

Solidification and wetting behaviour of SnAgCu solder alloyed by reactive metal organic flux

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Abstract

Purpose – The purpose of this paper is to develop a new alloying method for solders by using a metal organic modified flux in solder pastes.

Design/methodology/approach – This paper presents the impact of six metal organic compounds (Co, Fe, Al; stearate, oxalate, citrate) on the melting and solidification behaviour in comparison to the revealed microstructure.

Findings – It could be shown that Co and Al influence the supercooling whereas Fe exhibits no effect. Co reduces the supercooling of the cast of about 10 K and affects the nucleation. Al retards the solidification up to 185°C. Doping of the solder by flux containing metal organic compounds is successful and the alloying elements Co and Fe are found in the microstructure.

Research limitations/implications – This paper provides a starting-point for the new alloying method – so far only fluxes for solder pastes have been investigated.

Originality/value – The reactive alloying method enables the use of new alloying elements for solder pastes in unmodified soldering processes.

Keywords Metals, Solder, Alloys, Flux, Organic compounds, Solidification

Paper type Research paper

Introduction

The shift towards lead-free solders was triggered by environmental regulations, e.g. the Waste from Electrical and Electronic Equipment and the Restriction of Hazardous Substances (European Union, 2008) Directives. Hence, lead-free solders, mostly near eutectic SnAgCu (SAC), are widely used. At the moment, the majority tends towards hypoeutectic SAC with 3.0 wt%-Ag and 0.5 wt%-Cu (SAC 305), a trend mainly due to the increasing price of silver (London Metal Exchange, 2008, www.lme.co.uk/historical_data.asp) and decreasing materials brittleness. Furthermore, a loss in thermal fatigue properties using hypoeutectic SAC can be neglected (McCormick *et al.*, 2006; Syed *et al.*, 2006). Looking on the processing of solder alloys, it is possible to estimate a slight shift for the liquidus point from 217 to 220°C from the phase diagram (Moon *et al.*, 2000).

If one compares SAC with a common eutectic Sn-Pb solder there are different mechanical, physical and chemical properties which are directly linked in terms of reliability. It is well known that the creep resistance of SAC is higher than that of eutectic Sn-Pb (Choi *et al.*, 2000). Typical damage during thermal

cycling is caused by mechanisms of creep of the solder material (Lee *et al.*, 2000). It can be concluded for packages designed for higher lifetime that a high creep resistance of a solder alloy yields better performance and higher lifetime of solder joints (Schubert *et al.*, 2003). Nevertheless, technical requirements (e.g. operation temperature) in automotive electronics increase continuously (Mango, 2005). Employed solder alloys should have a high creep resistance at elevated temperatures and should not be susceptible to coarsening. Coarsening changes the microstructure and thereby the mechanical properties of the solder significantly (Allen *et al.*, 2004). Hence, many investigations have been carried out in order to analyse the thermal fatigue and creep properties of lead-free solders (Henderson *et al.*, 2004; Andersson *et al.*, 2005; Liang *et al.*, 1996; Barry *et al.*, 2007).

An established approach for improving the characteristics of an alloy is to use an additional dopant. These dopants can, for instance, strengthen the solder matrix by refining the β -tin dendrites, increase the fraction of the eutectic domain, or

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segregate as pro-eutectic phases. All three possibilities are common strategies (Anderson and Harringa, 2004). Many elements like rare earth (Wu *et al.*, 2002), Al (Swenson, 2007; Lin *et al.*, 2002), Zn (Song *et al.*, 2006), Co (Gao *et al.*, 2006; Amagai, 2008; Andersson *et al.*, 2006) and Fe (Kim *et al.*, 2003; Andersson *et al.*, 2006; de Sousa *et al.*, 2006) have already been investigated as solder dopants. Many of the elements mentioned are advantageous for lead-free solder alloys but this paper focuses only on three elements: Co, Fe and Al. Co is reported to give a higher ductility in drop tests (Syed *et al.*, 2006), a higher nucleation rate (Anderson and Harringa, 2004) and a changed growth of the intermetallic compound between the circuit board metallisation and the solder. Fe increases the shear strength (Anderson, 2007), reduces the Kirkendall voids (de Sousa *et al.*, 2006) and forms precipitations (Kim *et al.*, 2003). Al as dopant shows CuAl₂ precipitations after ageing, reveals a softening of the alloy (Huang *et al.*, 2007) and changes solidification behaviour (Lin *et al.*, 2002). All of these alloys have been prepared from bulk materials and were mostly used as solder performs or sometimes as solder wires.

