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Abstract During last few decades, emerging environmental regulations worldwide, more notably in Europe and Japan, have targeted the elimination of Pb usage in electronic assemblies due to the inherent toxicity of this element. This situation drives to the replacement of the Sn-Pb solder alloy of eutectic composition commonly used as joining material to suitable lead-free solders for microelectronic assembly. Sn-based alloys containing Ag, Cu, Bi, and Zn are potential lead-free solders, usually close to the binary or ternary eutectic composition. For this reason a great effort was directed to establish reliable thermophysical data fundamental to interpret the solidification process and fluidity of alloys belonging to these systems. In this work, an analysis of the solidification process of pure Sn, binary Sn-Ag, Sn-Cu, Sn-Bi, Sn-Zn, Sn-Pb and ternary Sn-Ag-Cu eutectic alloys was carried out using computer aided-cooling curve analysis and differential scanning calorimetry.

1 Introduction

In the modern electronics industry, soldering is the most important technique of connecting the electronic devices and substrates. As a joining material, solder provides electrical contacts and facilitates thermal paths for heat dissipation, as well as mechanical integrity in electronic

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assemblies [1-3]. Sn–Pb based alloys are the most popular solders. In the last years the microelectronics industry has been moving away from Lead-containing solder alloys due to the harmful effects of Lead on environment and human health combined with the strict legislation (the so-called WEEE and RoHS directives) on banning the use of Leadbased solders [4, 5]. Simultaneously, there is a growing need for solders that can be used for applications with more demanding service conditions such as automotive or aerospace. For these purposes Lead Free Solders (LFS) alloys have been developed during the past decade to replace classic Sn-Pb eutectic solders [6]. A basic requirement for a candidate solder alloy to replace a Pbcontaining solder is to have a specific freezing range compatible with existing equipment and components. For these reasons, the ideal solder alloy would be eutectic or near-eutectic, with a liquidus temperature low enough to avoid damaging components and a solidus temperature high enough to maintain joint reliability during thermomechanical fatigue. There are several candidates LFS Snbased alloys such as Sn-Ag-Cu alloys (SAC alloys), containing Zn or Bi, which have considerably attracted attention especially for automotive, industrial and electronic applications. Two of the main benefits of this system are its low melting temperature compared with the Sn-Ag binary eutectic alloy and its distinguished wetting behavior without loosing strength. Also, the SAC solder-joint electrical properties appear to be attractive for both surfacemount and ball-grid-array assembly methods. Thus, Sn-Ag-Cu is considered one of the most favorable system as a Lead-free standard alloy replacement [7]. As a part of the process of developing new LFS alloys, thermodynamic calculations have been extensively used [8-11] and a number of thermodynamic databases have been developed specifically for this purpose [8, 9]. However, it is well

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Table 1 List of used alloys		
Alloy	Alloy chemical composition (wt.%)	
#1:Sn	Sn	
#2:SnCu	Sn-0.7 % Cu	
#3:SnAg	Sn-3.5 % Ag	
#4:SnAgCu	Sn-3.5 % Ag-0.9 % Cu	
#5:SnZn	Sn-9 % Zn	
#6:SnPb	Sn-37 % Pb	
#7:SnBi	Sn-57 % Bi	

Table 1 List of used allows

known that a purely thermodynamic approach does not provide direct information on general material properties, such as, thermophysical and mechanical properties that are the key to the application of new solder alloys. Reliable knowledge of such material properties is essential for the interpretation of alloy solidification and numerical modeling methods for the casting and soldering process. Most of the thermophysical properties such as density, thermal conductivity, specific heat, solidus and liquidus temperatures, latent heat of fusion and viscosity are affected by changes in the chemical composition of the alloy. For some materials, such as pure metals, steels, Aluminum and Copper alloys and some superalloys, this data is available in handbooks or in the open literature. For LFS alloys, there are several initiatives to obtain alloy property databases [12-15], but even, there is still a lack of experimental data for these alloys, e.g. on latent heat of fusion.

Several thermal analysis techniques can be used to measure the required thermophysical properties. Melting, solidus and liquidus temperatures, solid fraction, latent heat and microstructural evolution of multicomponent alloys can be determined from computer aided-cooling curve analysis (CA-CCA) and this technique was used to investigate the development of microstructure during solidification of casting alloys [16–23], Fe and Al alloys [24, 25]. Differential Scanning Calorimetry (DSC) is useful for determining energy associated with transformation such as the latent heat of fusion and specific heat of the alloy in smaller samples.

