0.5Tb	82.2	30.3	95.7	0.025

3.4.2 Effect of Tb on micro-hardness measurements

The hardness measurement, especially, Vickers microhardness, gives a good indication of the mechanical properties of materials. The material hardness is often balanced with its resistance to attrition or wear and is a characteristic of practical interest since it calculated the material durability during use and the suitability particular applications of the material depend on it. The configuration of grains and the motion of dislocations and growth have an effect on the micro-hardness of a solder alloy. This process is more matched to the processing temperature, the microstructure, and the composition. By using an indentation hardness test the

resistance to plastic indentation was calculated, by a standard load a small hard indenter is pressed into the surface and the indentation size resulted from it is obtained. Fig. 7 displays the influence of Tb content on Vickers hardness Hv at 10 gf applied load and indentation time 5 s. For Sn-3.5Ag, Hv has the minimum value 149.6 MPa. After 0.2wt.% level Tb addition the Hv increases up to 308.9 MPa approximately 100% became two times eutectic Sn-3.5Ag composition as shown in Table 5. The micro-hardness value of Sn-3.5Ag solders increased with terbium content as a result of both dispersion strengthening and solid solution strengthening besides the increasing in intermetallic compound Ag₃Sn after rare earth addition as identified by XRD and refinement the grain size. Also, this conduct is illustrated in terms of the alloy structural properties. As therefor, the relatively high percent of hard Ag₃Sn phase is almost present.

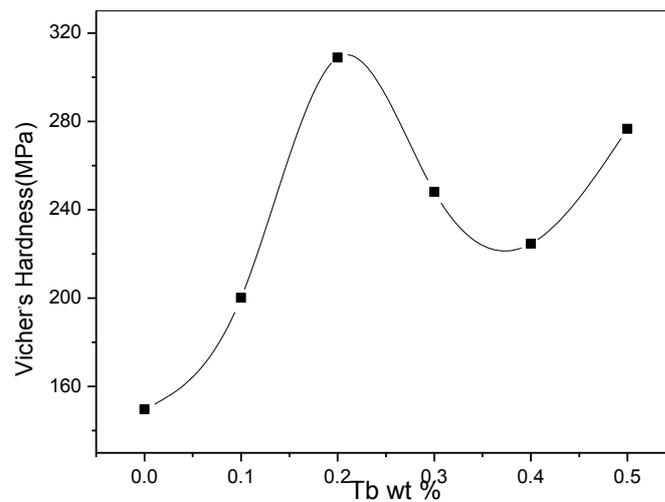


Fig 7: Vichers Hardness of Sn-3.5Ag Lead Free Solder alloys

Table 5: Vickers hardness of Sn-3.5Ag-Tb lead free solder alloys

Lead free solder wt. %	Vickers micro-hardness MPa
Sn100	90.22
Sn-3.5Ag	149.6
Sn-3.5Ag- 0.1Tb	200.1
Sn-3.5Ag- 0.2Tb	308.9
Sn-3.5Ag- 0.3Tb	248.1
Sn-3.5Ag- 0.4Tb	224.6
Sn-3.5Ag- 0.5Tb	276.6

3.5 The wettability of Pb-free solder doped with RE, Tb.

One of the famous ways to measure wettability of surface of material is contact angle Fig. 8 shows the contact angles for the prepared alloys formed at the solder substrate's flux triple point on Cu substrate in air. The results display that the wettability is enhanced by adding trace amount of rare earth Tb and the lowest contact angle was obtained when Tb content is 0.2. The RE Tb can segregate on liquid solder surface because of its reliability and surface activity. This segregation could lower the surface tension of the solder and makes the wettability of the solder much better. As much as rare earth elements are liable to be oxidizing during soldering forming slag. So with increasing of Tb addition led to lessen the solderability of solder.