However, the increased amount of surface mounted devices (SMDs) needs solder paste for manufacturing and the major drawback of these new alloys is the powder production for the solder paste. To produce solder powder out of the alloy three methods are commonly used: gas, spinning disc or ultrasonic atomisation (Bartels *et al.*, 2006). Solder powder requirements (constant composition, etc.) are difficult to realise due to the very high cast temperatures which are necessary to create a homogeneous melt. Furthermore, some of the powders are prone to severe oxidation, reducing the solderability. Additionally, it cannot be guaranteed that evolving intermetallic phases inside the solder balls are completely dissolved again during the short time of the reflow process.

For this reason, it was necessary to investigate a new method to alloy lead-free solders, with the aim of using them as solder pastes. Two types of ideas were thought of: on the one hand there are reaction solders, where two metals melt together during the reflow process (Nowotnick *et al.*, 2008). However, this process uses powders again and therefore was discarded. On the other hand, reactive alloying of solders by using organic and inorganic substances was shown in the literature (Wittke *et al.*, 2001).

Consequently, an *in situ* alloying of a standard solder powder by a chemical reactive flux containing Co, Fe and Al was investigated. After proving a successful doping (Zerrer *et al.*, 2008), this paper focuses on the melting and solidification behaviour of the new solder. Differential scanning calorimetry (DSC) experiments were carried out to investigate single and multiple reflow and soldering *in situ* on a copper pads. The results are accomplished by microstructural analyses. The aim of the study was to investigate the effect of the dopant, its reproducibility and to look for the behaviour under boundary conditions.

Experimental

Experimental solder paste was manufactured by W.C. Heraeus. First, a doped flux was prepared by mixing around 2,000 ppm of metal organic compound (aluminium stearate, aluminium acetate, cobalt oxalate, cobalt stearate, iron citrate or iron stearate) to a commercially available, halide containing

flux (F 610 Heraeus). Then, the new flux, later on called nanoFlux, was mixed with a solder powder with the composition Sn-96.5 wt per cent, Ag-3.0 wt per cent, Cu-0.5 wt per cent. The result of the chemical analysis for the base alloy is shown in Table I. The nanoFlux solder pastes obtained had a metal content of 89 wt per cent.

These solder pastes were analysed by DSC. For this purpose, 25 mg solder paste was placed in a sealable DSC crucible containing an ambient atmosphere. For all experiments the heating ramp was 20 K/min, whereas the cooling rate was 10 K/min. Three different sets of experiments were carried out:

- 1 For evaluating the temperature effect, two maximum temperatures, either 240 or 300°C, were chosen.
- 2 Multiple reflows on printed circuit boards (PCBs) were emulated by a second re-melting. Hence, some samples were simply measured twice, whereas cleaning of the PCB or storage before the second reflow were investigated on cleaned and re-embedded solder samples. The cleaning was done with chloroform in an ultra sonic bath.
- 3 A special setup to analyse the reaction behaviour of the solder paste with a copper pad was used, shown in Figure 1.

Therefore, a copper pad (Cu 99.99 per cent) was cleaned in a cleaning agent (Rohm and Haas) and slightly etched in a solution of sodium dioxysulphate and sulphuric acid. To reduce the dissolution of Cu into the solder, the maximum temperature was set to 230°C for these tests.

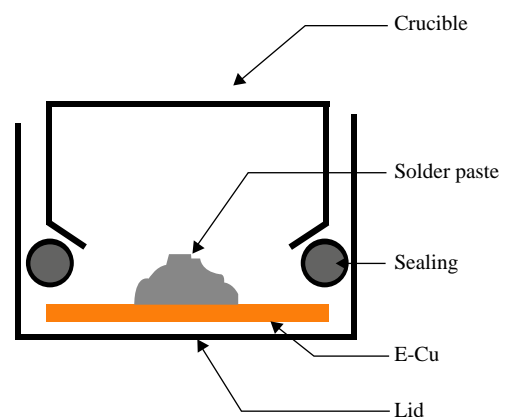
Before evaluating the results, the crucibles were X-rayed to ensure a sole solder ball inside. Additionally, the crucibles were opened, cleaned and the solder balls were embedded in epoxy and subsequently ground and polished. Metallographical analyses were carried out using optical microscopy (OM) and scanning electron microscopy (SEM), the latter one equipped with an energy dispersive X-ray detector (EDX) and a wavelength dispersive X-ray unit (WDX).

