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# TEMPERATURE-INDUCED THICKNESS REDUCTION OF MICROMECHANICAL PROPERTIES OF Sn-0.7Cu SOLDER ALLOY

## TEMPERATURNO INDUCIRANO ZMANJŠANJE DEBELINE IN MIKROMEHANSKIH LASTNOSTI ZLITINE ZA SPAJKANJE Sn-0,7Cu

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Solders are used in electronic packaging for metallurgical interconnections. Thermomechanical methods are used to modify the properties of a material. Cubic Sn-0.7Cu solder alloy was subjected to heat treatment at 30–150 °C for 20 min, followed by 80 % compression. The control samples used in this study were only subjected to heat treatment. This study used the nanoindentation approach to investigate the reductions in the modulus and hardness of the lead-free Sn-0.7Cu solder alloy after thermomechanical treatment. Samples with 80 % compression showed slight changes in the reduced modulus (approximately 24 %) and hardness (approximately 14 %) after thermomechanical treatment. In contrast, the solder alloy that underwent heat treatment alone (the control sample) showed shifts in the hardness and reduced modulus of approximately 54 % and 66 %, respectively. The production of new recrystallized grains resulted in smaller changes in the micromechanical properties. These findings demonstrated that thermomechanical treatment can both modify and stabilize the properties of the Sn-0.7Cu solder alloy, such as micromechanical properties.

Keywords: micromechanical properties, solder alloy, nanoindentation, thermomechanical treatment

Spajkanje se uporablja pri elektronskem pakiranju medsebojnih metalurških povezav. Izbrane termomehanske metode se pogosto uporabljajo za modifikacijo lastnosti materiala. Predmet raziskave je bila zlitina za spajkanje Sn-0,7Cu v obliki kvadra. Spajko so toplotno obdelovali 20 min v temperaturnem območju med 30 °C in 150 °C v korakih po 30 °C, nato je sledila njena 80 % tlačna deformacija. V raziskavi so uporabili le toplotno obdelane kontrolne vzorce. Za ugotavljanje učinka toplotne obdelave so uporabili metodo nanoindentacije in ugotavljali zmanjšanja modula in trdote spajke Sn-0,7Cu, ki ni vsebovala ekološko spornega svinca (Pb). Vzorci, ki so bili 80 % tlačno deformirani so po termomehanski obdelavi kazali rahlo spremembo oziroma zmanjšanje modula elastičnosti (približno 24 %) in trdote (približno 14 %). V nasprotju s kontrolnim vzorcem, ki ni bil toplotno obdelan se je pokazal obrat v smeri zmanjšanja modula elastičnosti in trdote za približno 54 % oz. 66 %. Nastanek novih rekristaliziranih zrn je povzročil majhno spremembo mikromehanskih lastnosti. Ugotovitve in raziskave avtorjev tega članka so pokazale, da termomehanska obdelava lahko spremeni in stabilizira mikromehanske lastnosti spajke Sn-0,7Cu.

Ključne besede: mikromehanske lastnosti, zlitina za spajkanje, nanoindentacija, termomehanska obdelava

## 1 INTRODUCTION

Temperature is one of the factors that affect the mechanical properties of a material by altering its microstructure. Heat treatment, such as aging at relatively high temperatures, is well appreciated as an effective technique for changing the microstructures of materials, resulting in changes in their mechanical performances.[1] Thermomechanical treatment is similar to heat treatment, but it includes a mechanical action such as compression to change the microstructure and characteristics of the material.[2] This treatment is commonly utilized for the structural materials used in the automotive and construction industries, which must exhibit high mechanical properties and long-term reliability.[3] The microstructure of the affected material is regulated by simultaneously varying the temperature and applying a mechanical load to the material. This can produce microstructural changes such as grain-size refinement and an increased dislocation density. It has been proven that refining its grains improves a material's mechanical properties, including its hardness and tensile strength. Li et al. reported that the application of hot rolling to Nb-Ti steel led to a higher tensile strength than that without hot rolling.[4] However, thermomechanical treatment at specific temperatures and forms of stress such as compression, tension or shear can cause a material to deform. Compression loads cause a reduction in the material thickness and have varying effects on the mechanical characteristics. For example, a study was conducted on the wire formation using thermomechanical treatment with mechanical processes at different reduction percentages.[3] The results revealed that the hardness increased at a higher proportion of reduction, which was influenced by a grain refinement and more efficient dislocation density. As a result, it is critical to understand the relationships be-

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tween various processes and material properties because they are linked.

