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The microstructural, mechanical and electrical properties of pb-sn and lead-free sc0.7 solders containing sub micron active carbon particles

Mikron altı aktif karbon parçacıkları içeren pbsn ve kurşunsuz sc0.7 lehimlerin mikroyapısal, mekanik ve elektriksel özellikleri

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The Microstructural, Mechanical and Electrical Properties of Pb-Sn and Lead-Free SC0.7 Solders Containing Sub Micron Active Carbon Particles

Highlights

- * The added active carbon particles lowered the resistance of lead free solders but not in Pb-Sn solder
- The additions of active carbon particles contributed to increased yield strength properties
- * Active carbon particle did not affect the melting points of both solders
- The hardness of both solders with active carbon additions increased the most at 0.045 wt% addition.

Graphical Abstract

In this study, different level additions of active carbon particles has been added to find out the optimum addition based on the best mechanical and electrical properties using SC 0.7 lead free solder and Pb-Sn solder.



Figure. SEM images of Pb-Sn solders with active sub micron carbon added a) 0.003, b) 0.015, c) 0.03 and d) 0.045 wt%.

Aim

The aim of the study is to reveal the effect of various sized active carbon particles on the physical and mechanical properties of active carbon particle in Pb-Sn and lead SC 0.7 free solders.

Design & Methodology

Solders containing 0.003 wt%, 0.015 wt%, 0.03 wt% and 0.045 wt% active carbon particles were prepared with lead-free (SC0.7, Tin-Cu 0.7) and Pb-Sn solders (63/37). Cu stripes were soldered using produced solders and mechanical physical and microscopical studies were carried out.

Originality

In this study, four different active carbon additions into the lead free SC 0.7 and Pb-Sn solders have been studied with extensive range of characterization techniques.

Findings

Active carbon additions positively affected the hardness and yield strength for both types of solders but negatively affected the electrical resistance for Pb-Sn solders.

Conclusion

The addition of active carbon particles hasmostly positive effects on the properties of both lead free and Pb-Sn solders.

Declaration of Ethical Standards

The author(s) of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

The Microstructural, Mechanical and Electrical Properties of Pb-Sn and Lead-Free SC0.7 Solders Containing Sub Micron Active Carbon Particles

Araştırma Makalesi/Research Article

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ABSTRACT

Soldering is performed in order to easily assemble electronic components and also to provide electrical conductivity. The strengths, hardness, physical properties and electronic properties of the solders, i.e. reduced energy loss, hardness, melting point and longer service life, can be achieved when their usage is improvised by the help of necessary alloying or neutral additions. In this study, the effect of the addition of sub micron sized activated carbon on the mechanical, physical and electrical properties of industrially used solders, i.e. Pb-Sn and lead free SC0.7 solder was investigated. The thermal studies showed that the melting point of Pb-Sn was lowered against lead free solders with increasing amount of activated carbon. The tensile shear strength of both solders did not improve with increasing amount of activated carbon. In lead-free solders, the electrical resistance values decrease with respect to increasing active carbon ratio, however, the resistance of Pb-Sn solders increased. The addition of active particles have positively affected the microstructure of Pb-Sn solders, resulting in a finer grains, whereas, the addition of active carbon particles have no effect on the grain structures of lead free solder.

Keywords: Soldering, lead -free SC0.7 solders, activated carbons, mechanical properties, electrical properties.

Mikron Altı Aktif Karbon Parçacıkları İçeren Pb-Sn ve Kurşunsuz SC0.7 Lehimlerin Mikroyapısal, Mekanik ve Elektriksel Özellikleri