Table I Stoichiometry of the solder powder analysed by inductively coupled plasma with optical emission spectrometry

Sn	Ag	Cu	Al	Co	Fe
96.5%	2.8%	0.48%	< 0.001%	< 0.001%	0.003%
± 0.1	± 0.1	± 0.02	± 0.001	± 0.001	± 0.002

Note: Only the important elements are stated

Figure 1 Special setup for *in situ* soldering



Results

In the following the impact of the metal organic compounds on the melting and the solidification behaviour of the solder alloys is presented. Successful doping was presented in a previous publication (Zerrer *et al.*, 2008), the amount of transferred dopants in weight per cent is shown in Table II.

1. Different maximum process temperatures

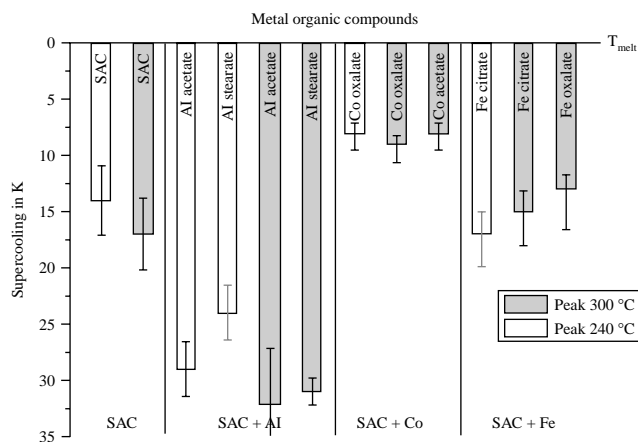
No substantial difference between the solder pastes was found during heating. The melting of the solder alloys occurred at around 220°C. During cooling, however, significant effects could be observed for the Co and Al containing solder pastes, as can be seen in Figure 2. Both, Co oxalate and Co acetate decrease the supercooling of the molten solder. Interestingly, these alloys showed the smallest standard deviation and solidify at around 211°C. Furthermore, a distinctive double peak is shown in Figure 3 giving the measurement signals for four different solder pastes. In comparison to all other pastes the solder alloys obtained from Al containing pastes had an extended liquid range. The Fe containing solder pastes behaved similar to the reference alloy, except for a slightly changed peak shape shown in Figure 3.

Figure 2 shows the influence of a change in the maximum reflow temperature on different solder pastes. The influence of the metal organic compound can be clearly seen for the

Table II Amount of the dopant in the solder alloy after remelting, given in weight per cent determined by inductively coupled plasma optical emission spectroscopy

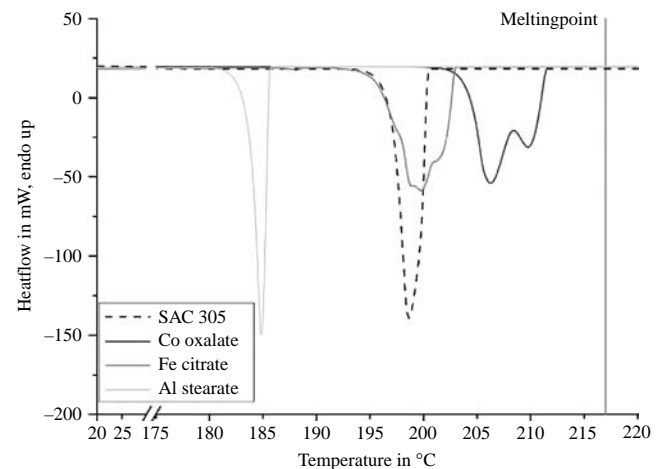
Metal organic	Amount of dopant (wt%)
Al acetate	Al < 0.001
Al stearate	Al < 0.001
Co oxalate	Co 0.12
Co acetate	Co 0.10
Fe citrate	Fe 0.02
Fe oxalate	Fe 0.05

Figure 2 Influence of the Al and Co containing metal organic compounds



Notes: Al retards the solidification whereas Co supports the nucleation. Fe seems to have no significant influence. Additionally, no influence on the peak temperature was observed

Figure 3 Measured solidification signals for four different solder pastes



Notes: For the Co containing paste a distinctive double peak can be observed and an increased liquidus range is visible for Al stearate. Co has a higher solidification temperature whereas Al retards the solidification. The peak for Co is spread into two peaks

Al and Co containing metal organic compound. Al retards the solidification whereas Co supports the nucleation. Fe seems to have no significant influence. Within the uncertainty of the experiments, the lower temperature of 240°C provided the same results. The only exception was the Al stearate solder paste; its supercooling was 8 K lower compared to the reflow experiments at 300°C.