In electronic packaging, solder alloys are frequently employed as the connecting materials. Because of their beneficial features, lead solder alloys such as Sn-Pb used to be particularly popular. However, for environmental and human health reasons, the use of Sn-Pb in electronic packaging is no longer permitted because of the toxicity of lead.[5] Nowadays, lead-free solder alloys such as Sn-Cu, Sn-Bi, Sn-In and Sn-Zn are potential candidates to replace lead solder alloys.[6] Many studies investigated the potentials of lead-free solder alloys in terms of their mechanical properties and reliability. The properties of solder materials continue to improve over time to meet the challenges posed by continuous advances in the electronic packaging technology, which aims at miniaturization and multi-functionality to ensure the solder joint reliability.[7] These developments mean that solder joints are not only responsible for ensuring effective electrical-current connections with sufficient conductivity but also need to a have high mechanical strength to ensure good performance in the long term. For these studies, changes in the solder alloy properties depend on the process and the micromechanical changes that occur as a result of the process. Therefore, it is vital to study the potentials of the lead-free solder alloys subjected to a thermomechanical treatment to better understand the relationship between their properties and the process.

Small-scale mechanical property characterization of solder materials is necessary because of the miniaturization of solder joints and understanding of the reliability of electronic devices. Microhardness tests, shear tests, impact tests, Vickers tests, bending tests and tensile tests were all used in the past to assess the mechanical properties of solder alloys.[8] Giuranno et al. studied the mechanical properties of SAC solder alloys using the Vickers method.[9] Indentations were made in the region of the solder alloy and substrate. However, this conventional method can only determine the mechanical properties of bulk materials. In contrast, the nanoindentation method can determine the mechanical properties locally.[10] Nanoindentation is a widely used method to characterize the mechanical properties of small structures without damaging the sample. This method also makes it possible to control the load, depth and exact test position. The mechanical properties and deformations can be obtained from a load versus depth curve. For example, the mechanical properties of intermetallic compounds (IMC) at the interface of Sn-3.0Ag-0.5Cu/Cu solder joints were investigated using the nanoindentation method.[11] The results showed that the hardness, elastic modulus and creep properties of $Cu_3Sn$ and $Cu_6Sn_5$ could be determined. Therefore, this study investigated the effect of the thermomechanical treatment temperature on the microstructure and micromechanical properties of Sn-Cu solder alloys via nanoindentation.

## 2 EXPERIMENTAL PART

Cube-shaped Sn-0.7Cu samples, with dimensions of (6 × 6 × 10) mm (length × width × height), were individually subjected to heat treatment in an oven at (30, 60, 90, 120 and 150) °C for 20 min. Subsequently, the samples were compressed with a push-pull gauge until the thickness was reduced by 80 % of the original thickness, from 10 mm to 2 mm (**Figure 1**), and then quickly quenched in a water medium. Samples that were not exposed to compression tests (heat-treated) were used as control samples. Cross-sections of the Sn-0.7Cu alloy samples were obtained using a metallographic technique. The samples were cold-mounted in epoxy resin and cured for 3–4 h at room temperature. The cold mounting with a sample was removed from the mold container and subjected to a grinding process using silicon carbide (SiC) paper in grits of 800, 1000, 1200, 2000, and 4000.

