Abstract

Purpose – Tin-Silver-Copper is widely accepted as the best alternative to replace Tin-Lead solders in microelectronics packaging due to their acceptable properties. However, to overcome some of the shortcomings related to its microstructure and in turn, its mechanical properties at high temperature, the addition of different elements into Tin-Silver-Copper is important for investigations. The purpose of this work is to analyze the effect of lanthanum doping on the microstructure, microhardness and tensile properties of Tin-Silver-Copper as a function of thermal aging time for 60 h, 120 h and 180 h at a high temperature of 150°C and at high strain rates of 25/s, 35/s and 45/s.

Design/methodology/approach - The microstructure of un-doped and Lanthanum-doped Tin-Silver-Copper after different thermal aging time is examined using scanning electron microscopy followed by digital image analyses using ImageJ. Brinell hardness is used to find out the microhardness properties. The tensile tests are performed using the universal testing machine. All the investigations are done after the above selected thermal aging time at high temperature. The tensile tests of the thermally aged specimens are further investigated at high strain rates of 25/s, 35/s and 45/s.

Findings - According to the microstructural examination, Tin-Silver-Copper with 0.4 wt% Lanthanum is found to be more sensitive at high temperature as the aging time increases which resulted in coarse microstructure due to the non-uniform distribution of intermetallic compounds. Similarly, lower values of microhardness, yield strength and ultimate tensile strength come in favors of 0.4 wt% Lanthanum added Tin-Silver-Copper. Furthermore, when the thermally aged tensile specimen is tested at high strains, two trends in tensile curves of both the solder alloys are noted. The trends showed that yield strength and ultimate tensile strength increase as the strain rate increase and decrease when there is an increase in thermal aging.

Originality/value – The addition of higher supplement (0.4 wt%) of Lanthanum into Tin-Silver-Copper showed a lower hardness value, yield strength, ultimate tensile strength, ductility, toughness and fatigue in comparison to un-doped Tin-Silver-Copper at high temperature and at high strain rates. Finally, simplified material property models with minimum error are developed which will help when the actual test data are not available.

Keywords Electronic Solders; Thermal Aging Time; High Strain Rate; Microstructure; Mechanical Properties
1. Introduction

Tin-Lead (Sn-Pb) alloy has a long history as a soldering material in the electronics industry, but its negative impact on environment has restricted its use (Sadiq et al., 2013). Therefore, different Lead (Pb)-free solders have been developed to replace Sn-Pb solders (Yang et al., 2001). Tin-Silver-Copper Sn-Ag-Cu (SAC) alloy is acknowledged as one of the commercially acceptable Pb-free electronic solders (Aamir et al., 2017c). A survey shows that almost 70% acceptable Pb-free solders are SAC alloys due to their good properties over other Pb-free eutectic alloys used in microelectronic manufacturing (Shnawah et al., 2012). Moreover, SAC series gives better joint strength, mechanical, thermal and fatigue properties for providing good mechanical support in electronic devices (Lee et al., 2015).

Tin (Sn) has good ductility and corrosion resistance in air. Also, higher Sn concentration leads to good tensile and shear strength of solders (Zhang, 2011). Silver (Ag) has good strength and improves resistance to fatigue from thermal cycling. However, high Ag contents with high strength show reasonably low ductility. Therefore, it is highly suggested that the concentration of Ag in SAC should be 2 - 3.9 wt% (Che et al., 2010). Addition of Copper (Cu) in SAC is responsible for the improvement of wettability, thermal-mechanical performance of the alloy and lowers their melting temperature (Nimmo, 2004). However, higher Cu content increases the pasty range and, the gap between the liquidus and solidus temperature which is not suitable for solder joint reliability. Therefore, Cu content in SAC should be limited to 0.5 - 0.9 wt% (Belyakov, 2009).

In SAC series, SAC305 got more attention due to its acceptable wetting properties, better mechanical properties, suitable melting temperature, excellent fatigue resistance and solder joint reliability (Aamir et al., 2017b). Moreover, it is widely used for surface mount technology card assembly and for ball-grid-array interconnection in the microelectronic packaging industry (Yasmin et al., 2014). However, one of the core issues pertaining to SAC-305 is the formation and growth of large IMCs in this alloy (Yasmin and Sadiq, 2014).