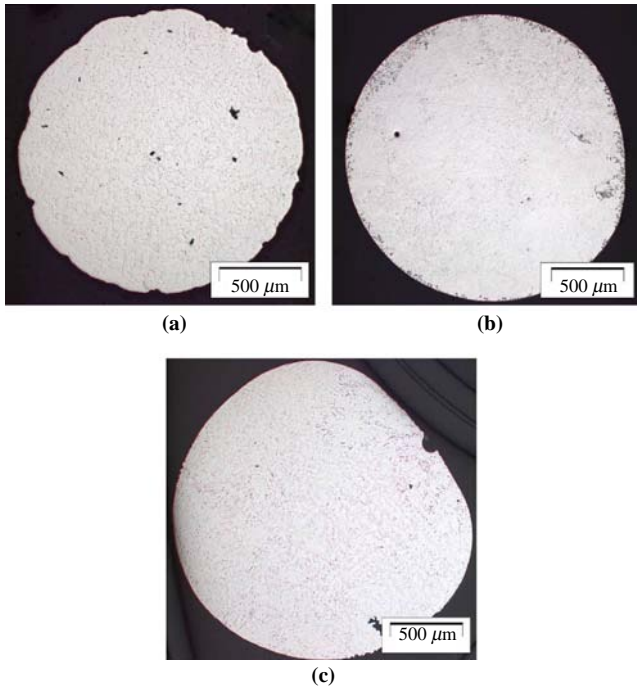
The images in Figure 4 were taken by an optical microscope and show CoSn_2 phases for the sample processed at 300°C, whereas the sample reflowed at 240°C only showed finely dispersed $(\text{Cu}, \text{Co})_6\text{Sn}_5$ phases determined by WDX analyses. The CoSn_2 phases are mostly found in the outer area of the solder balls. Typical CoSn_2 phases are shown in Figure 5(a) with their plate-like structure. Microstructural analyses on Fe solder balls showed that small Fe_xSn_y phases were present, as can be seen in Figure 5(b). However, these phases are so small that the stoichiometry could not be estimated by our measurement equipment. Nevertheless, assuming an equilibrium and according to the phase diagram (Massalski, 1990), the stoichiometry should be FeSn_2 .

2. Influences of cleaning procedures

To understand how the solder and the flux behave in a second reflow, as required for double sided SMD PCBs, some samples were re-melted once more. Figure 6 shows the results of these experiments. It is obvious that the first reflow had a higher uncertainty than the second reflow, shown by the larger error bars on the baseline. This is due to the melting behaviour of a paste containing flux compared to a single solder ball. Furthermore, only the Al samples changed significantly during the second solidification, but no change in the microstructure can be observed.

3. In situ solder joint formation and wetting behaviour

Reflowing the solder paste on Cu pads was possible without major difficulties for the experimental setup. The supercooling of the different melts are shown in Figure 7. Typically, the supercooling was around 18 K. Two remarkable results were found. Co had still an earlier solidification at

Figure 4 Solder balls obtained from different solder pastes and reflow times at 240°C

