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INFLUENCE OF THERMOMECHANICAL PROCESSING ON MICROMECHANICAL PROPERTIES OF SN-0.7CU SOLDER ALLOY VIA NANOINDENTATION APPROACH

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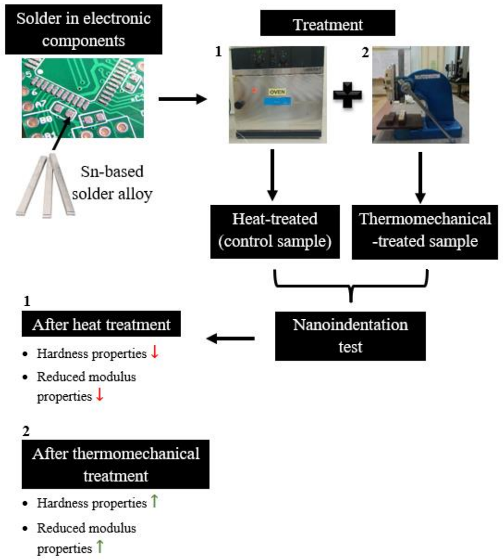
Graphical abstract



Abstract

Sn-based solder alloys are commonly utilized in electronic packages as an interconnection. In this study, nanoindentation was used to explore the impact of thermomechanical processing on the micromechanical characteristics of Sn-0.7Cu solder alloy. Bar-shaped Sn-0.7Cu solder alloy was cut into 9 cubic-shaped samples with dimension of 6 mm (l) X 6 mm (w) X 10 mm (h). First, cubic-shaped Sn-0.7Cu solder alloys was heat-treated for 20 minutes at temperatures of 30°C, 90°C, and 150°C, followed by compression until the thickness reduced to 40% and 80% and quick quench in water medium. As a control, solder alloys without compression process are employed. The result shows that thermomechanical-processed samples with 80% thickness reduction have the least hardness changes along temperature increment. These values were 6.46 MPa (from 30°C to 90°C) and 23.73 MPa (from 90°C to 150°C), respectively. The smaller gap of values when temperature increased were obtained through formation of new recrystallize grains which also referred as grain refinemet. The reduced modulus for thermomehanical-procesed sample with 80% thickness reduction sample also showed the same trend as the hardness value. The value dropped from 56.22 GPa to 42.12 GPa before rising slightly to 48 GPa as the temperature increased. The change of reduced modulus values were lower when compared to control and thermomechanical-processed sample with 40% thickness reduction samples. The findings demonstrate that as the temperature rises, thermomechanical processing with an 80% thickness reduction stabilizes the micromechanical properties of the Sn-0.7Cu solder alloy.

Keywords: Hardness, nanoindentation, reduced modulus, solder alloy, thermomechanical processing, thickness reduction

Abstrak

Aloi pateri berasaskan timah (Sn) sering digunakan dalam pempakejan elektronik sebagai antarasambungan. Dalam kajian ini, kaedah pelekukan nano digunakan untuk meneroka kesan pemprosesan termomekanikal terhadap ciri mikromekanikal aloi pateri Sn-0.7Cu. Bar aloi pateri Sn-0.7Cu dipotong kepada 9 sampel berbentuk kiub dengan ukuran 6 mm (p) X 6