It is found that the presence of Rare-earth (RE) elements in SAC is considered a good supplement because of their special surface-active properties for the refinement of the microstructure and improvement in mechanical properties (Zhang et al., 2010). However, to avoid further coarsening of the microstructure and degradation in mechanical properties, it is important to select its proper and optimal concentration (Sun and Zhang, 2015). In this regard,
many researchers have tried the addition of RE elements, for instance, Lanthanum (La), Cerium (Ce), and Yttrium (Y) were studied by (Dudek and Chawla, 2010), Ce by (Chen et al., 2011) and (Tu et al., 2017) Ce and La by (Yu et al., 2004). Y by (Hao et al., 2007). Erbium (Er) by (Shi et al., 2008). Furthermore, Praseodymium (Pr) and Neodymium (Nd) by (Gao et al., 2010b.Gao et al., 2010a), Ytterbium (Yb) by (Zhang et al., 2014). Among all RE supplements, La is considered as a good additive due to its lower cost, wide availability, and low melting point as compared with other RE elements (Aamir et al., 2017b). Furthermore, La got considerable attention from many researchers including (Pei and Qu, 2008a), (Pei and Qu, 2008b), (Yasmin et al., 2014), (Ali, 2015), (Aamir et al., 2017b) (Sadiq et al., 2013), but still it is taken to be in account that greater than optimum composition has negative impact on the properties of the bulk solder (Zhou et al., 2007).

Thermal aging is important in effecting the microstructure and properties of any solder system to get a better understanding of the performance by considering the operating conditions during service (Aamir, 2015). During thermal aging, the microstructure changes significantly particularly the Intermetallic compounds (IMCs) which have great contribution in the reliability of any soldered joint under the service conditions (Wang et al., 2014). Because the operating temperature of many new electronic systems could be as high as 150°C to 200°C, like electronics in oil and gas exploration, avionics, automotive industry, and defense applications typically have more demanding thermal life cycle environments than consumer electronics (McCluskey et al., 1996). These demanding high-temperature conditions of use, together with the need for greater reliability of all electronic systems motivate further research on the effects of high-temperature aging of solder materials (Anderson and Harringa, 2004).

In addition, the microhardness is also considered as one of the acceptable ways to find the mechanical properties of materials including electronic solder alloys (Wei et al., 2009). The microhardness of the solder is often connected to how the metallic material resists wearing or abrasion (Chen et al., 2016). Moreover, the impact of high strain on the solder joint is another important issue because solder joints in electronic applications experience high strain rate deformation under conditions such as accidental drop/impact (An and Qin, 2014). Lower strain rates were normally defined at less than 1/s and any change in strain rate to the exposed electric components altered these properties. Furthermore, information related to high strain rate is uncommon when the electronics experienced a typical drop-impact, shock and vibration at high strain rates of 1/s to 100/s (Lall et al., 2014). Therefore, it is vital to investigate the behaviour of electronic solders at high temperature and at high strain rates.
In our previous work, it was concluded that the addition of La (0.4 wt %) into SAC and thermal aging resulted in bulk IMCs due to destabilization of the boundaries, thus making the microstructure coarsen and leads to degrading the mechanical properties. However, the investigation was done at different thermal aging temperature at 60°C, 100°C and 140°C for aging time of 50 hours. For more details, readers are referred to (Aamir et al., 2017b). In this work, the study of microstructure evolution and tensile properties including yield strength (YS) and ultimate tensile strength (UTS) are further extended to different aging times of 60 h, 120 h and 180 h at high temperature of 150°C. The reason of selecting this aging time is due to the fact that the rapid growth of IMCs starts after 50 h aging time when the modern electronic devices are subjected to severe thermal conditions (Sadiq, 2012). Furthermore, in this study the microhardness is also taken into consideration, therefore, un-doped and La-doped SAC305 are investigated at different aging time and at high temperature. Moreover, the thermally aged tensile specimens are investigated at high strain rates of 25/s, 35/s and 45/s. The main objective of this paper is to study the inclusion of La on the microstructure, microhardness and tensile properties of SAC305 as a function of thermal aging time for 60 h, 120 h and 180 h at a high temperature of 150°C and at high strain rates of 25/s, 35/s and 45/s. In addition, some discussion based on ductility, toughness and fatigue is also presented. Finally, simplified material property relations are developed for the prediction of IMCs at different aging time and, mechanical properties at different aging time and high strain rates which gives a very minimum percent error.