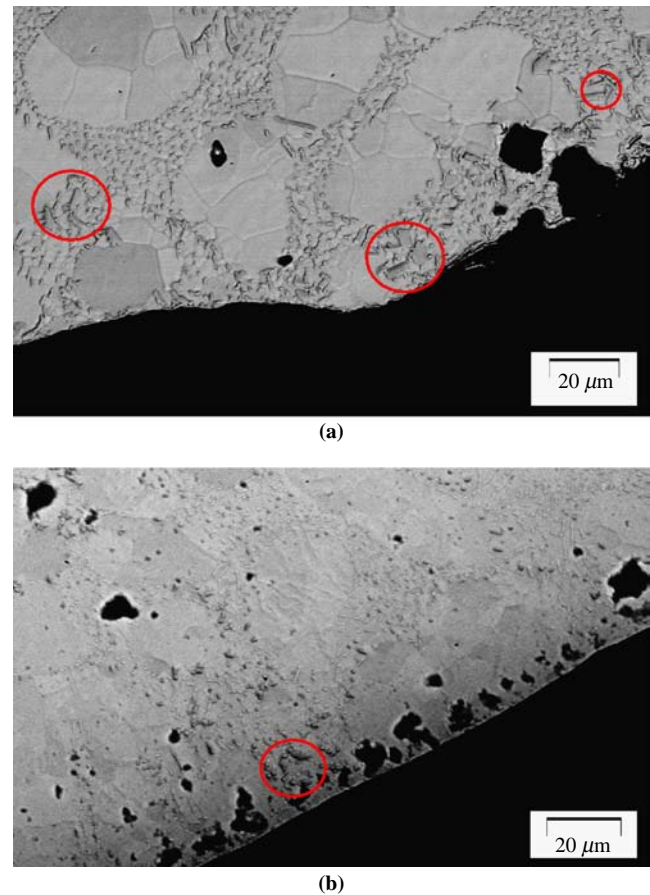
Notes: (a) Co containing solder paste; 3 min above liquidus line; (b) same solder paste around 30 min above liquidus; (c) reference solder paste (similar after 3 and 30 min)

$211 \pm 0.5^\circ\text{C}$ which can directly be compared to the experiments carried out solely with solder paste. In contrast, the Al metal organic compounds lost their effects on supercooling when the melt was forming a solder joint.

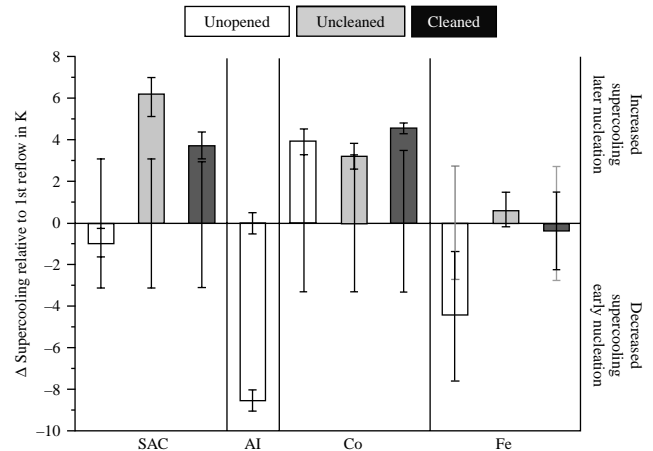
To analyse the wetting behaviour the soldered copper pads were rinsed in chloroform to remove the resin residues at first. Images of the samples are shown in Figure 8(a)-(c). The wetting areas of the samples were determined by OM and shown in Figure 9. The activity of the basic solder paste should be the same. However, it was observed that the wetting area was affected by the Al containing solder pastes. The stearate showed an increased wetting behaviour in contrast to the Al acetate which had a reduced wetting area. Cross sections had been prepared for all samples and the solid contact angles were measured. The difference between the wetting angles of the reference solder and the joints formed by Al acetate containing solder paste is shown in Figure 10. On some Fe containing samples some dewetting could be observed. All measured wetting angles are given in the bottom part of Figure 9; as a general rule, the lower the angle, the better the wetting. Both, the contact angle and the wetting area test give the same results.

Discussion

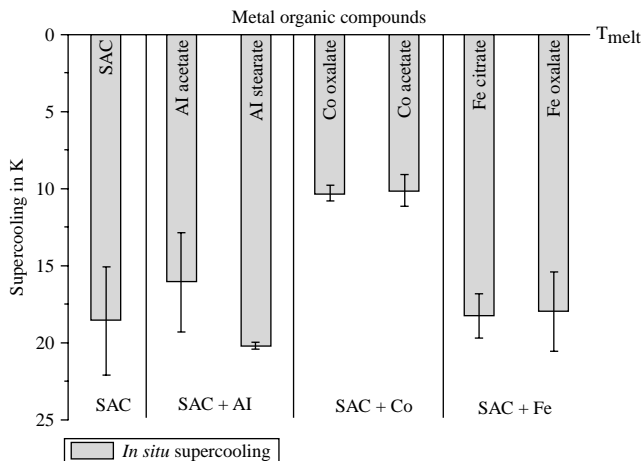
The decreased supercooling for Co-containing solders has already been published by other authors (de Sousa *et al.*, 2006) and whose results are in good agreement with ours. This provided us with evidence that the doping of the solders was successful, even at the level of a single DSC solder ball. Furthermore, the element was found with EDX and WDX