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mm (l) X 10 mm (t). Aloi pateri Sn-0.7Cu berbentuk kiub dirawat secara haba selama 20 minit pada suhu 30°C, 90°C dan 150°C, diikuti dengan pemampatan dengan pengurangan ketebalan 40% dan 80% dan pelindapan di dalam medium air. Sebagai kawalan, aloi pateri tanpa proses mampatan digunakan. Hasil kajian menunjukkan bahawa sampel selepas proses termomemaknikal dengan pengurangan ketebalan 80% mempunyai perubahan kekerasan paling kecil pada suhu yang lebih tinggi. Nilai ini ialah 6.46 MPa (dari 30 ℃ hingga 90 ℃) dan 23.73 MPa (dari 90 ℃ hingga 150 ℃). Jurang perubahan nilai kekerasan yang kecil apabila suhu semakin meningkat diperolehi melalui pembentukan butiran baru atau dirujuk sebagai perhalusan butiran. Modulus terkurang untuk sampel selepas proses termoemkanikal dengan 80% pengurangan ketebalan juga menunjukkan trend yang sama dengan nilai kekerasan. Nilai modulus terkurang menyusut daripada 56.22 GPa kepada 42.12 GPa sebelum meningkat pada julat yang kecil kepada 48 GPa apabila suhu meningkat. Perubahan nilai modulus terkurang bagi sampel ini adalah lebih rendah jika dibandingkan dengan sampel kawalan dan sampel selepas proses termomekanik dengan 40% pengurangan ketebalan. Penemuan ini menunjukkan bahawa dengan peningkatan suhu, pemprosesan termomekanikal dengan 80% pengurangan ketebalan menstabilkan ciri mikromekanikal aloi pateri Sn-0.7Cu.

Kata kunci: Aloi pateri, kekerasan, modulus terkurang, pelekukan nano, pengurangan ketebalan, proses termomekanikal,

1.0 INTRODUCTION

In electronic packaging, solder alloys have been widely used as interconnection materials. Lead solder alloys such as Sn-Pb are widely used due to their advantageous properties. However, due to the toxicity of lead compounds, the usage of Sn-Pb in electronic packaging has been discontinued due to environmental and human health considerations [1, 2]. Nowadays, lead-free solder alloys such as Sn-Cu, Sn-Bi, Sn-In, and Sn-Zn have recently emerged as viable alternatives to lead solder alloys [3,4]. Many studies have been done to look at the potential of lead-free solder alloys in terms of mechanical properties [4–7]. The properties of solder materials continue to be improved over time to address challenges due to continuous advances in electronic packaging technology towards size reduction and multifunction devices [8, 9]. These developments result in solder joints not only being responsible in ensuring effective electrical current and conductivity connections, but also needing to have high mechanical strength to maintain good performance in the long run. Based on the studies that have been done, changes in the properties of solder alloys depend on the process used and the micromechanical changes that occur due to the process. Therefore, it is critical to investigate the potential of lead-free solder alloys when subjected to a thermomechanical treatment in order to better understand the relationship between their properties and the process.

Thermomechanical process is a procedure that combines heat treatment with a mechanical process to create plastic deformation with the goal of altering the microstructure and improving the properties of the materials involved [10]. This process is frequently used as a structural application material in the construction and automotive industries, which demands structural materials with excellent mechanical properties in order to be employed throughout time [11]. Through thermomechanical process, the mechanical properties of the material can be manipulated by regulating the temperature and applying simultaneous mechanical loads. This statement is supported by a study of Nb-Ti micro alloy steel who showed that thermomechanical processes through hot rolling technique produce higher tensile strength than those not through hot rolling [12]. In addition, this process also cause deformation to the material after being subjected to certain temperatures and types of loads such as compression. Compression loads will give different thickness reductions to the material and give different effects on mechanical properties. For example, a studies on wire formation through thermomechanical process using mechanical processes at different reduction percentages have been conducted [13]. According to the study, hardness alloy steel increased at a higher percentage of thickness reduction due to grain refinement and more efficient dislocation density. As a result of the interdependence of these two components, a related understanding of properties and process relationships is critical in a study.

To investigate such interactions, small-scale characterisation of solder materials is required to assess the reliability of electronic packaging. Previous research used tensile tests, impact tests, Vickers tests, tensile tests, and shear tests to determine the mechanical properties of solder alloys [14]. By making indentations on the solder and substrate areas, the Vickers method was used to investigate the mechanical properties of SAC solder alloys [15]. The method, on the other hand, is a traditional method that can only provide mechanical properties in bulk. As an outcome, the nanoindentation method is used to obtain local mechanical properties [16]. Nanoindentation is a technique for characterising mechanical properties on small scale materials without causing material damage. This method also enables precise load, depth, and test position control. The load versus depth curve can be used to collect information on mechanical properties as well as distortions in small scale constructions. For example, the nanoindentation method was used to investigate the mechanical properties of intermetallic compounds (IMC) at the Sn-3.0Ag-0.5Cu/Cu solder connection interface [17]. The hardness and elastic modulus of IMC Cu_3Sn and Cu_6Sn_5 can be determined as a result of the research. Hence, this research used a nanoindentation approach to investigate the effect of thermomechanical processing on the mechanical properties of Sn-0.7Cu solder alloy.