2. Materials and methods

The samples of pure Sn, Ag, Cu and La obtained in powder form are cast by putting them in Alumina crucible, after weighing the pure metals in the proportion of weights. To ensure complete melting, the crucible containing metals is heated in the furnace and finally, the molten material is poured into the preheated die to get the targeted alloys. Figure 1 shows the tensile specimen with uniform dimensions obtained after the die casting process with the 3D CAD model. The thickness of the resulting specimens is 2 mm.
For the investigation of microstructure examination, the specimens are cut into pieces using cutting machine (Techcut 4™ cutting machine, 10-500rpm) and mounted in Bakelite for proper handling and to avoid any distortion. After initial metallurgical Silicon carbide sandpaper grinding, the specimens are polished with a polycrystalline diamond suspension of 6 μm, 1 μm and 0.25 μm as the abrasive particle size on cotton silk paper to give extra shine to the surface. For grinding and polishing a Twin Prep 3TM grinding/polishing machine (25-500rpm) is used. The specimens are then cleaned by distilled water to remove any residue left during polishing. For thermal aging, the specimens are exposed in a drying oven (Thomas Scientific Model 605) at different levels of aging times (60 h, 120 h and 180 h) at high temperature of 150°C. The specimens are also etched with 95% ethanol and 5% hydrochloric acid solution. The etching time is selected based on previous study in (Sadiq, 2012) that used 9 seconds for SAC305, 28 seconds for SAC-0.25La and 58 seconds for SAC-0.5La. Therefore, the etching time in this study for SAC305 is selected as 9 seconds and for SAC305-0.4La the etching time is checked between 28 - 58 seconds with a number of rounds of polishing and etching. Finally, etching time of 40 seconds is selected for SAC305-0.4La. The purpose of etching is to help reveal different aspects of the microstructure to observe the IMCs clearly to get the desired results. Scanning electron microscopy (SEM) images are taken using Scan electron microscope (Model: JSM5910, JEOL, Japan with maximum magnification of 300,000X) to examine the microstructure. Electron dispersive X-ray (EDX) analysis is carried out to verify the chemical compositions. Afterwards, tensile tests are carried out using universal testing machine. At least three specimens are used for each tensile test. The microstructure analysis and mechanical properties are investigated after each stage of aging time for comparison. Furthermore, tensile tests are investigated at different strain rates using the same tensile specimens as shown in
3. Results and discussions

3.1. Microstructure analyses at different aging time

Since, the microstructure in this study is investigated in terms of IMCs; therefore the SEM images of SAC305 (Figure 2(a)) and SAC305-0.4La (Figure 3(a)) are further analyzed using ImageJ as shown in Figure 2(b) for SAC305 and Figure 3(b) for SAC305-0.4La. The composition of SAC305 and SAC305-0.4La confirmed by EDX is given in Figure 2(c) and Figure 3(c), respectively.

The SEM images displaying the IMCs formation and growth rate of SAC305 and SAC305-0.4La at elevated temperature (150 °C) for 60 h, 120 h and 180 h aging time analyzed in ImageJ are shown in Figure 4. The data extracted from ImageJ with 5% error is given Figure 5. The size of average IMCs particle size of SAC305 and SAC305-0.4La is given in Table 1. The percent increment of IMCs particle size of SAC305 at 180 h and 120 h with respect to 60 h aging time is 17.4% and 25.77%, respectively. However, in comparison to SAC305, the percent increment of IMCs size of SAC305-0.4La is identified to be more i.e., 19.70% at 120 h and 26.37% at 180 h aging time with respect to 60 h. Therefore, it is clear that on increasing aging time from 60 h to
120 h and 180 h at high aging temperature of 150°C, the IMC particles started to join together to form the bulk IMCs due to atomic diffusion. However, in comparison to SAC305 alloy, the IMCs formation and growth rate of La doped SAC305 at elevated temperature for different aging time are more that caused the microstructure to coarsen due to its high supplement (0.4 wt %) which is in consistent with (Zhou et al., 2007) and our previous study in (Aamir et al., 2017b). Therefore, addition of La in SAC305 should be limited to less than 0.4 wt% for different aging time and at high temperature. Furthermore, there are chances that this coarsening of microstructure either due to bulk IMCs affected by thermal aging time at high temperature or non-uniform distribution of IMCs due to the 0.4 wt% La concentration in SAC305 should contribute in degrading the mechanical properties (Aamir et al., 2017b) (Ali, 2015). To justify and confirm this conclusion, mechanical properties of both solder alloys are evaluated at different aging times (60 h, 120 h, and 180 h) at elevated temperature (150°C), to justify the microstructural transformation with mechanical properties.
Figure 4: SEM images analyzed in ImageJ at different aging times at 150 °C
In order to predict the average IMCs size at different aging time, simplified material property models are developed. Eqs (1) - (2) represent the relation between the aging time and IMCs.