ÖZ

Lehimleme, elektronik bileşenlerin kolayca monte edilebilmesi ve aynı zamanda elektriksel iletkenliğin sağlanması amacıyla yapılmaktadır. Lehimlerin mukavemetleri, sertlikleri, fiziksel özellikleri ve elektronik özellikleri, yani azaltılmış enerji kaybı, sertlik, ergime noktası ve daha uzun hizmet ömrü, kullanımları, gerekli alaşımlama veya nötr ilaveler yardımıyla doğaçlama yapıldığında elde edilebilir. Bu çalışmada, endüstriyel olarak kullanılan lehimlerin yani Pb-Sn ve kurşunsuz SC0.7 lehiminin mekanik, fiziksel ve elektriksel özelliklerine mikron altı boyutta aktif karbon ilavesinin etkisi incelenmiştir. Termal çalışmalar, Pb-Sn'nin erime noktasının, artan aktif karbon miktarı ile kurşunsuz lehimlere karşı düştüğünü göstermiştir. Her iki lehimin çekme kesme mukavemeti, artan aktif karbon miktarı ile gelişmemiştir. Kurşunsuz lehimlerde artan aktif karbon oranına göre elektriksel direnç değerleri düşerken, Pb-Sn lehimlerin direnci artmıştır. Aktif partiküllerin eklenmesi, Pb-Sn lehimlerinin mikro yapısını olumlu yönde etkileyerek daha ince taneler oluşmasına neden olurken, aktif karbon partiküllerinin eklenmesinin kurşunsuz lehimin tane yapıları üzerinde hiçbir etkisi olmamıştır.

Anahtar Kelimeler: Lehimleme, kurşunsuz SC 07 lehimler, aktif karbonlar, mekanik özellikler, elektriksel özellikler.

1. INTRODUCTION

Soldering is an important joining technique used in many fields ranging from electrical and electronics industry, machine, automobile, manufacturing industries, aircraft, aerospace and aerospace applications, radio equipment, telephones to daily installation works. The solder wire used in the electrical and electronics sector consists of a mixture of Sn and Pb metals, and as the amount of tin in the solder increases. the melting temperature decreases and the quality of the solder also increases [1-3]. The sensitivity of the circuit is determined by the quality of the solder. Pb-containing solders have been used for the manufacture of circuit boards for a long time, but with favorable soldering, ease of manufacture, reliability and price effect, the preferred Pb-containing low temperature solders are also toxic and the European Union's regulation on the safe disposal of hazardous substances (RoHS) is being slowly removed from use. Developing Pb free (Lead free) solders, instead, has become widespread especially with the efforts of Japan and the EU as the toxicity has become an issue in many sectors some researches are carried out to eliminate such negative outcomes [5-9].

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Alloying elements added in place of Pb in Pb-Sn solders are not likely to have same effect as in PbSn solders due to their metallurgical properties and there are many studies to make these more expensive class of solders capable of forming a cheaper, easily manufactured and reliable connection [10]. Some properties can be improved with Cu addition, while the Ag content of around 3 wt.% in lead free solders makes this type of soldering quite expensive. Therefore, the addition of various alloying elements and compounds into SAC (SnAgCu) solders to reduce the amount of Ag and maintain their properties have been experimented by many researchers over the past 10 years [11-14]. Since the melting temperature, flux composition and mechanical properties vary with the changing composition, the selection of a suitable solder composition is a non-standard procedure. Pb-free dealloying and intermetallic formation are serious problems of these alloys in the reactions that occur when they come into contact with Cu paths on the circuit. The amount of intermetallics and melting temperature decreases the gap filling capacity as the amount of Cu in the solder increases as the Ag easily oxidizes [11, 14-16]. Alternative studies are focusing on less oxidation products and additions to reduce Cu dissolution. While some of these studies were on flux chemistry, the effect of nano additions such as Alumina, TiO2 on the chemical and mechanical properties of solders was also investigated [18-20]. Although the contribution of nano additions to thermal properties is limited, its effect on mechanical properties, especially creep resistance is quite evident. The addition of metallic nanoparticles in particular Fe, Ni, Cu etc..., affects the chemistry of the solder and promote the formation of intermetallic phase [16-24]. Considering that thickened intermetallic layers reduce the mechanical properties, the mechanical property of the solder is also diminished, especially during the thermal cycle [16, 19, 20, 22, 24, 25]. The effect of chemically inert additives, such as Alumina and TiO2, against the grain refinement and the growth of intermetallic phases is positive, but its behavior in flux is negative [21, 29]. The use of active sub micron particles were studied on Pb-Sn and Sn-Cu (0.3 % and 3 %) type lead free alloys by Talas et al. [27] with varying amounts of active carbon additions (0.14 wt% and 0.3 wt%) produced from orange peels using a reactor operated at approximately 2 Bar. The study showed that the addition of active carbon particles improved the electrical and hardness properties of solders but there was no microscopically considerable difference in lead free solders.