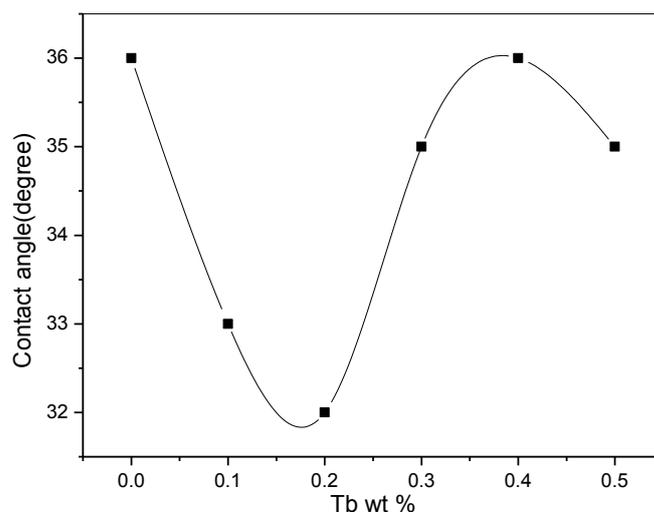


fig.8: Contact angles of Sn-3.5Ag-Tb lead free solder alloys

3.6 Electrical resistivity of Pb-free solder doped with rare earth, Tb.

In microelectronic devices assemblies the solder joint has a great effect on electrical interconnection. Therefore the solder interconnect resistivity in most microelectronic should be very thin that it does not have an effect on the circuit functionality. Fig.9 displays the electrical resistivity of prepared alloys at room temperature. The increasing in resistivity may be the result of the crystalline defects. The presence of crystalline defects is because of the formation of Sn-Ag intermetallic compound that increased with Tb addition and works as scattering center for conduction electrons. Also, the small grain (crystallite) size of the alloy that obtained with Tb addition, the large number matrix, which may exist, can probably cause anomalous increase in the resistivity [29].

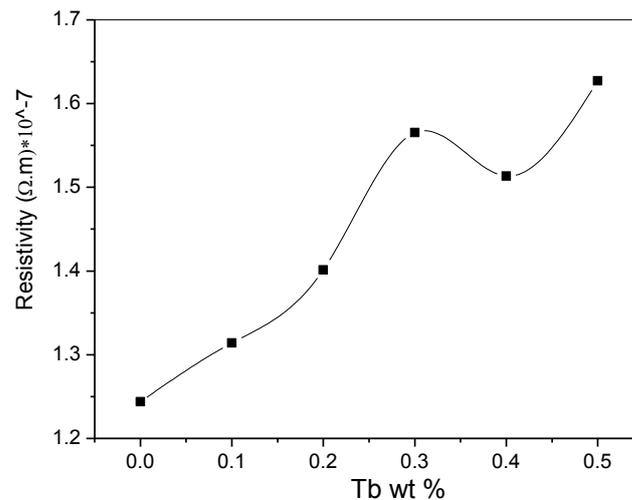


fig.9: Electrical resistivity of Sn-3.5Ag-Tb lead free solder alloys

Conclusions

In general, adding trace amount of RE element plays an important role on the microstructure, mechanical, thermal and electrical of Sn-Ag solder has investigated. The results show that, the micro-structure of Sn-3.5Ag solder become finer and uniform by adding Tb that may be due to the surface activity of REE. RE doping can also cause mechanical property change in solder alloys. With refined microstructure from RE doping, solder show improved elastic modulus and Vickers hardness that was evident in 0.5Tb addition .while at 0.3Tb It be noticed that size of β -Sn particle size was increased Causing an increase in the distance for dislocations to pile up, consequently the doped alloy has low elastic modulus. Increasing the intermetallic compound phases led to improve the elastic modulus of the Sn-3.5Ag-0.4Tb melt-spun solder alloys slightly. At the same time the rare earth addition has small effect on the melting temperature. Also, at different addition of REE, Tb, the electrical resistivity values have little increasing that may be as a result of forming of Ag_3Sn IMC which acts as scattering centers for conduction electrons.

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