This is the first time, up to the authors' knowledge, that simplified mathematical relations between the aging time and growth rate of average IMCs particle size are developed. These equations are valid for satisfying and predicting values of average IMCs for the given values of aging time presented below:

\[
\begin{align*}
IMCs_{(SAC305)} &= A_1 \cdot (t_g)^{A_2} \\
IMCs_{(SAC305-0.4La)} &= B_1 \cdot (t_g)^{B_2}
\end{align*}
\]

Where \( t_g \) is the aging time, and \( A_1, A_2, B_1 \) and \( B_2 \) are the material constants given in Table 1.

The predicted values of average IMCs particle size as a function of aging time computed from the above Eqs. (1) - (2) for the SAC305 and SAC305-0.4La shows a close correspondence with the experimental values. The predicted values in comparison with the experimental values are presented in Table 2 with error analysis which shows that the predicted values of average IMCs are accurate with experimental data with a maximum of 1.661% and a minimum of 0.145%.
Table 1: Material property constants of average IMCs particle size

<table>
<thead>
<tr>
<th>Solder Alloy</th>
<th>Material constants used in Eqs. (1) - (2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SAC305</td>
<td>A1</td>
</tr>
<tr>
<td></td>
<td>0.1898</td>
</tr>
<tr>
<td>SAC305-La</td>
<td>B1</td>
</tr>
<tr>
<td></td>
<td>0.2268</td>
</tr>
</tbody>
</table>

Table 2: Experimental and predicted data of average IMCs particle size and % errors at different aging time

<table>
<thead>
<tr>
<th>Solder alloy</th>
<th>Thermal aging time at 150 ºC</th>
<th>Average IMCs particle size (µm)</th>
<th>% Errors</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Experimental values</td>
<td>Predicted values</td>
</tr>
<tr>
<td>SAC305</td>
<td>60 h</td>
<td>0.576</td>
<td>0.577</td>
</tr>
<tr>
<td></td>
<td>120 h</td>
<td>0.698</td>
<td>0.696</td>
</tr>
<tr>
<td></td>
<td>180 h</td>
<td>0.776</td>
<td>0.777</td>
</tr>
<tr>
<td>SAC305-La</td>
<td>60 h</td>
<td>0.701</td>
<td>0.708</td>
</tr>
<tr>
<td></td>
<td>120 h</td>
<td>0.873</td>
<td>0.858</td>
</tr>
<tr>
<td></td>
<td>180 h</td>
<td>0.952</td>
<td>0.960</td>
</tr>
</tbody>
</table>

3. 2. Tensile testing at different aging time

Investigating the mechanical properties of solder joints such as YS and UTS are of paramount importance due to the fact that solder joints are the weakest link as far as the structural integrity of the electronic modules is concerned. For instance, in the applications where higher fatigue and vibration loadings are acting, the role of mechanical properties is evident. Furthermore, at higher temperature, the solder assemblies are subjected to tensile loading during services and in some cases, due to flexing down the substrate, excessive deformation takes place in the solder joints as shown in Figure 6 (Abtew and Selvaduray, 2000). Therefore, it is important to investigate the tensile test of the solder assemblies at different aging times and at high temperatures to determine the tensile deformation which the solder joint can sustain before ultimate failure (Sadiq, 2012).
The tensile testing of SAC305 and SAC305-0.4La at different aging times of 60 h, 120 h and 180 h at high temperature of 150ºC is shown in Figures 7 and 8, respectively. In all thermal aging time, it is noted that both candidate alloys (SAC305 and SAC305-0.4La) showed a decrease in the YS and UTS. The percent increment of YS and UTS of SAC305 solder alloy at 120 h aging time at 150ºC is (16.68 % and 5.45 %) and (29.36 % and 11.51 %) at 180 h with respect to 60 h. In comparison to SAC305, a significant decrease in YS and UTS is identified. The YS and UTS at 120 h aging time at 150ºC is (17.91% and 30%) and (8.40% and 14.06%) at 180 h aging time with respect to 60 h aging. However, the SAC305 with 0.4 wt% La shown the lowest mechanical properties and are found to be more sensitive to the thermal aging time at high temperature. Since, mechanical properties have strong link with the microstructure evolution. Therefore, it is quite clear that the reason of lower strength of 0.4 wt% La is due to the coarsen microstructure because of the bulk IMCs and these thick IMCs are responsible for the deterioration of mechanical properties. This also reflects that the doping of 0.4 wt% La did not facilitate the formation of IMCs particles at the interface of La-bearing solder joints to the increase of the strength of solder alloy. Therefore, this comprehensive study expects that SAC305 joint might be resulted in the best mechanical performance when the La concentration should be lower than 0.4 wt% even after aging time at high temperatures.
Figure 7: Stress strain curve of SAC305 at different aging time at 150°C