Consequently, the samples were polished using a polishing cloth with 6-μm and 1-μm diamond sprays. The microstructure of each sample was captured using an infinite focus microscope (IFM) at a magnification of 50×. Nanoindentation tests were performed to determine the hardness and reduced modulus. The maximum load was 10 mN, the loading and unloading rate was 0.5 mN/s, and the dwell time was 180 s. Each sample underwent five nanoindentation tests and the average result was cal-

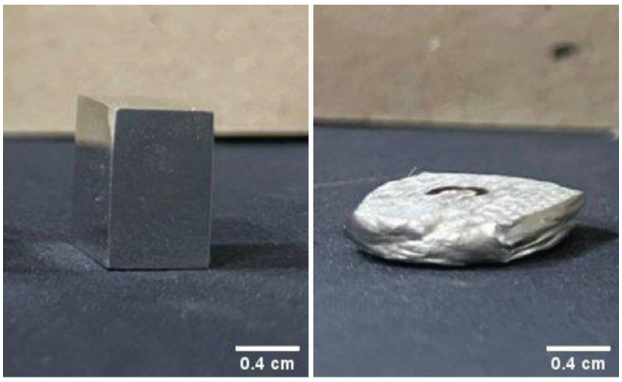


**Figure 1:** Sn-0.7Cu solder alloy before (left) and after compression (right)

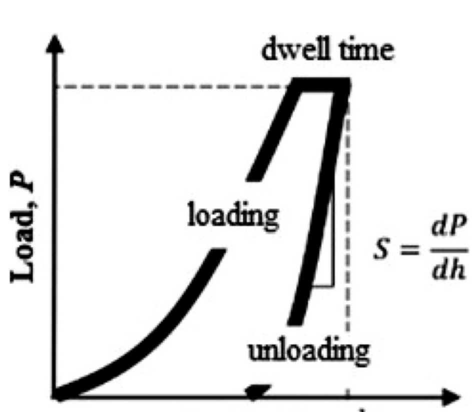


**Figure 2:** Load, *P*, against depth, *h*, during the nanoindentation test

culated. This test recorded the loading and unloading curves when the load ($P$) was plotted against the indentation depth ($h$) (**Figure 2**). During the nanoindentation test, hardness properties were obtained from the $P$-$h$ profile using the Oliver-Pharr method, as shown in Equation (1):

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

where $H$ is the hardness of the material, $P_{\max}$ (MPa) is the maximum load applied to the material, and $A_{\mathrm{c}}$ is the contact area. In addition to the hardness properties, the reduced modulus ($E_{\mathrm{r}}$) could also be obtained from the curve of $P$ against $h$. $E_{\mathrm{r}}$ was calculated using Equation (2):

$$\frac{1}{E_{\mathrm{r}}} = \frac{(1-v_{\mathrm{s}}^2)}{E_{\mathrm{s}}} - \frac{(1-v_i^2)}{E_i} \tag{2}$$

where $E_{\mathrm{s}}$ and $v_{\mathrm{s}}$ are Young's modulus and Poisson's ratio of the sample, respectively; $E_i$ and $v_i$ are Young's modulus and Poisson's ratio for the indentation, respectively.

## 3 RESULTS

**Figure 3** depicts the load-depth ($P$-$h$) profiles obtained with the nanoindentation tests of the Sn-0.7Cu solder alloy after it was subjected to different temperatures and treatments. During the micromechanical test, a load was applied using an indenter, which was referred to as the loading, causing the tip of the indenter to penetrate the surface and enter the structure of the solder alloy. During the load increment, the penetration depth was calculated until a maximum load of 10 mN was reached. At 10 mN, the indenter was left static for 180 s, which was called the dwell time. After 180 s, the indenter started to move out of the solder alloy structure (unloading), causing a decrease in the penetration depth. The variation in the penetration depth seen at different temperatures was caused by micromechanical property differences.