2.0 METHODOLOGY

A commercial bar-shape Sn-Cu solder alloy with 99.3% tin (Sn) and 0.7% copper (Cu) was provided from RedRing Solders (M) Sdn. Bhd. A 28.6 cm long bar of Sn-0.7Cu solder alloy was cut into nine samples with dimensions of 6 mm (l) X 6 mm (w) X 10 mm (h). The samples were each treated to a 20-minute heat treatment in an oven at 30°C, 90°C, and 150°C. The samples were then compressed using a push-pull gauge until the thickness was reduced to 6 mm (40% thickness reduction) and 2 mm (80% thickness reduction) from 10 mm, and then rapidly cooled in a water medium. In Figure 1, the sample thickness (h) has been lowered from 10 mm to 2 mm, a reduction of about 80%. Control samples are samples that have not been compressed.

The thermomechanical processed Sn-0.7Cu samples are clamped using sample holder and placed in the mould container's centre. Cold mounting material was made with a 2:1 ratio of hardener resin powder and epoxy resin liquid, resulting in 20 g of hardener resin powder and 10 g of epoxy resin liquid. Both materials were combined in a polystyrene cup and gently stirred for 30 seconds to ensure a homogeneous solution. The solution is then poured into a mould containing the sample and allowed to harden at room temperature for three to four hours. The sample-filled cold mounting is taken from the mould container. Next, the samples were then grind using a Buehler grinding machine, starting with a coarse silicon carbide (SiC) paper grade of 1200 grit, progressing to 2000 grit, and finally 4000 grit (finest grade). The grinding operation is performed at a rotational speed of 50-70 rpm, with a continuous flow of water to clear the grind residue from the sample. After that, the samples were polished with a polishing cloth and a 6 μm and 1 μm diamond spray. The nanoindentation test was used by using Micro Materials Nanotest™ to determine mechanical parameters such as maximum depth, hardness, and reduced modulus. The maximum load was 10 mN, and the loading and unloading rates were 0.5 mN/s, with a dwell time of 540 seconds. Dwell time of 540 s was chosen because it produced the most perfect Berkovish concave shape indentation. This test records the loading and unloading curves when load (P) is plotted against indentation depth (h), also known as P-h profile as shown in Figure 2. In the nanoindentation test, the hardness properties were obtained from the P-h profile using the Oliver-Pharr method as per equation below:

$$\mathrm{H} = \frac{P_{max}}{\mathrm{A_c}} \tag{1}$$

where H is the hardness value of the material in units of MPa, P_{max} the maximum load applied to the material and A_c is the contact area. In addition to the hardness properties, the reduce modulus (E_r) can also be obtained through the curve P against h. The calculation for E_r is as in equation below:

$$\frac{1}{E_r} = \frac{(1-v_s^2)}{E_s} - \frac{(1-v_i^2)}{E_i} \tag{2}$$

where E_s and v_s are the Young's modulus and the Poisson's ratio for the sample, while E_i and v_i are the Young's modulus and Poisson's ratio of the indenter.

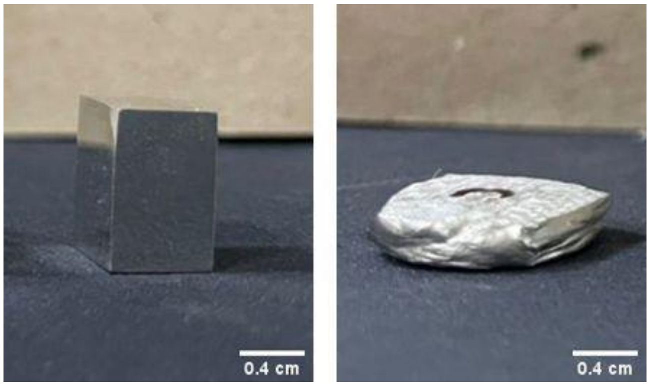


Figure 1 Sn-0.7Cu sample (a) before and (b) after thermomechanical process at 150°C with 80% thickness reduction