Figure 8: Stress strain curve of SAC305-0.4La at different aging time at 150°C
Another important mechanical property is the ductility which is the measure of elongation to failure of a material and in which the amount of plastic deformation takes place in a material before ultimate failure. Figures 7 and 8 depict that the elongation to failure for SAC-0.4La as compared with SAC305 experience some decrease in elongation to failure. This claim is in agreement to the findings of (Ali, 2015) which concluded that ductility is decreased when the concentrations of La doping is enhanced beyond 0.3 wt%. Furthermore, it was discussed that a material with coarsen microstructure, lower strength and ductility also exhibits inferior toughness which is the substantial amount of impact energy a material can absorb before fracture (Yu et al., 2014). Combining all the investigations in this study, it is now logically expected that SAC-0.4La should be having a lower toughness due to its lower strength, lower ductility and a coarse microstructure than SAC305 and therefore, 0.4 wt% La doped SAC305 is expected to have more attraction towards fracture during impact.

Similarly, fatigue is a very important type of loading which must be considered when the structures are introduced into the cyclic loading. For example, the electronic panels/cards embedded in the flight computer of an aircraft which must be ruggedized to withstand the severe shocks and fatigue loading encountered in the service. To ascertain the electronic connections in the panels, solder joints must be strong enough to perform in the desired environment and must be qualified. In solder joints the formation of failure crack initiates at their corner and propagates along the region of strain concentration (Zhu et al., 2014). It is believed by different researchers in (Chicot et al., 2013; Jankowski et al., 2014; Pei and Qu, 2008a; Ali, 2015) that those solder alloys which have a better combination of strength and ductility also offer an acceptable resistance to fatigue. Based on these justifications by different researchers, along with the experimental observations in this study, it logically follows again that SAC-0.4La should show less fatigue behaviour due to lower combination of strength and ductility compared with SAC305. This expected decrease in fatigue behavior might be associated with the worse bonded grains (coarsen microstructure) that gives lower damage resistance and thus do not prevent cracks from initiation.

To further discuss the tensile tests, a fractography analysis is done to check the fracture surface. Figure 9 shows the SEM images of both the solder alloys after thermal aging time at high temperature analyzed in ImageJ. The reason of displaying the SEM images in ImageJ is to give a clear reflection of the fracture surface. The fractograph comprises the fracture humps or terraces and there are several regions revealing sticky surface and shows a ductile character while, the SAC305-0.4La also shows some small granular particles.
From the analysis of testing results, the relationship between mechanical properties such as YS and UTS are developed and rendered in Eqs (3) – (4).

\[
YS_{(SAC305)} = X_1 \cdot (t_g - X_2)^{X_3}
\]

\[
UTS_{(SAC305)} = Y_1 \cdot (t_g - Y_2)^{Y_3}
\]

\[
YS_{(SAC305 - 0.4La)} = X_4 \cdot (t_g - X_5)^{X_6}
\]

\[
UTS_{(SAC305 - 0.4La)} = Y_1 \cdot (t_g - Y_2)^{Y_3}
\]

Where \( t_g \) is the aging time and the material constants are listed in Table 3. These material property models can be used to calculate the mechanical properties at different aging times. Table 4 shows the calculated values and error analysis to appraise the accuracy of mathematical relations. It can be seen that calculated values are in agreement with experimental values with minimum \( 1.35 \times 10^{-14} \) error and maximum \( 6.14 \times 10^{-1} \) for YS and, \( 1.46 \times 10^{-2} \) minimum error and \( 6.01 \times 10^{-1} \) maximum error for UTS. These % errors are very small and near to negligible reflecting that the Eqs (3) – (6) are valid for mechanical properties when the solder joint reliability is taken into consideration at different aging times.
Table 3: Material constants for YS and UTS at different aging time