**Figure 4** shows that the hardness decreased with the increasing temperature. The hardness of the heat-treated solder alloy (the control sample) decreased from 320 MPa at 30 °C to 147 MPa at 150 °C. The thermomechanically treated solder alloy also showed the same hardness trend up to 90 °C, with a slight increase to 167 MPa at 150 °C. In general, the thermomechanically treated samples showed almost the same hardness trend as the control samples. This is known as softening, and it confirmed the hypothesis that the hardness of a metal material decreases as the temperature increases during heat treatment.[12–14] Several studies were conducted to evaluate this process and relate different temperatures to mechanical properties of solder alloys. Abdullah et al. performed tensile and nanoindentation tests with a Sn-3.0Ag-0.5Cu (SAC305) solder wire at a temperature range of 25–200 °C. They found that the yield strength, ultimate tensile strength and hardness decreased with an increase in the temperature at a given strain rate. They

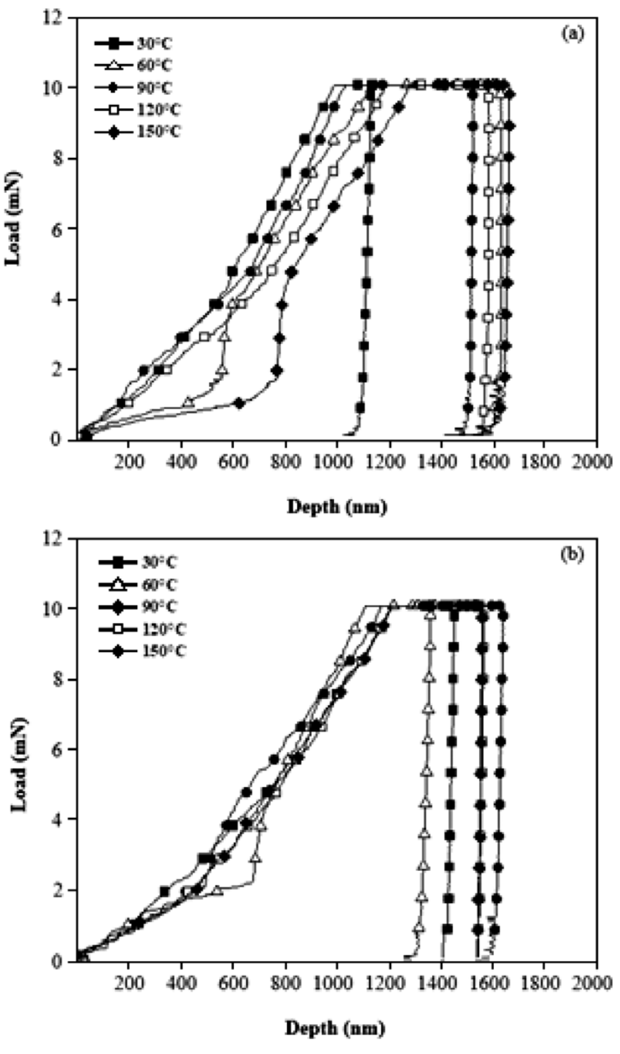


Figure 3: Load-depth graphs of Sn-0.7Cu solder alloy after: a) heat treatment, b) thermomechanical treatment

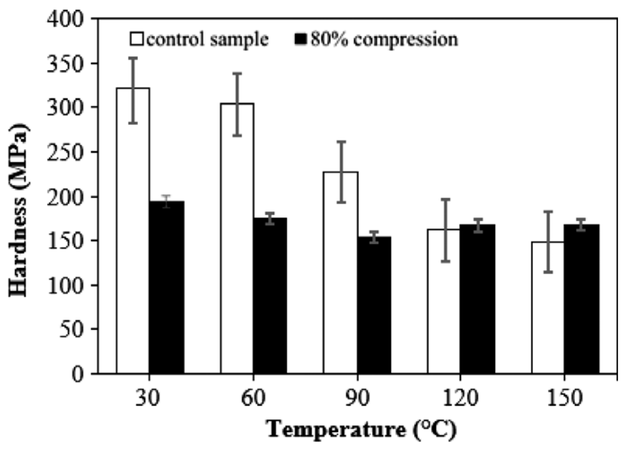


Figure 4: Hardness versus temperature for the control and thermomechanically treated solder-alloy samples

also stated that the reduction in the hardness was mainly caused by recrystallization due to the application of elevated temperatures.[15] **Table 1** lists the results of the studies on the mechanical properties of alloys at various temperatures.[1,16,17] According to the table, the tensile strength and ultimate tensile strength decreased as the temperature increased. These results show that our research was consistent with earlier alloy studies.