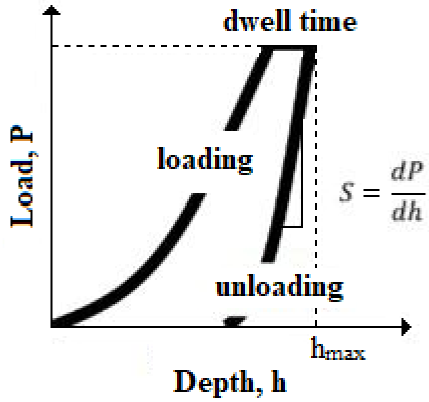
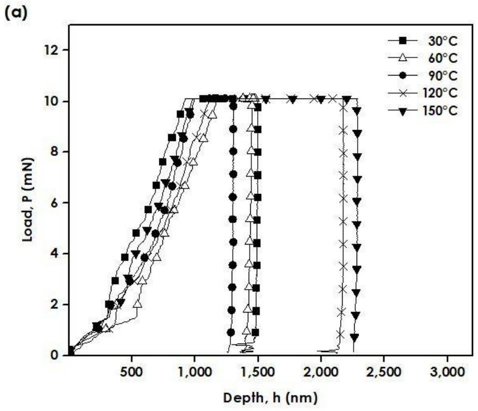
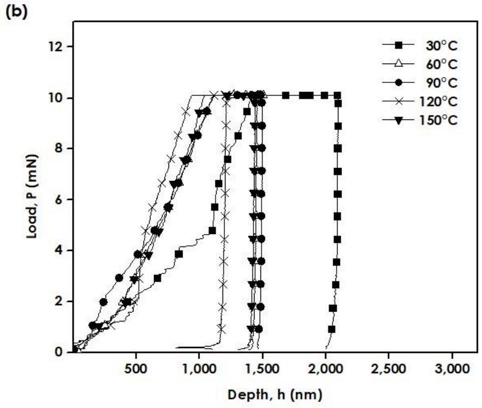


Figure 2 Schematic of the load, P against depth, h during nanoindentation test

3.0 RESULTS AND DISCUSSION

The P-h profile for Sn-0.7Cu samples after heat treatment and thermomechanical process are shown in Figure 3. The y-axis denotes P, whereas x-axis denotes h. During the nanoindentation test, P is applied to the indenter, causing the indenter tip to penetrate from the surface into the sample structure. As P grows, h increases as well, until the maximum load (P_{max}) of 10 mN is attained. The indenter tip is left static for 540 seconds at maximum load, which is referred to as dwell time [18]. Following the end of the dwell time, P begins to drop (called unloading), and h falls as well, but at very slow rate. Each sample has a different trend for h based on the P-h profile, which is influenced by changes in micromechanical properties.





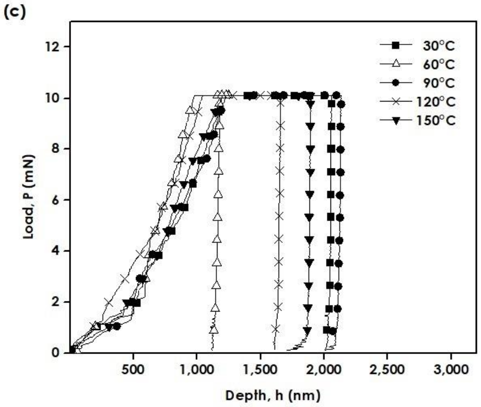


Figure 3 P-h profile for (a) controlled and thermomechanical processed samples with (b) 40% and (c) 80% thickness reduction