<table>
<thead>
<tr>
<th>Solder Alloy</th>
<th>X1</th>
<th>X2</th>
<th>X3</th>
<th>Y1</th>
<th>Y2</th>
<th>Y3</th>
</tr>
</thead>
<tbody>
<tr>
<td>SAC305</td>
<td>$1.755 \times 10^3$</td>
<td>$-1.739 \times 10^2$</td>
<td>$-8.316 \times 10^{-1}$</td>
<td>$3.547 \times 10^2$</td>
<td>$-3.454 \times 10^2$</td>
<td>$-4.642 \times 10^{-1}$</td>
</tr>
<tr>
<td>SAC305-0.4La</td>
<td>$8.897 \times 10^2$</td>
<td>$-1.498 \times 10^2$</td>
<td>$-7.878 \times 10^{-1}$</td>
<td>$5.723 \times 10^1$</td>
<td>$-7.895 \times 10^1$</td>
<td>$-2.439 \times 10^{-1}$</td>
</tr>
</tbody>
</table>

Table 4: Experiment and predicted data of (YS and UTS) and % errors at different aging time

<table>
<thead>
<tr>
<th>Strain rate</th>
<th>Thermal aging time at 150 ºC</th>
<th>Experimental values</th>
<th>Predicted values</th>
<th>% Error</th>
</tr>
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<tbody>
<tr>
<td>SAC305</td>
<td></td>
<td>YS</td>
<td>UTS</td>
<td>YS</td>
</tr>
<tr>
<td>60 h</td>
<td>18.770</td>
<td>21.804</td>
<td>18.795</td>
<td>21.847</td>
</tr>
<tr>
<td>120 h</td>
<td>15.640</td>
<td>20.616</td>
<td>15.544</td>
<td>20.492</td>
</tr>
<tr>
<td>180 h</td>
<td>13.260</td>
<td>19.295</td>
<td>13.319</td>
<td>19.370</td>
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<tr>
<td>SAC305-0.4La</td>
<td></td>
<td>YS</td>
<td>UTS</td>
<td>YS</td>
</tr>
<tr>
<td>60 h</td>
<td>13.180</td>
<td>17.180</td>
<td>13.180</td>
<td>17.180</td>
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<tr>
<td>120 h</td>
<td>10.820</td>
<td>15.738</td>
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<td>15.740</td>
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<td>180 h</td>
<td>9.2260</td>
<td>14.764</td>
<td>9.230</td>
<td>14.760</td>
</tr>
</tbody>
</table>

3. Effects of aging time and high strain rate on mechanical properties

Electronic components when subjected to shock and vibration may experience high strain rates of 1/s to 100/s (Lall et al., 2012). Therefore, properties of Pb-free solder alloys at high strain rates and high temperatures experienced by the solder joint during typical mechanical shock, drop-impact, and vibration are important to analyze. Furthermore, there is little discussion available in the previous studies for commonly used SAC305 and SAC305-0.4La solders at high strain rates. Therefore, experimental works in this study are to investigate the related behaviours of solders at strain rates higher than 1/s i.e., 25/s, 35/s, and 45/s, and similar tensile specimens as shown in Figure 1 are used. The stress-strain curves under different strain rates are shown in Figure 10 (a) – (f) and the extracted data is summarized in Table 5. The mechanical properties (YS and UTS) extracted from the stress-strain curve at different aging times and strain rates for SAC305 are given in Figure 10 (a)-(c) and for the SAC305-0.4La are shown in Figure 10 (d)-(f). Test results indicate that the YS of SAC305 for 60 h aged at a temperature of 150 ºC increases from 21.6929 N/mm$^2$ to 37.7236 N/mm$^2$ at a strain rate of 25/s.
and the UTS increases from 29.2434 N/mm$^2$ to 44.6641 N/mm$^2$. While at these same conditions the YS and UTS of SAC305-0.4La show lower values of 19.5563 N/mm$^2$ and 24.4140 N/mm$^2$, respectively. It is also noted that as the strain rate increases there is an increase in the YS and UTS of both alloys. Furthermore, as discussed in the previous section, thermal aging has an inverse relation with mechanical properties, therefore, when aging time is increased from 60 h to 180 h the values of YS and UTS consistently shows a lower value of both solder alloys i.e., SAC305 and SAC305-0.4La shown in Figure 10 and given in Table 6. These trends have shown that mechanical properties such as YS and UTS increase as the strain rate increase and decrease when there is an increase in the thermal aging time. The reason for decrease in YS and UTS due to increase in thermal aging is already discussed in the previous section. However, the reason for justifying the claim of higher values in YS and UTS at high strain rates is attributed to the fact that the creep deformation makes larger contribution to the strength reduction at lower strain rate as noted by (Shi et al., 1999). Therefore, it is logically expected that at higher strain rates, the creep deformation should be smaller which contributed to the high strength.
Figure 10: Stress strain curves under high strain rates at different aging times at 150ºC
The above two sections above discussed the influence of aging times at higher temperatures on the microstructure and tensile properties of the SAC305 and SAC305-0.4La. For the given solders of SAC305 and SAC305-0.4La the average IMCs can be determined using Eqs. (1) – (2) while, YS and UTS can be evaluated using Eqs. (1) – (6). For a certain aging time, the investigation of mechanical properties can be further extended at high strain and can be determined using the material property models given in Eqs. (7) – (10). Therefore, the effect of aging time and high strain rate on mechanical properties is considered concurrently to develop and satisfy the following models given in Eqs. (7) - (10).