In this study, the effect of submicron size commercially produced activated carbon in different proportions on the mechanical, physical and electrical properties of Pb-Sn and a lead-free solder used industrially in the electronics industry was investigated to determine if the activated carbon would improve the conductivity, increase the strength of both solders to make them viable for possible applications in different industrial sectors.

2. MATERIALS and METHODS

In this study, activated carbon particles were obtained from Alfa Aesar (Sub-micron powders were used after using 500 nm sieve), and were added in the amount of 0.003 wt%, 0.015 wt%, 0.03 wt% and 0.045 wt.% into lead-free (SC0.7: Sn-Cu 0.7) and Pb-Sn solders (63/37) manufactured by Solderex. After melting 20 grams of solders in the melting pot with and without sub micron activated carbon, the melt was poured into 8 mm diameter aluminum casting mold. The samples were then sliced using precision cutting device and metallurgically polished by very fine 1200G sand paper and alumina solutions (1 micron and 0.5 micron powder solutions) followed by examination by optical (Olympus BX-60) and Scanning Electron Microscope (SEM) LEO 1430VP series electron microscopy attached with RÖNTECQX2 EDX (Energy dispersive X-Ray analysis) in BSE (Back Scattered Electron) image mode. Structure, melting point and phase transformation characteristics of the samples were also determined by using Netzsch brand DSC/DTA device at heating rate of 10 C/min and Bruker D8 Advance XRD device at a scanning rate of 3°/min between 2 theta of 10°-100°. In order to test the strength of the produced solders, the copper plates were soldered by using produced solders and subjected to lap joint tensile-shear test as shown in Figure 1.



Figure 1. Lap joint shear test specimens and measurements; total length of the specimens is 200mm and lap joint length is 10mm.

The soldering process temperature was set to 200 °C during the production of joints with Pb-Sn solders with and without active carbon. The soldering temperature was set to 270 °C for lead-free solders with and without active carbon. 1 Kg load was applied on the joint while soldering to ensure homogenous soldering thickness. Then, samples were tensile–shear tested after preloading by using Shimadzu AGS 10 model 10kN

capacity tensile testing machine. The force-elongation curves were recorded and the resulting stress on each sample was calculated and the force / shear area relationship was investigated by measuring the shear cross sectional area. In addition, the specimens were also subjected to electrical resistivity measurement and microhardness tests. Microhardness tests were made using Shimadzu HMV-2 series micro hardness tester using 50g load (HV0.05). For electrical conductivity measurement, electrical resistance measurement was carried out using GWINSTEK brand GOM 802 model miliohm meter with test probes at the ambient temperature of 25 $^{\circ}$ C.

3. RESULTS AND DISCUSSION

3.1. Microscopical Analysis: Additive-free solders

According to the lead-tin reference phase diagram [3], lead dissolves 19.5 wt% tin, while tin dissolves 2.5 wt% lead. These phases in solid solutions are called α and β , respectively. The microstructure image (taken in BSE mode) in Figure 1a shows that the phase containing a high amount of lead, ie, α phase, is white. The dark gray region is the β phase, i.e. the region rich in tin. The phases will then be defined as α (BCC: Body Centered Cubic) and β (BCT: Body Centered Tetragonal). As shown in Figure 2a, the PbSn solder without active sub micron carbon particles, is composed of α and β phases and the phases are homogeneously dispersed, and Figure 2b shows that the lead-free solder without active sub micron-carbon particles is also binary phase.



Figure 2. a) Microstructures of Pb-Sn



Figure 2 (continue). b) Lead free solders without additive

3.2. Microscopical Analysis: Active carbon added Pb-Sn and lead free solders

Figure 3 shows the internal structure pictures of Pb-Sn solders containing active sub micron carbon particles added in percent by weight. As seen in Figure 3, there is no apparent porosity resulting from casting, on the other hand, sub micron carbon particles are seen in α phase, although very small in size. It can be considered that carbon occupy the lattice cavities due to its size or can be partially dissolved in α and β solid solutions. There is no information about how much carbon can be dissolved in Pb in the literature. As a result of rapid cooling from the aluminum die used during casting, the grains remain fine due to the increased number of nuclei during rapid solidification and subsequent heat treatment (Figure 2a). In Figure 3b, in the Pb-Sn sample to which 0.015 wt% of activated sub micron carbon particles is added, a small amount of carbon is believed to be dispersed in the matrix, as well as the solidification cavities and post-casting cracks that occur during casting can be easily observed. According to Figure 3a, the grains are larger and the homogeneous distribution of Pb-Sn a phase is considered. Similarly, it is believed that due to rapid cooling, α phase is dispersed in small size and this is due to the melting temperature of the β phase being lower than the α phase and separated from the liquid phase as given in Pb-Sn phase diagram [3].