In this study computer aided-cooling curve analysis was used to follow the solidification path of LFS alloys, as a method to obtain controlled equiaxed solidification of small samples of Sn-Ag-Cu, Sn-Zn and Sn-Bi eutectic alloys, so as pure Sn and Sn-Pb eutectic alloy for comparison purposes. This schema let us determine solidus and liquidus temperatures, the range of solidification and the amount of undercooling associated with the solidification. Differential Scanning Calorimetry was also used to determine latent heat of fusion of these alloys. Thermal properties found would be useful to interpretated the fluidity behavior L_f which depends on mould-metal heat transfer



Fig. 1 Temperature-time cooling curves obtained for different eutectic alloys of the electronic Sn family

coefficient, solidification mode, latent heat of solidification of the alloy and the relative quantity of each phase present during solidification of LFS alloys [26-28].

2 Experimental

The alloys used in this study were obtained starting from 99.99 % purity Sn, Ag, Cu, Zn, Bi and Pb elements. These were melted in a SiC crucible coated internally by ceramic protective coating, and heated in an electric resistance furnace under argon gas flow, cast in a graphite mold and cooled in air. The chemical compositions of the studied alloys have been extracted in Table 1.

Cooling curves were carried out in an electric furnace, with an appropriated electronic controller. Chamber and sample temperatures were taken by using K type thermocouples wired to a NI-USB 9211A acquisition data interface, connected to a personal computer, as suggested in literature [29]. The curves were taken at different cooling rates, in the range of 1–10 °C/min. Figure 1 shows typical Temperature-time cooling curves from the alloys studied in this work, and Fig. 2 shows the derivative of some of the taken cooling curves, useful to evaluate the phase fraction and the heat evolution. From these curves it is possible to stablish the temperature of solidification which has been referenced as T_S during the text.

The evolution of heat during the fusion and solidification processes was determined using a Differential Scanning Calorimeter DSC Rheometric Scientific, model SP at several scan velocities. A sapphire sample was used as reference. The melting temperature T_M of the alloys was taken during the heating of the samples. Although it is known that DSC is not the preferred method to determine processes temperatures, these values were taken for calibration and comparison purposes. Both T_S as T_M are



Fig. 2 Derivative of cooling curves corresponding to the solidification of a pure Sn, b Sn–Cu, c Sn–Ag and d Sn–Ag–Cu eutectic composition

Table 2 Characteristic T_S and T_M

Alloy	$T_S \pm 0.5$ (°C)	$T_M \pm 0.5$ (°C)	T_M^E (°C)
#1:Sn	230.4	230.9	231.9
#2:SnCu	224.6	227	226.8
#3:SnAg	218.4	220.7	220.2
#4:SnAgCu	214.6	217.2	216.9
#5:SnZn	197.2	198.3	198.5
#6:SnPb	177.7	183	182.2
#7:SnBi	134.7	138	138.8

included in Table 2. Figures 3 and 4 show typical thermograms obtained during the solidification whilst Fig. 5 shows the theoretical enthalpy change during the melting or solidification of the alloys.

Standard polishing procedures were used for metallographic preparation of the samples, using alumina grinding papers up to 0000 grit. Final polishing was achieved using micropolish with a 2 μ m Al₂O₃ slurry. The samples were observed using Optical (OM) and Scanning Electron Microscopy (SEM). The phases were identified using Energy Dispersive Spectroscopy (EDS). For Scanning electron microscopy observations all samples were etched with 2 % HCL–methanol. Examples of



Fig. 3 Calorimetric DSC curves corresponding to the melting of Sn solder family alloys obtained at $\alpha = 10$ °C/min

Temp (°C)



Fig. 4 Heat evolution as function of time during the eutectic ternary melting of Sn-3.5 % Ag-0.9 % Cu alloy. The experimental curve seems to be formed by two different components, a ternary and a binary eutectic reactions

the microstructures observed are shown in Figs. 6, 7, 8, 9, 10 and 11.