\[ YS_{(SAC305)} = Q_1 + Q_2 \ln(t_g) + Q_3 \ln(\dot{\varepsilon}) \]  
\[ UTS_{(SAC305)} = R_1 + R_2 \ln(t_g) + R_3 \ln(\dot{\varepsilon}) \]  
\[ YS_{(SAC305-0.4La)} = Q_4 + Q_5 \ln(t_g) + Q_6 \ln(\dot{\varepsilon}) \]  
\[ UTS_{(SAC305-0.4La)} = R_4 + R_5 \ln(t_g) + R_6 \ln(\dot{\varepsilon}) \]  

Where \( t_g \) is the aging is time and \( \dot{\varepsilon} \) is the strain rate. The material constants (Q1, Q2, Q3 Q4, Q5 and Q6) for YS, and (R1, R2, R3 R4, R5 and R6) for UTS of both the solder alloys are given in Table 5.

Table 5: Material constants for YS and UTS at different aging time and high strain rate

<table>
<thead>
<tr>
<th>Solder Alloy</th>
<th>Material constants used in Eqs. (7) – (10)</th>
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<tbody>
<tr>
<td></td>
<td>Q1</td>
</tr>
<tr>
<td>SAC305</td>
<td>-42.773</td>
</tr>
<tr>
<td>SAC305-0.4La</td>
<td>Q4</td>
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<tr>
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<td>-27.582</td>
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These equations are valid to predict the values of YS and UTS for the given values of aging time at high strain rates. The predicted values computed from Eqs. (7) – (8) for the both solder alloys give a minimum % error and are presented in Table 6.
Table 6: Experimental and predicted data of (YS and UTS) and % errors at different aging time and at high strain rates

<table>
<thead>
<tr>
<th>Aging time/Strain rate</th>
<th>Calculated values</th>
<th>Predicted values</th>
<th>% Error</th>
<th>YS</th>
<th>UTS</th>
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</tr>
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<tr>
<td>25/sec</td>
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<td>26.8890</td>
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<td>1.0644</td>
</tr>
<tr>
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</tr>
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<td>25.6550</td>
<td>33.6227</td>
<td>-1.8546</td>
</tr>
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</table>
3. 4. Microhardness determination

The measurement of microhardness is to examine the structural changes in association with mechanical properties. The higher the hardness leads to having maximum mechanical strength but with a compromise on ductility (Gain et al., 2011). Figure 11 shows the microhardness values of SAC305 and SAC305-0.4La solder alloy as a function of different aging times at 150°C. It is cleared that the hardness of SAC305-0.4La consistently displayed lower values than SAC305 due to the formation of bulk IMCs. The results depicted in Figure 11 also shows peak values of microhardness for SAC305 as 31 HB, while with 0.4 wt% La dose the value is decreased to 25 HB at 60 h aging time. These lower values of hardness are due to the heterogenous dispersion of coarse IMCs in the Sn-matrix. Furthermore, after thermal aging, the specimen became softer (lower HB value) and accordingly, the microhardness of electronic solder decreased as a result of increased aging time. The lowest values obtained for SAC305 and SAC-0.4La are 19 HB and 10 HB respectively at aging time of 180 h. In all three levels of the aging time, the hardness of un-doped and La-doped solder alloys is decreased. However, SAC305-0.4La is found to be more attractive to thermal aging time at high temperature. The percent increment of hardness of SAC305 at the aging time of 120 h with respect to 60 h is 16.13 % while the percent increment for La-doped SAC305 is recorded as 28%. A significant decrease in the microhardness of SAC305 up to 26.92% is observed at 180 h aging time respectively, at a high temperature of 150°C. However, in contrast to SAC305-0.4La, the percent increment of hardness of thermally aged La-doped alloy is found to be significantly high i.e., 44.44%.
4. Conclusions