**Table 1:** Studies on mechanical properties of alloys at different temperatures

| Authors | Temperature (°C) | Tensile strength (MPa) | Ultimate tensile strength (MPa) |
|---|---|---|---|
| Tang, Long, and Yang[1] year 2000 | No annealing | 36.0 | – |
| | 75 | 35.0 | |
| | 150 | 27.0 | |
| | 210 | 27.3 | |
| | 230 | 18.9 | |
| Filizzolab et al.[16] year 2001 | 50 | 29.0 | – |
| | 100 | 30.0 | |
| | 150 | 28.7 | |
| | 200 | 28.0 | |
| | 250 | 20.0 | |
| | 300 | 22.0 | |
| Tang et al.[17] year 2020 | 100 | – | 479.2 |
| | 175 | | 461.6 |
| | 200 | | 457.8 |
| | 225 | | 451.3 |
| | 250 | | 444.3 |
| | 300 | | 422.6 |
| | 350 | | 379.4 |
| | 400 | | 376.7 |

However, a difference between the Sn-0.7Cu solder-alloy samples treated at 30 °C and 150 °C could be clearly observed, as shown in **Figure 5**. The thermomechanically treated samples showed an approximately 14 % difference between their hardness values, indicating a greater stability than that of the control samples, which showed an almost four times higher difference of 54 %.

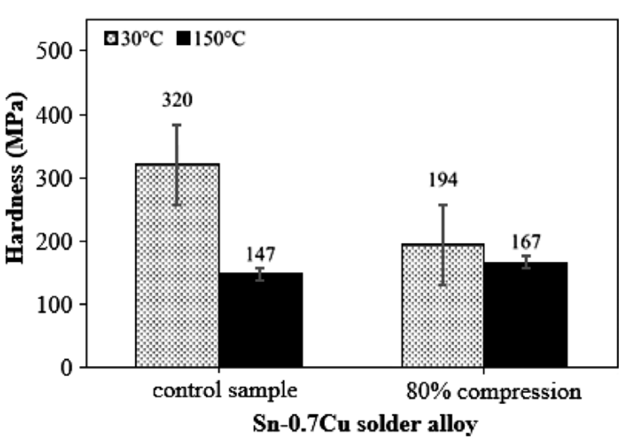


**Figure 5:** Comparison of the hardness values for Sn-0.7Cu solder-alloy samples treated at 30 °C and 150 °C

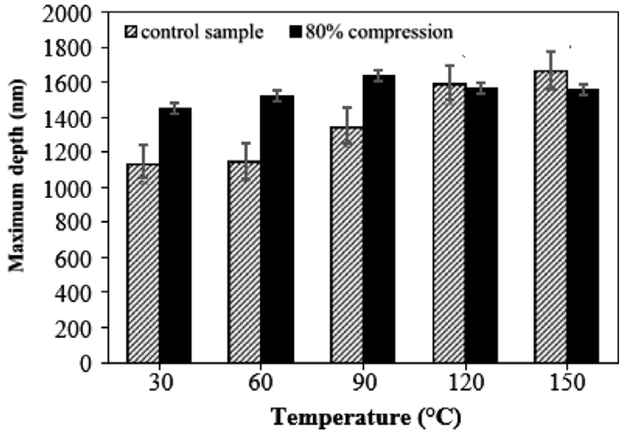


**Figure 6:** Maximum depth for the Sn-0.7Cu solder-alloy sample after heat treatment and thermomechanical treatment

Hardness is a measure of a material's resistance to plastic deformation.[18] The definition of hardness in a nanoindentation test is the resistance of a sample to the indenter as it penetrates the sample surface when a load is applied. This indicates that a decrease in the indentation depth signifies an increase in the localized hardness. The surface-modified layer of 18CrNiMo7-6 steel after case hardening was investigated with nanoindentation, and it was found that an increase in the hardness affected the nanoindentation depth.[19] The maximum depth results (**Figure 6**) in this study were consistent with the localized hardness properties as an increase in the hardness made it more difficult for the indenter tip to penetrate the surface of a sample. For instance, for a sample with 80 % compression and a hardness of 194 MPa, a maximum depth of 1451 nm was achieved, while a hardness value of 167 MPa resulted in a maximum depth of 1558 nm. This was due to the occurrence of softening caused by microstructural changes influenced by thermodynamic conditions such as temperature.[18]