Considering that the Pb-Sn solders have eutectic composition, both phases should be formed at the same time. It is possible that the reduction in grain size resulting from the rapid solidification provided by the aluminium die forces the beta phase to contract. Exposure of the beta phase to grain shrinkage can also be explained by the fact that it is the first solidifying phase, in which Pb-Sn solder is slightly hypoeuctoid due to the active sub micron carbon effect. In this structure, it is observed that activated carbon is mixed into the structure. However, it is possible to deduce from the microstructural image that activated carbon particle clustering is formed and homogeneously dispersed within the structure.



Figure 3. SEM images of Pb-Sn solders with active sub micron carbon added a) 0.003, b) 0.015, c) 0.03 and d) 0.045 wt%.

In Figure 3c, the SEM image of Pb-Sn sample added 0.03 wt% active sub micron carbon shows that α phase is more dominant large in ratio and β phase is broken down into very small grains. During casting, the β phase, which solidifies earlier, as in Figure 3b, is thought to be mechanically degraded both by the effect of rapid cooling and by the effect of thermal contraction during solidification.

Figure 3d shows that the grains in the SEM image of Pb-Sn sample containing 0.045 wt% activated sub micron carbon (matrix grains, 3.7 ± 2.3 µm) are finer compared to grains in 0.003 (matrix grain size 5.1±2.6 μ m), 0.015 (matrix grain size 6.3 \pm 1.5 μ m) and 0.03 wt% (matrix grain size 7.3±2.5 µm) activated sub micron carbon addition, carbon-rich phases are also placed at the interfaces of the lead-rich phases and the dimensions are smaller than or equal to 1µm. According to the copper-tin reference phase diagram [3], the solubility of tin increases above 350 °C in a phase. Therefore, tin bronze containing 15.5% tin exists as a single phase. Practically important is the tin alloy, which contains very little copper. The average melting temperature of lead free soldering alloy containing up to 0.7% by weight of copper is about 227°C and as shown in the copper-tin phase diagram, this temperature increases with increasing copper content as it is eutectic at this composition [28, 29].

In reference to the copper-tin phase diagram [28], there is no second phase in tin-based solders, i.e. lead-free solders containing copper, and the microstructure consists of a single phase solid solution of tin at room temperature. Figure 4 shows SEM images of lead free solders with and without activated carbon after casting. Active sub micron carbon additives added in different ratios can be observed in all images. In Figures 4a and b, very small amount of sub micron particles is observed due to the low amount of active sub micron carbon added. However, in Figures 4c and 3d, the active sub micron carbon is distributed more homogeneously in the matrix since a higher proportion of active sub micron carbon is added, i.e. 0.045 wt.%. The absence of grain boundaries suggests that the grains are too coarse or there is insufficient contrast in the BSE mode. In the first case, the absence of a second phase to impede grain advancement and/or movement of the activated carbon particles together with the interface supports coarse grain formation. The images of lead-free solders with different weight percentages of active sub microncarbon added lead-free solders show that grain boundary does not reveal itself in lead-free solders as in Pb-Sn solders. Since Pb-Sn solders have different solubility values for each phase, the crystal formation is expected during nucleation and growth stages. However, in single-phase lead-free solders, no significant grain formation is observed or it is very large in size, regardless of the amount of active carbon.



Figure 4. SEM Images of lead-free solders containing active sub micron-carbon containing a) 0.003 b) 0.015 c) 0.03 and d) 0.045 wt%.





3.3. EDX Analysis of Pb-Sn and Lead-Free Solders

Figure 5 shows SEM-EDX images of Pb-Sn and leadfree samples containing 0.045 wt% active sub micron carbon. The EDX analyzes in Figure 4 indicate the Pb (α) and Sn (β) peaks. Furthermore, there is no aluminum peaks available in EDX results, which may have been originated from the casting mould. There is no evidence of carbon in the EDX analysis of both Pb-Sn and leadfree solders. In lead-free solders, tin was found and other alloying elements could not be observed in the matrix, the amount was not possibly sufficient to be detected.