Figure 5

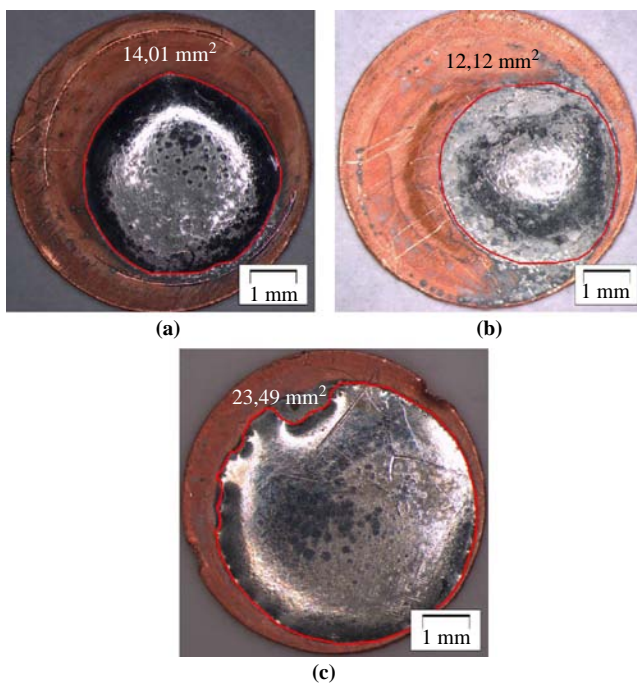
Notes: (a) SEM image of Co containing solders showing CoSn_2 phases; (b) FeSn_2 in an Fe containing solder

Figure 6 The influence of the second reflow process on the supercooling for DSC experiments after different treatments of the sample before the second remelt

Notes: Unopened – crucible remains closed; uncleaned – the solder ball was transferred into a new crucible; cleaned – the solder ball was rinsed in chloroform before being transferred into a new crucible. Fe, Co and SAC have the similar solidification temperature in the second reflow. However, Al has a decreased supercooling with an earlier nucleation

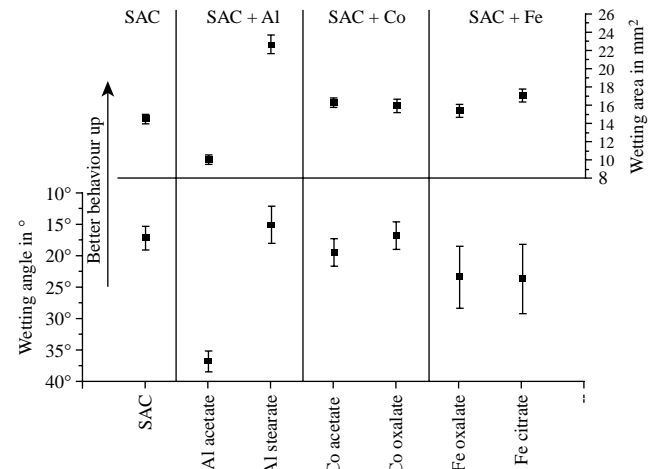
Figure 7 Supercooling during *in situ* experiments

Notes: No further difference between Al and the SAC reference is observed. However, Co still shows the same behaviour than in simple paste reflow

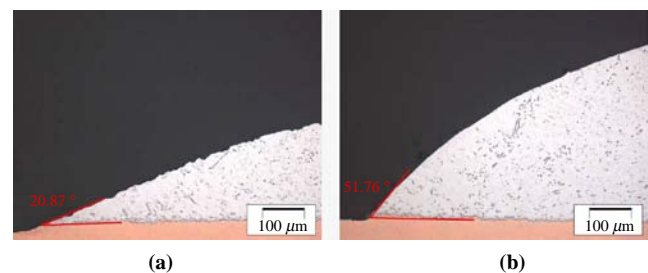
Figure 8

Notes: (a) Wetting area of the SAC 305 reference solder paste; (b) decreased wetting area of Al acetate containing solder paste; (c) increased spreading of Al stearate containing solder paste

and an effect on the microstructure was visible for Co and Fe shown in Figure 5(a)-(b). The explanation for the big CoSn_2 phases in the samples prepared at 300°C can be a higher amount of transferred dopant reaching the limit of solubility, allowing the formation of CoSn_2 , especially in the outer area of the solder ball where cobalt can still diffuse into the solder. In the range of our doping (0.1-0.2 wt per cent), nucleation was always affected. Hence, at our doping levels the effect is independent of the amount of dopant. Probably, the CoSn_2 phases caused the double peak in Figure 3 by a changed