**Figure 7** shows that the reduced modulus of Sn-0.7Cu decreased with the increasing temperature. The reduced modulus of the control sample showed more sig-

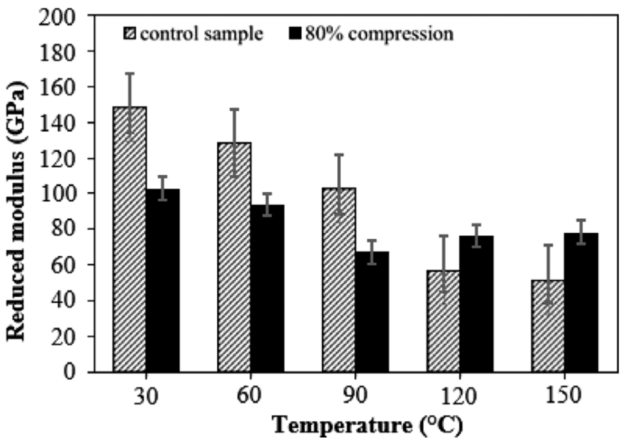


**Figure 7:** Reduced modulus against temperature for the Sn-0.7Cu alloy at different conditions

nificant changes than the samples with 80 % compression. The reduced modulus of the control sample was 149 GPa at 30 °C and it decreased to 51 GPa at 150 °C, which was a change of approximately 66 %. The reduced modulus of the Sn-0.7Cu samples with 80 % compression showed smaller changes of approximately 24 %, decreasing from 103 GPa at 30 °C to 78 GPa at 150 °C. According to one study, the reduced modulus is related to the intrinsic properties rather than the microstructure.[10] These significant changes of the control sample could have been due to abrupt changes in the intrinsic properties or crystallographic orientation after the thermal treatment. This conclusion is supported by a study on high-entropy alloys (HEAs), which form FCC phases when the reduced modulus drops sharply.[20] The small changes in the reduced modulus values for the Sn-0.7Cu solder alloy with 80 % compression indicated little or no change in the intrinsic properties.

## 4 DISCUSSION

The effect of microstructural evolution on the micromechanical properties of the Sn-0.7Cu solder alloy was examined through a microstructural examination. Different trends for the hardness values of the control samples and the thermomechanically treated samples were due to the formation recrystallization (RX) of the grains. **Figures 8** and **9** show IFM images of the samples after the heat and thermomechanical treatments at 30 °C and 120 °C, respectively. The microstructure of the solder alloy at 120 °C was selected as the starting point for hardening. The bright-field image shows $\beta$-Sn (tin-rich) phase grains, while the dark-field image shows $Cu_6Sn_5$ (eutectic) phase grains.[21] Before the heat treatment, the microstructure of the Sn-0.7Cu solder alloy had distinct $\beta$-Sn grains with large gaps between them (**Figure 8a**). A further increase in the temperature during the heat treatment resulted in an increase in the number of $\beta$-Sn grains (**Figure 8b**).

In general, during thermal activation such as heat treatment, the material under consideration tends to transform to a lower-energy state through a sequence of microstructural changes. There are three phases in this process: recovery, recrystallization and grain growth. Before the heat treatment, the Sn-0.7Cu samples contained defects such as dislocations, which were sparsely distributed in the microstructure. When the control sample was heat-treated at a higher temperature (120 °C), rapid recovery occurred. The extinction and rearrangement of dislocations occurred, some of them merged, leading to a