3.4. Analysis of DTA Results

Figure 6 shows the melting points of lead free and Pb-Sn solders with different amounts of activate sub micron carbon additions. Pb-Sn solders have an increase of few degrees in melting point up to 0.015 wt% active carbon addition. However, the melting temperature of Pb-Sn solder with active carbon addition drops slightly but below the melting point of Pb-Sn solder.

It is possible that the melting temperature of the Pb-Sn solder depends on the additive ratio and may due to the effect of additional active carbon which suppressing the nucleation of liquid phase by preventing the wetting of solid phase, which causes the superheating of the matrix and increase in the melting point. It is ideally possible to cause heterogeneous melting by inserting a neutral surface but active carbon may have reasonably low entropy driving force if there is any solubility which was not detected in this study. As shown in Figure 6, however, it is seen that lead-free solders have a higher melting point which increases with the amount of active sub micron carbon, from 219.8 °C up to 230.9 °C. In contrary to this study, the melting point of SnCu0.7 solder was given as 227 °C in ref [30] however the measuring technique was not stated. Similarly, increasing the amount of active sub micron carbon increases the melting temperature of Pb-Sn solder, from 179.05 °C to 179.91 °C then it goes down to 179.5 °C.



Figure 6. DTA results of melting points (start - finish) of leadbased and lead-free solders with respect to active carbon addition.

3.5. Analysis of XRD Results

Figure 7 shows the XRD peaks of Pb-Sn solders with and without active sub micron carbon in different proportions by weight. According to this graph, it is seen that Pb-Sn solders consist of α and β phase and there is no change in peak counts due to increasing amount of active sub micron carbon which does not cause a new phase formation. XRD peaks of Pb-Sn and lead-free solders to which active sub micron carbon is added in different proportions by weight are given in Figure 8. There are no new peaks appeared in the XRD peaks results of lead-free samples in addition to Sn. In this case, it is thought that the active sub micron carbon additions added in different amounts by weight do not contribute to the formation of a new phase. It indicates that the entire structure consists of a single phase.



Figure 7. XRD results of lead-based solders with and without additives



Figure 8. XRD results of lead-free solders with and without additives

3.6. Microhardness Test Results

Experimental results on both Pb-Sn solder and lead-free solders (Figure 9) showed that the amount of activated carbon was not fully correlated with hardness changes. PbSn solders appears to show an increase in the micro hardness with respect to activated sub micron carbon particles after 0.03 wt% addition, whereas, lead free solder with sub micron carbon additive was rarely

affected by the increase in the amount of activated sub micron carbon except for 0.045 wt% with a little increase in hardness. It is surprising that PbSn solders with activated submicron carbon additive showed a softening effect between 0.003 and 0.03 wt% which may be due to grain coarsening observed between 0.003 - 0.03 wt.% additions in Pb-Sn solders. It is considered that the high hardness values at the highest additive ratio are due to the increase in volume of the activated carbon. In this way, it is believed that due to the increased second phase ratio in the matrix, the hardness due to the dislocation movement restriction in the matrix increases. It should also be considered that, apart from the amount of the second phase, one or both of the Pb-Sn microstructure may be able to dissolve carbon, which may have resulted in increased matrix hardness. The micro hardness of SnCu0.7 solder is found to be consistent with study [30].



Figure 9. Microhardness test results of PbSn and lead free solder samples

3.7. Lap joint tensile shear test results

The tensile-shear strength test results (from lap joints) of the PbSn soldered specimens are shown in Figure 10a. The results are calculated as shear stress and total elongation amount is entered as horizontal axis. It is seen that Pb-Sn and lead-free solders have an opposite behavior with a downward trend. The shear stresses of PbSn solders are higher in pure and low active carbon contents. It is seen that the effect of active carbon additive is low at values of 0.03 wt% and above. According to this result, activated carbon is thought to be effective in low contribution rates in terms of mechanical strength and is especially effective in leadbased solders. The Pb-Sn solders in the double phase structure affect the phase transformation kinetics during solidification and thus change the grain size or shape of grains, and when added in high amounts, the activated carbon particles become coarse and ineffective. The results of the tensile-shear tests of lead free solders are given in Figure 10b. Results of both groups show that, the lap joint shear strengths generally change strongly with the increasing the amount of addition. As can be observed in Figure 10 that active carbon addition to