3 Results and discussion

The samples were taken from the melting of each alloy, cast in small ceramic containers to proceed with the cooling curves under controlled solidification conditions. Figure 1 shows the curves obtained for the different studied alloys. The typical aspect of Temperature-time behavior could be observed in this Figure. The cooling curve can be interpretated as a balance between the evolution of heat in the sample and the heat flux away from the sample. Then,

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Fig. 5 Enthalpy variation during the phases change, calculated under equilibrium conditions



Fig. 6 Binary Sn-3.5 % Ag in \mathbf{a} optical, and \mathbf{b} scanning electron micrographs

the start of solidification is easily detected due to the release of latent heat associated with the liquid-solid transformation. Some other variations on the curve can be



Fig. 7 Binary Sn-0.7 % Cu alloy, \boldsymbol{a} optical, and \boldsymbol{b} Scanning electron micrographs

related with other processes, reactions and, of course, the end of solidification[30]. In analytical form

$$\frac{dQ}{dt} = m \left[f C_P^S + (1-f) C_P^L \right] \frac{dT}{dt} + m \Delta H_f \frac{df}{dt}$$
(1)

where *m* is the mass of the sample, $C_P^{S,L}$ are the heat capacity in solid and liquid respectively, (dT/dt) is the cooling rate of the process, ΔH_f is the latent heat of solidification and *f* is the fraction of formed solid. Also, Eq. (1) can be written as

$$\frac{dQ}{dt} = m \left[f C_P^S + (1-f)C_P^L + \Delta H_f \frac{df}{dT} \right] \frac{dT}{dt}$$
(2)

where (dQ/dt) is constant and represents the heat flow imposed externally to the system.

After that, (df/dt) or (df/dT) can be evaluated from (1) and (2). In the general, $C_p^S \neq C_p^L$. However it is usual to consider that $C_p^S = C_p^L$ for calculation purposes, in which case the previous equations can be simplified as

$$\frac{df}{dt} = \frac{1}{m\Delta H} \left[\frac{dQ}{dt} - mC_P \frac{dT}{dt} \right]$$
(3)

which can be easily graphically evaluated. Although this approximation is not strictly true, it does not carry an important loss of precision in the zone where solidification takes place.

In the cases where more than one reaction is expected all previous equations must be modified to include the observed behavior, but note that these different contributions would be in the form of the last term of equation (2) to reflect changes in the (dT/dt) curve.

For some of the studied alloys including pure Sn an initial undercooling at the start of the nucleation was observed followed by a jump to reach the solidification temperature T_s . This shift reachs up to approximately 5 °C, for pure Sn, binary Sn-Cu, Sn-Ag and ternary Sn-Ag-Cu eutectic alloys. It could be assumed that the undercooling before the equilibrium state is reached due to metastable undercooled liquid. As the rate of nucleation depends on the difference of temperature deviates from the equilibrium temperature, it is better detected in the derivative (dT/dt)curve due to the latent heat released during the first part of the process [29], as it can be seen in Fig. 2 for ternary eutectic Sn-Ag-Cu, and pure Sn. It could be interesting to remark that the classical binary Sn-Pb, Sn-Zn and Sn-Bi eutectic alloys does not show this particular behavior during the solidification of small samples. In these cases, a lower value than the equilibrium temperature was obtained, and the posterior recalescence was absent. After this initial undercooling an isotherme was reached, corresponding to the eutectic reaction, binary or ternary or pure Sn depending the case. Even when these temperatures were not obtained under equilibrium conditions, they were considered as the fusion temperature T_s for each studied system as reference and comparison. Actually the equilibrium temperatures are well determined by different authors and appear in several databases [8, 13, 31, 32]. As it can be noted, the greater of these temperature values correspond to pure Sn, then binary Sn-Cu and Sn-Ag, ternary Sn-Ag-Cu, Sn-Zn and Sn-Pb eutectic. The lower temperature is obtained for binary Sn-Bi eutectic, even lower than traditionally used Sn-Pb. In Table 1 are resumed these results.