Figure 4 shows the hardness of Sn-0.7Cu samples. The hardness of control sample increase from 180.75 MPa (30℃) to 236.14 MPa (90℃), before drastically decrease to 78.52 MPa at 150℃. The hardness of the control sample at 150°C is clearly the lowest of all samples. These values were respectively 2.5× and 1.5× lower than the hardness of thermomechanical-processed sample with 40% and 80% thickness reduction at the same temperature. The trend of the result is same as previous study conducted on SAC305 solder wire that were heat-treated at temperature range of 25-200°C [6]. The results show that increasing temperature caused negative properties of hardness of SAC305 solder wire. This situation occur due to softening effect, which enable indenter tip to penetrate the sample structure from the surface more easier [8,9,19,20]. However, thermomechanical-processed samples produced a different trend from control sample. Hardness for thermomechanical-processed sample with 40% thickness reduction continuously increased about 95% and 9% throughout temperature increased. Hardness for thermomechanical-processed sample with 80% thickness reduction also show increasing trend along temperature increment which are about 7% and 18%, but it exhibited smallest value change when compare to thermomechanical-processed sample with 40% thickness reduction. The change of hardness value for thermomechanical-processed sample with 40% thickness reduction were 87.27 MPa (from 30°C to 90°C) and 16.61 MPa (from 90°C to 150°C). For thermomechanical-processed sample with 80% thickness reduction, much lower of hardness value changes were obtained which are 6.46 MPa (from 30°C to 90°C) and 17.27 MPa (from 90°C to 150°C). The lower change in hardness value is due to an increase of dislocation density in the structure [21]. During thermomechanical process at 30°C with 80% thickness reduction, newly created dislocation was formed and continuously added in the microstructure which caused high dislocation density. This high density dislocation will accumulated at the grain boundary and act as nucleation site to form low-angle grain boundary (LAGB) or subgrain [22]. Further increase of temperature up to 150°C has led to subgrain transform into high-angle grain boundary (HAGB) or new recrystallize grain [23]. The formation of new recrystallize grain have resulted grain refinement which increase the hardness of Sn-Cu solder alloy up to 114.34 MPa. This result was supported by a study on SAC305 impact response at different strain rates. In the study, they claimed release of heat rapidly during loading at high strain rates aided in the refinement of grains [24].

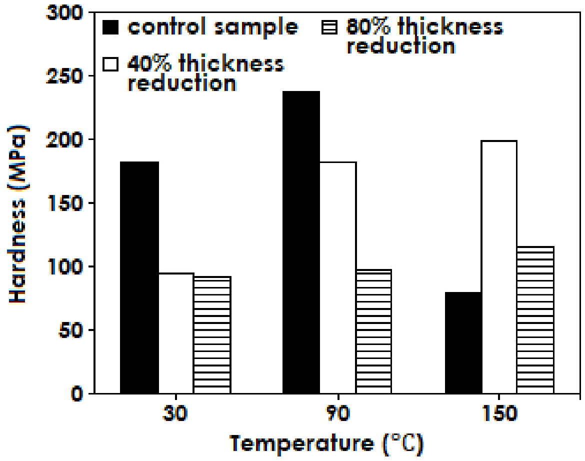


Figure 4 Hardness for Sn-0.7CU samples at various temperature and thickness reduction

The hardness of a material determines its resistance to plastic deformation [25]. In a nanoindentation test, hardness is defined as the sample's ability to withstand the indenter as it penetrates the sample's surface when a load is applied [26]. This explains how a decrease in indentation depth affects the increase in Nanohardness. Zhou *et al.*, (2020) backed up this claim by stating that nanoindentation hardness is strongly impacted by nanoindentation depth [27]. Based on Figure 5, thermomechanical-processed sample with 40% thickness reduction have decreasing trend of maximum depth when temperature increase which were 2096.65 nm, 1496.49 nm and 1439.88 nm. These values are inverse with their hardness values that increase along the temperature increment as shown in Figure 4. This is because softening occurs as a result of microstructural changes, which are impacted by thermodynamic activities such as temperature (19).