PbSn types of solders have a softening effect. This may be a result of coagulation of active carbon particles however there would be an initial increase in the strength at low additions, which is not the case in lead





Figure 10. Tensile-shear test results of solder joints produced with a) PbSn and b) lead free solders with and without activated carbon.

free solders but Pb-Sn solders show an initial increase and then decrease in the strength at maximum addition. A different mechanism regarding the softening behavior is given in reference [31] in which a strong bonding in the cluster of additives causes the formation of cracks which reaches the surface due to the difference in plastic behavior of matrix and clustered region. After the tensile shear experiments, it was observed on failed surfaces that all samples failed from the soldering zone. The strength of Pb-Sn soldered samples in Figure 10 is initially increased but later decreased with increasing carbon content due possibly to the coarse grains in 0.015 wt% and 0.03 wt% additions. Shear elongation, i.e. the deformation of the sample, also increased from 0.015 wt% up to 0.03 wt% with a slight decrease at the highest additions, i.e. 0.045 wt%, where the effect of large amount of activated carbon addition begins to appear. Accordingly, the shear forces of the samples are also reduced. The shear strengths of the joints made with lead-free solder have a decreasing trend and the elongation decreases with the increasing amount of activated carbon, especially in lead-free solders. This suggests that in the tensile-shear samples joined with lead-free solders, the coagulation of the activated carbon during casting may be responsible in addition to single phase microstructure. Because activated carbon is not an effective second phase owing to its very small size, it does not also strongly affect the phase structure by nucleation or other mechanisms. However, in general, the strength of tensile-shear samples soldered with leadfree solder was lower than that of Pb-Sn ones due mostly to the formation of single phase structure with large grains. While the elongation values of samples produced with lead-free solders generally remain the same, however, this value varies in Pb-Sn ones.

The tensile shear values of PbSn solders and lead free solders do not correlate with hardness test results but with the coarseness of grains (in case of PbSn solders) with respect to increasing activated sub micron carbon content. The second phase particle hardening seems to be an effective mechanism in case of lead free solders when tensile shear test is concerned.

3.8. Electrical Resistivity

The results obtained in electrical measurements are given in Figure 11. Because the resistivity of a metal or conductor is dependent on the size and length of the cross-sectional area, a method of length and crosssection is used instead of spatial measurement. As the effective cross-sectional area and length of the solder bar were taken into consideration, the measurement results were calculated as ohm.meter. According to these values, the electrical resistance values of lead-free solder samples decreased with respect to the increasing contribution rate, i.e. approximately 27% decrease with the addition of 0.003 wt% active carbon and finally 45% decrease with the addition of 0.045 wt%, whereas the electrical resistance of Pb-Sn solders was increased unlike the study by Talas et. al [27] where ten times more active carbon particles were used. There are research results in the literature suggesting that the resistance of lead-free solders with CNT (Carbon Nano Tube) is reduced [31, 32], but the literature is limited for active sub micron sized or activated carbon added lead based solders.



Figure 11. Electrical resistivity test measurement results

4. CONCLUSIONS

Following conclusions can be drawn from this study:

1. In lead-free solders, the electrical resistance values decrease with respect to the increasing additional activated carbon ratio, however, the resistance of Pb-Sn solders increased.

2. It has been revealed that the amount of active carbon and hardness changes are not strongly interrelated in both Pb-Sn and lead-free solders.

3. It is observed that shear strengths of leaded solders increase with the increasing amount of activated carbon, however, shear strengths of lead free solders decrease with increasing amount of activated carbon additives.

4. It has been observed that the additives cause a softening effect as in shear strength of lead free solder.

5. No new phase peaks appeared in the XRD peaks results of lead-free and Pb-Sn solders.

6. The addition of active sub micron carbon reduces the melting point of the Pb-Sn solder, while in lead-free solders, the melting temperature increases depending on the amount of active sub micron carbon.

7. The introduction of activated carbon particles to the lead free solder (SC0.7) make this solder more useful where the high conductivity is concerned. It is possible to introduce particle in solder paste, however, an experiment is needed to find out how the wetting angles to change.