A differential scanning calorimeter device (DSC) was used to determine the evolution of heat during the melting and solidification. Under this schema, the measured evolution of heat is a balance between the heating (or cooling) of the sample and the reaction flowing heat. During the melting and solidification, this corresponds to the latent heat, being the surface enclosed below the heat curve in the interval of interest:

$$\Delta Q = \frac{1}{\alpha} \int_{T_i}^{T_f} \left(\frac{dQ}{dT} - g(T) \right) dT$$
(4)



Fig. 8 Ternary Sn-3.5 % Ag-0.9 % Cu eutectic, \mathbf{a} optical, and \mathbf{b} scanning electron micrographs

Table 3 Latent heat of fusion

Alloy	Latent heat $\Delta H_m \pm 0.05 \text{ (J/g)}$	Latent heat (J/g) [8, 13, 31–33]
#1:Sn	53.9	59.2
#2:SnCu	56.3	62.3
#3:SnAg	56.1	63
#4:SnAgCu	55.1	66.9
#5:SnZn	56.2	65.4
#6:SnPb	52.1	43.68
#7:SnBi	42.4	49.1

In the last equation α is the scanning velocity expressed in (K/s), (dQ/dT) is the heat evolution and g(T) is a baseline corresponding to the characteristic bias that appears in absence of transformation and could be affected by the specific heat of the sample and different heat capacity between sample and reference. Usually this function is approximated by a right line.



Fig. 9 Binary Sn-9 % Zn eutectic alloy, a optical, and b scanning electron micrographs

Figure 3 shows the curves of heat flow against T obtained for different LFS alloys, pure Sn and eutectic Sn-Pb in function of temperature during the melting. Associated latent heat can be calculated as the integral of the heat flow curve for each studied alloy. The curves were taken using scanning velocities of 2, 5 and 10 °C/min, founding no deviations with the scanning velocities as it was expected. Obtained values of latent heat of fusion can be seen in Table 3. In the Figure the scanning velocity was established at 10 °C/min. In all cases, the calorimetry signal forms a well-defined peak due to the delivered latent heat. By normalizing the data with the scanning velocity and the sample masses, the integral over the baseline is the total heat delivered during the solidification or melting. As it can be seen the behavior is similar for all the LFS Sn-based alloys, Sn-Pb and pure Sn, varying only in the temperature of solidification. Eutectic Sn-Pb shows higher and thinner peak, although the surface below the curve and so the latent heat is lower than the values of LFS alloys as it can be seen in Table 3.

Reference values of melting temperatures and heat of fusion are shown in Fig. 5, where the heat evolution is

represented in function of temperature. The calculus is performed under strict equilibrium conditions by using Thermocalc software package and lead-free specific database [8, 31, 33].

Figure 4 shows the heat evolution as a function of reference temperature, during the solid-liquid transformation of ternary Sn-Ag-Cu eutectic as an example. As it can be seen, the figure is not a single gaussian. The complete transformation could be interpreted like formed by two different heat developments. This situation can be accomplished if (1) the composition does not corresponds exactly to the ternary eutectic, or (2) the solidification starts with a primary phase close, but out of equilibrium, driving the composition of remnant liquid shifts out the ternary eutectic composition. Figure 4 also shows a temporal deconvolution of the calorimetric curve, normalized by mass and scan velocity. It can be seen that the resultant signal could be formed by at least two normal components. Taken the start of each distribution as the beginning of the phase change, two eutectic reactions were detected: the fundamental signal is due to the ternary eutectic and after that a binary eutectic. Under this frame the melting starts with the dissolution of the ternary eutectic and followed by the binary eutectic, which could be $(Sn) + Ag_3Sn$ or $(Sn) + Cu_6Sn_5$ depending on the composition of remnant liquid. In this example, the sample starts to melt approximately at 217 °C and the second process starts at 221 °C. Also, from the relative area between the components is clear that the quantity of ternary eutectic is greater than the binary eutectic which is expected from the composition used.

The solidification path could be interpretated through the microstructure observation, using optical (OM) and scanning electron microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) in all cases. Typical Optical and Scanning electron Micrographs of the as-solidified solder eutectic alloys are shown in Figs. 6, 7, 8, 9, 10 and 11, corresponding to the specimens before the calorimetric tests. Figures 6a and 7a exhibit optical micrographs of Sn-3.5 % Ag and Sn-0.7 % Cu binary eutectic alloys, while Figs. 6b and 7b are scanning electron micrographs. The binary eutectic microstructure either in Sn-3.5 % Ag or Sn-0.7 % Cu is a mixture of the intermetallic particles, Ag₃Sn or Cu₆Sn₅, in the matrix of the β -Sn phase. Both consist of two regions, i.e, white regions (primary Sn phase) surrounded by a dark network. These alloys exhibit a facetednonfaceted morphology corresponding to irregular eutectics, where the nonfaceted phase is Sn and the faceted phase is Ag₃Sn or Cu₆Sn₅ respectively.