Figure 9 Wetting area and the wetting angle, the higher the dot the better the behaviour

Note: Except for the Al-containing fluxes the wetting is only slightly affected, by the metal organic compounds

Figure 10

Notes: (a) Micrograph of *in situ*DSC sample obtained from SAC 305 solder paste; (b) micrograph of *in situ* DSC sample with Al acetate solder paste showing a much larger contact angle

solidification path, leading to a different formation sequence of the intermetallic phases. Moreover, the cleaning procedures did not show an effect on solidification, proving that the element and not the flux itself is responsible for the effect on the supercooling. When looking at the *in situ* experiments, the solidification was promoted, too. This explains the refined solder joints resented on PCBs (Zerrer *et al.*, 2008) which is promising for a higher reliability (Song *et al.*, 2006). Also, the wetting behaviour is not affected by Co, as is shown in Figure 4.

The Fe metal organic compounds seem to have no effect on the solidification behaviour of the solders obtained, even though Fe was alloyed to the solder as can be seen in Table II. In the solder balls small FeSn_2 phases are present. These phases are very small and seem to have no influence on the other microstructure. In the literature (de Sousa *et al.*, 2006), an effect of Fe on the supercooling was found. However, this effect was achieved by measuring pre-alloyed samples which contained large FeSn phases, whereas such large phases were absent in our solder balls. Latest experiments show that FeSn_2 phases are mostly present as sub-micrometer particles (Hutter *et al.*, 2009). Multiple reflows did not change the behaviour. Therefore, the solder can be called stable under the test conditions chosen. If one compares the wetting area, Fe samples were similar to the reference spreading area. If one

has a look on the wetting angles, the error is increased due to the fact that in some cases the micrograph hit a region of dewetting. This dewetting is probably caused by oxidation and formation of Fe_xO_3 from the flux.

Aluminium containing samples behave completely differently from those mentioned above. No noteworthy transfer of Al into the solder was found. As a result the Al has still to be in the flux residues. However, the metal organic compounds of Al had a major impact on the supercooling and the spreading area. Especially, interesting was the different behaviour of the latter one. For Al stearate, the wetting is enhanced possibly due to an interaction between the stearate and the impurities. This effect is comparable to a flux and makes Al stearate a promising fluxing agent (Patent, 2008), whereas the Al acetate is a very unstable compound for storage in solder pastes. It easily forms Al_2O_3 which is very likely to be the reason for the bad wetting behaviour. Although these are possible mechanisms, the type of reactions for the Al compounds remain unclear at the moment. Unfortunately, the effect on the supercooling could only be observed in the first reflow cycle; a second cycle causes the solidification temperature to rise to a level close to the reference. Therefore, the effect cannot be explained by forming a new alloy with a lower melting point. This is an indicator for a complex interaction of the metal organic compound and the melt, not the element itself.

Conclusions

Using a modified flux, the melting behaviour of all solder pastes was not influenced and therefore these solder pastes can be processed in a standard process with the boundary conditions between 230 and 300°C. Even though the solidification temperature was influenced, processing is still possible. Hence, in the manufacturing lines, all four versions of the solder pastes could be used as a drop-in alternative for SAC in respect to their thermal properties.

The doping of the solder by the flux was successfully shown for Co and Fe. This is in good agreement with the chemical analyses already published. However, the reaction mechanisms cannot be determined from our current experiments. Co and Fe containing solder pastes are promising for improving the microstructure, since they are forming CoSn_2 , refined $(\text{Cu},\text{Co})_6\text{Sn}_5$ or FeSn_2 phases. In addition, the solidification in Co is promoted and therefore it can be beneficial for a refined grain structure in a solder joint. To clarify the behaviour of the microstructure under different loading conditions such as ageing, creep and thermal shock, further analyses have to be done.

Both the Al stearate and the Al acetate are not suitable for doping. However, taking into account the effect of Al stearate on the supercooling and the wetting, Al stearate can be a promising part of a new halide and resin free fluxing agent.

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