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DECLARATION OF ETHICAL STANDARDS

The author(s) of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

AUTHORS' CONTRIBUTIONS

Şükrü TALAŞ: Wrote the manuscript. Performed the experiments and analysed the results, Contributed to the development of the conceptual processes of the study.

Elif ÖZKAN: Performed the experiments, data collection and data analysis.

Bahattin AYAR: Performed the experiments, data collection and data analysis.

CONFLICT OF INTEREST

There is no conflict of interest in this study.

REFERENCES

- Kotadia, H. R., Howes, P. D., Mannan, S. H., "A review: on the development of low melting temperature Pb-free solders", *Microelectronics Reliability*, 54(6– 7):1253-1273.
- [2] Jones, W. K., Liu, Y. Q., Zampino, M. A., Gonzalez, G. L., "the at-temperature mechanical properties of lead-tin based alloys", In: Harman G., Mach P. (eds) *Microelectronic Interconnections and Assembly*. NATO ASI Series (3. High Technology), Springer, Dordrecht, (1998).
- [3] Karakaya, I., Thompson, W. T., "The Pb-Sn (lead-tin) system. *Journal of Phase Equilibrium*, 9: 144–152. (1988).
- [4] Demayo, A., Taylor, M. C., Taylor, K. W., Hadson, P. V. and Hammond, P.B., "Toxic effects of lead and lead compounds on human health, aquatic life, wildlife plants, and livestock", *Critical Review in Environmental Science and Technology*, 12(4);257-305, (1982).
- [5] European Union, Directive 2002/95/EC of the European Parliament and of the Council of 27 January 2003 on the Restriction of the Use of Certain Hazardous Substances in Electrical and Electronic Equipment. *Official Journal of European Union*, 46:19-23, (2002).
- [6] Suganuma, K., "Advances in lead-free electronics soldering", *Current Opinion in Solid State Materials Science*, 5(1):55-64, (2001).
- [7] Aşçı, T., Keskin, H., "Strengthening the retention amount and leaching resistance of boron compounds used as impregnation material". *Journal of Polytechnic*, 24:103-112, (2021).
- [8] Aydogan, T., Şenberber Dumanlı, F. T., Möröydor Derun, E., "Effect of lemon peel extract concentration on nano scale Fe/Fe3O4 synthesis". *Journal of Polytechnic*, 25:1423-1427, (2022).
- [9] Laad, M., "Synthesis and characterization of copper metal matrix composite reinforced with ceramic oxide extracted by the green route". *Journal of Polytechnic*, 25:1185-1189, (2022).
- [10] Zeng, K., Tu, K. N., "Six cases of reliability study of pbfree solder joints in electronic packaging technology", *Materials Science and Engineering R: Reports*, 38(2):55–105, (2002).
- [11] Seo, S-K., Kang, S. K., Shih, D-Y., Lee, H. M., "The evolution of microstructure and microhardness of Sn-Ag and Sn-Cu solders during high temperature aging". *Microelectronics Reliability*, 49(3):288–295, (2009).
- [12] Felberbaum, M., Ventura, T., Rappaz, M., Dahle, A. K., "Microstructure formation in Sn-Cu-Ni solder alloys", *Journal of Materials*, 63(10):52–55, (2011).
- [13] Wang, J-X., Xue, S-B., Han, Z-J., Yu, S-L., Chen, Y., Shi, Y-P., Wang, H., "Effects of rare earth ce on microstructures, solderability of Sn-Ag-Cu and Sn-Cu-Ni solders as well as mechanical properties of soldered joints", *Journal of Alloys and Compounds*, 467(1):219– 226, (2009).
- [14] Reeve, K. N., Handwerker, C. A., "Beta-tin grain formation in aluminum modified lead-free solder alloys", *Journal of Electronic Materials*, 47(1):61–76, (2018).