In this study, the properties of SAC305 and SAC305-0.4La is investigated and compared at different aging times of 60 h, 120 h and 180 h at a high temperature of 150°C. The results showed that at high temperature as the thermal aging time increases, the IMC particles of both the solder alloys grow together and form bulk IMCs which coarsen the microstructure. This coarsening of the microstructure results in a recognizable softening effect in both SAC305 and SAC305-0.4La after thermal aging which caused a rapid decrease in the microhardness. In the same manner, a decrease in the yield strength and ultimate tensile strength is noted as the aging time increases. However, 0.4 wt% La doped solder alloy showed lower hardness, yield strength and ultimate tensile strength in comparison with SAC305, and is found to be more sensitive due to the non-uniform distribution of IMCs because of its high supplement (0.4 wt%). Based on these investigations, it is also expected that 0.4 wt% La concentrations should have minimum ductility, toughness and fatigue as compared to the original SAC305. Furthermore, when the tensile specimens are tested at high strains after thermal aging time and at high temperature, two trends were noted showing that the yield strength and ultimate tensile strength of both solder alloys increase as the strain rate increases which are likely due to less creep deformation, and decrease when there is an increase in the thermal aging. In addition to experimental study, empirical material property correlations have been developed for the
prediction of IMCs and mechanical properties at various aging times and strain rates. These proposed simplified mathematical relations provide a good fit of predicted values with experimental findings with a minimum % error and can be easily used when the actual test data are not available.

This work should be further extended to get the optimal La inclusion into SAC305 at high aging times at elevated temperature and at high strain rates in order to get a well refined microstructure with enhanced mechanical properties for the reliability of the solder joints. Also, the mechanical behaviour studied here shall be supplemented with the numerical study by exploiting the models specialized for strain rate effects and aging time available in commercial FEM codes.

Conflicts of interest

The authors declare that they have no conflict of interest.

References


An, T. & Qin, F. (2014). "Effects of the intermetallic compound microstructure on the tensile behavior of Sn3. 0Ag0. 5Cu/Cu solder joint under various strain rates". Microelectronics Reliability, 54, 932-938.


Figure 1: Tensile test specimen
Figure 2: SAC305 (a) SEM image (b) SEM images analyzed in ImageJ (c) EDX
Figure 3: SAC305-0.4La (a) SEM image (b) SEM images analyzed in ImageJ (c) EDX
Figure 4: SEM images analyzed in ImageJ at different aging times at 150 °C
Figure 5: Average IMCs particles size vs. aging time
Figure 6: Solder joints subjected to tensile loading due to substrate flexing (Sadiq, 2012)
Figure 7: Stress strain curve of SAC305 at different aging time at 150ºC
Figure 8: Stress strain curve of SAC305-0.4La at different aging time at 150ºC
Figure 9: Fractography analyses of tensile test at different aging time at 150 ºC
Figure 10: Stress strain curves under high strain rates at different aging times at 150ºC
Figure 11: Microhardness values before and after thermal aging

- SAC305
- SAC305-0.4La

Data points for different aging times (60 h, 120 h, 180 h) are shown in the graph.
### Table 1: Material property constants of average IMCs particle size

<table>
<thead>
<tr>
<th>Solder Alloy</th>
<th>Material constants used in eqs. (1) - (2)</th>
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</tr>
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Table 2: Experimental and predicted data of average IMCs particle size and % errors at different aging time

<table>
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<th>Solder alloy</th>
<th>Thermal aging time at 150 ºC</th>
<th>Average IMCs particle size (µm)</th>
<th>% Errors</th>
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Table 3: Material constants for YS and UTS at different aging time

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Table 4: Experiment and predicted data of (YS and UTS) and % errors at different aging time

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<th>Strain rate</th>
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<th>Experimental values</th>
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<th>% Error</th>
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<td>YS</td>
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<td>YS</td>
<td>UTS</td>
</tr>
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Table 5: Material constants for YS and UTS at different aging time and high strain rate

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Table 6: Experimental and predicted data of (YS and UTS) and % errors at different aging time and at high strain rates

<table>
<thead>
<tr>
<th>Aging time/Strain rate</th>
<th>Calculated values</th>
<th>Predicted values</th>
<th>% Error</th>
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