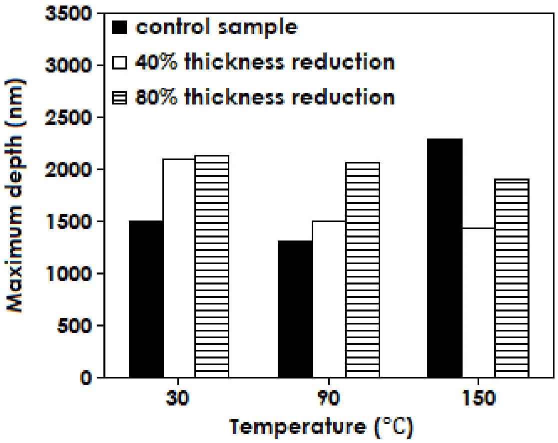


Figure 5 Maximum depth for Sn-0.7Cu samples at various temperature and percentage of thickness reduction

In comparison to hardness, the reduced modulus of Sn-0.7Cu solder alloy shows various fluctuations with increasing temperature and thickness reduction, as illustrated in Figure 6. From the figure, it is exhibited that sample after thermomechanical process with 80% thickness reduction have steady value of reduced modulus with temperature increment compared to that of control and 40% thickness reduction. The steady value expressed in this study is a small reduced modulus value gap from one temperature to another. The reduced modulus value gap for thermomechanical-processed sample with 80% thickness reduction were 14.1 GPa (approximately 25% changes) from 30℃ to 90℃, and 5.88 GPa (approximately 14% changes) from 90℃ to 150℃. On the other hand, thermomechanical-processed sample with 40% thickness reduction produced value gap of 50.65 GPa (approximately 93% changes) and 52.52 GPa (approximately 51% changes), respectively. Reduced modulus value is more related to intrinsic properties than microstructure [16]. This significant value changes for thermomechanical-processed sample with 40% thickness reduction may be due to abrupt changes in intrinsic properties or crystallographic orientation after thermal treatment. This discussion is supported by a study from Sun *et al.*, (2018) where high entropy alloys (HEAs) form FCC phases when the reduced modulus value decreases sharply [28]. The steady value of reduced modulus for thermomechanical-processed sample with 80% thickness reduction indicated there is slightly or no changes of intrinsic properties.

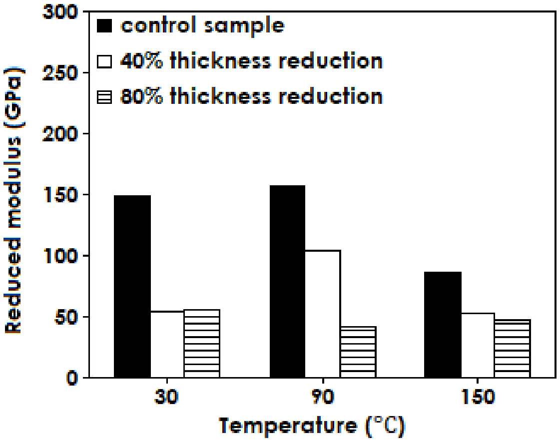


Figure 6 Reduced modulus for Sn-0.7Cu samples at various temperature and percentage of thickness reduction

Through the micromechanical analysis using nanoindentation approach, Sn-0.7Cu solder alloy that underwent thermomechanical process with 80% thickness reduction pronounced the most stable properties among the samples. Hence, the relationship thermomechanical process-micromechanical properties of solder alloy are valid which is very helpful to predict its properties.

4.0 CONCLUSION

The nanoindentation approach has successfully characterized the localized micromechanical properties of Sn-0.7Cu solder alloy subjected to thermomechanical process with variation of thickness reduction and temperature. The findings show that the thermomechanical-processed sample with 80% thickness reduction having lowest changes of hardness and reduced modulus. The change of hardness values for thermomechanical-processed sample with 80% thickness reduction were 6.46 MPa (about 7%) and 17.27 MPa (about 18%) as the temperature increased to 150°C. The reduced modulus for the same sample also shows lower change in values, which are 14.1 GPa (approximately 25% changes) and 5.88 GPa (approximately 14% changes) throughout temperature increment up to 150°C. The small change of micromechanical properties indicates thermomechanical-processed sample with 80% thickness reduction have more stabilize due to formation of new recrystallize grains or also referred as grain refinement. This study shows that thermomechanical processing is able to modify the micromechanical properties of the solder materials for electronics packaging application.

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