- [15] Laurila, T., Vuorinen, V., Paulasto-Kröckel, M., "Impurity and alloying effects on interfacial reaction layers in Pb-free soldering", *Materials Science and Engineering: R Reports*, 68(1-2):1-38, (2010).
- [16] Nogita, K., Gourlay, C. M., Nishimura, T., Cracking and phase stability in reaction layers between Sn-Cu-Ni solders and Cu substrates", *Journal of Materials*, 61(5):45–51, (2009).
- [17] Satyanarayan, K. N. P., "Reactive wetting, evolution of interfacial and bulk imc's and their effect on mechanical properties of eutectic Sn-Cu solder alloy", *Advances in Colloid and Interface Science*, 166(1):87–118, (2011).
- [18] Mohd Salleh, M. A. A., Mcdonald, S. D., Nogita, K., "Effects of Ni and TiO2 additions in as-reflowed and annealed Sn0.7Cu solders on Cu substrates", *Journal of Materials Processing Technology*, 242:235–245, (2017).
- [19] Lee, J., Chu, K., Patzelt, R., et al., Effects of Co addition in eutectic Sn-3.5Ag solder on shear strength and microstructural development", *Microelectronic Engineering*, 85(7):1577–158, (2008).
- [20] Alam, M. E., Nai, S. M. L., Gupta, M., "Development of high strength Sn-Cu solder using copper particles at nanolength scale", *Journal of Alloys and Compounds*, 476(1):199–206, (2009).
- [21] Ke, C. B., Zhou, M. B., Zhang, X. P., "Phase field simulation on microstructure evolution and growth kinetics of Cu6Sn5 intermetallic compound during early interfacial reaction in sn/cu soldering system", *Acta Metallurgica Sinica*, 50(3):294–304, (2014).
- [22] El-Daly, A. A., Hammad, A. E., "Enhancement of creep resistance and thermal behavior of eutectic sn-cu leadfree solder alloy by Ag and In additions", *Materials and Design*, 40:292–298, (2012).
- [23] Sabri, M. F. M., Salleh, M. F. M., Jaffery, S. H. A., Mahdavifard, M. H., "Properties of Sn0.7Cu solder alloys bearing Fe and Bi", In: Salleh, M.A.A.M., Abdul Aziz, M.S., Jalar, A., Izwan Ramli, M.I. (eds) Recent Progress in Lead-Free Solder Technology. *Topics in Mining, Metallurgy and Materials Engineering*. Springer, (2022).
- [24] Yang, L., "Effects of Ag particles content on properties of Sn0.7Cu solder", *Journal of Materials Science*, 24(5):1405–1409, (2013).
- [25] Zhao, N., Pan, X. M., Yu, D. Q., Ma, H. T., Wang, L., "Viscosity and surface tension of liquid Sn-Cu lead free solders", *Journal of Electronic Materials*, 38(6):828– 833, (2009).
- [26] Eid, E. A., Fouda, A. N., Duraia, E. M., "Effect of adding 0.5 wt% ZnO nanoparticles, temperature and strain rate on tensile properties of sn-5.0 wt% Sb- 0.5 wt% Cu (SSC505) lead free solder alloy", *Materials Science Engineering A*, 657:104–114, (2016).
- [27] Talas, S., Gökçe, B. and Çakmakkaya, M., "Sn-Pb and lead free solders containing active carbon particles", IOP Conf. Series: *Materials Science and Engineering*, 146:012043, (2016).
- [28] Fürtauer, S., Li, D., Cupid, D., Flandorfer, H., "The Cu-Sn phase diagram, part 1: new experimental results", *Intermetallics*, 34:142-147, (2013).

- [29] Ventura, T., Terzi, S., Rappaz, M., Dahle, A. K., "Effects of solidification kinetics on microstructure formation in binary Sn-Cu solder alloys", *Acta Materialia*, 59(4):1651–1658, (2011).
- [30] Nazri, S. F., Salleh, M. A. A. M., "Effect of rare-element (Ga) addition on the microstructure and mechanical properties of Sn-0.7Cu and Sn-0.7Cu-0.05Ni lead-free solder alloys", IOP Conf. Series: *Materials Science and Engineering*, 701: 012031, (2019).
- [31] Nai, S., Wei, J., Gupta, M., "Effect of carbon nanotubes on the shear strength and electrical resistivity of a leadfree solder", *Journal of Electronic Materials*, 37:515– 522, (2008).
- [32] Kang, S. K., Choi, W. K., Yim, M. J. et al., "Studies of the mechanical and electrical properties of lead-free solder joints", *Journal of Electronic Materials*, 31:1292–1303, (2022).