Figure 8 shows similarly in (a) optical and (b) scanning electron micrographs of the ternary Sn–Ag–Cu eutectic alloy. Different phases could be identified by energy dispersion spectrography as: β (Sn), binary [β (Sn) + Ag₃Sn]

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Fig. 10 Binary eutectic Sn-57 % Bi **a** optical, and **b** scanning electron images showing colony type microstructure surrounding a two phase fine spacing zone

eutectic and $[\beta(Sn) + Ag_3Sn + Cu_6Sn_5]$ ternary eutectic. A great quantity of a rich-Sn primary phase [Sn(92.45%) - Ag(6%) - Cu(1.49%)] composition can be detected, with Ag-rich and Cu-rich conglomerates (of [Sn(98.65%) - Ag(0.97%) - Cu(0.43%)] and binary Sn–Ag eutectic. The presence of a phase which crystallizes with faceted morphology and the fact that the primary phase tends to present a higher undercooling at the beginning of solidification, results in a coarse dendritic phase surrounded by the faceted binaries phases. This is a well known phenomenon, even with an eutectic composition, called asymmetric coupled growth.

Figure 9 shows (a) optical and (b) scanning electron micrographs of and binary Sn-9 % Zn eutectic alloy. The structure corresponds to an irregular eutectic, which is formed by a Sn-rich matrix and Sn–Zn needles, as it can be seen in the micrographs.

Figure 10 shows (a) optical and (b) scanning electron micrographs, the typical microstructure of Sn-57 % Bi. It is a regular globular structure, exhibits a nonfaceted-



Fig. 11 Binary Sn-37 % Pb eutectic alloy, \boldsymbol{a} optical, and \boldsymbol{b} scanning electron images

nonfaceted morphology, consisting on a mixture of Bi-rich crystals in an eutectic matrix composed by Sn (light phase) and Bi (dark phase). This is a lamellar regular micro-structure like Sn-37 % Pb.

Figure 11 shows (a) optical and (b) scanning electron micrographs of traditionally used Sn-37 % Pb. The microstructure consists on a mixture of fine Sn-rich (light regions) and Pb-rich (dark area) solid solutions forming a lamellar regular eutectic microstructure.

4 Conclusions

In the present study, an analysis of the thermal behavior of Sn–Ag, Sn–Cu, ternary Sn–Ag–Cu, Sn–Zn and Sn–Bi eutectic alloys were carried out by using Computer assisted-cooling curve analysis (CA-CCA) and Differential Scanning Calorimetry (DSC), fundamental techniques to the interpretation of solidification and fluidity behavior of

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electronic joining Sn-based family alloys. Pure Sn and traditionally used Sn-Pb were also included to comparison purposes. The results can be reviewed as follows: The cooling curves of pure Sn and Lead-free Sn alloys show a recalescence up to 5 °C in some cases, corresponding to the initial supercooling. This behavior is absent in Sn-Pb eutectic alloy. The melting and solidification characteristic temperatures were determined experimentally. The greater corresponds to pure Sn, and then Sn-Cu, Sn-Ag and ternary Sn-Ag-Cu for SAC alloys. As Zn and Bi are suggested as modifiers of SAC alloys, also were studied. Only melting temperature of Sn-Bi is lower than Sn-Pb eutectic. Despite the composition is close to the ternary eutectic, was possible to detect in many cases the presence of the binary eutectic in small quantities. This behavior could be attributed to a change in the liquid composition when the solidification of the ternary eutectic reaction starts. Solidus and liquidus temperatures determined from cooling curves, were found to be in good agreement with the Differential Scanning Calorimetric measurements.

The determined properties would be useful to interpretate the fluidity behavior which depends, among other factors, with the solidification way the thermal properties of the alloy and the relative quantity of each phase present during solidification of the alloys.

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