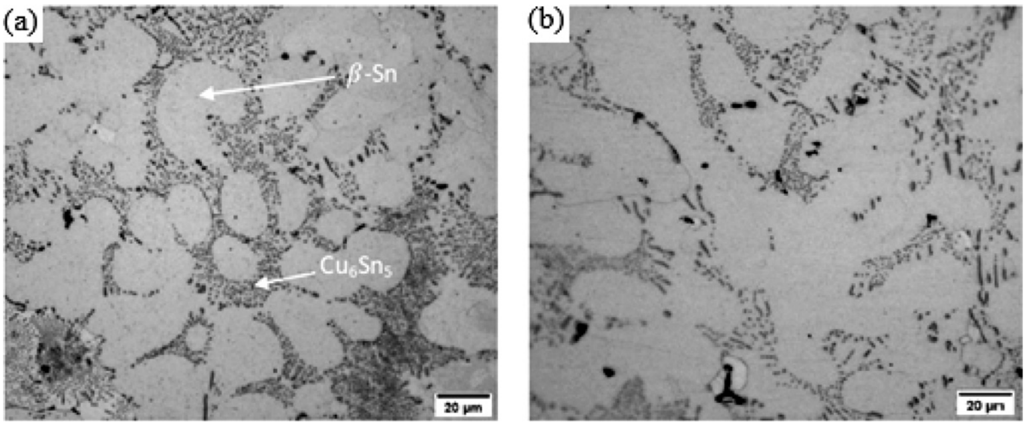


**Figure 8:** IFM images of Sn-0.7Cu after heat treatment at: a) 30 °C, b) 120 °C

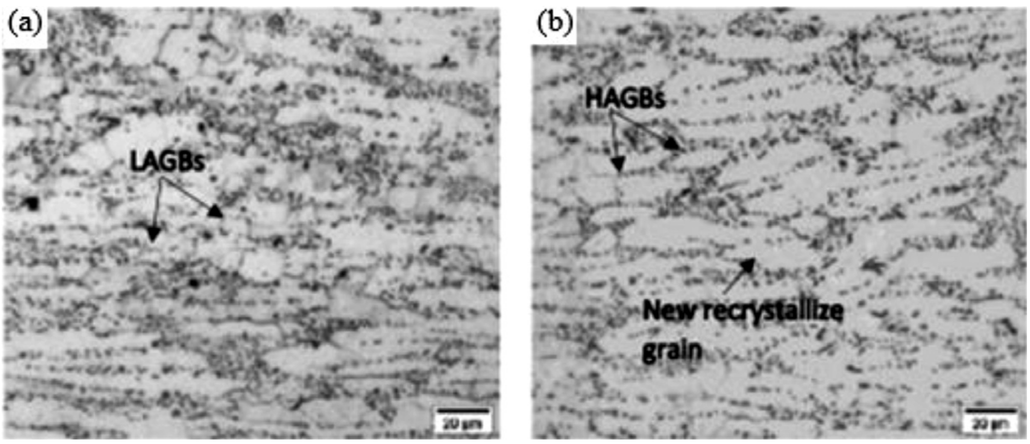


**Figure 9:** IFM images of Sn-0.7Cu after thermomechanical treatment at: a) 30 °C, b) 120 °C

decrease in the dislocation density. After the recovery was complete, nucleation occurred at the grain boundaries, and the grains began to grow to form a new grain structure. As the grains grew, the dislocations at the boundaries of the newly formed grains were obliterated. Consequently, the hardness of the Sn-0.7Cu sample decreased significantly, to 161 MPa at 120 °C.

Thermomechanical treatment with 80 % compression caused the $\beta$-Sn phase and $Cu_6Sn_5$ particles to be strongly deformed perpendicular to the compression direction, forming elongated $\beta$-Sn and $Cu_6Sn_5$, as shown in **Figure 9**. At 80 % compression, a new dislocation formed, which was trapped by the existing dislocations in the sample. Consequently, the sample became thermodynamically unstable. $Cu_6Sn_5$ particles, sparsely distributed in the microstructure, acted as the barriers to the movement of mobile dislocations, resulting in the accumulation of dislocations and a high dislocation density.[22] The thermomechanical treatment at the low temperature (30 °C) caused the highly accumulated dislocations to undergo cross-slip or rearrangement, resulting in the early formation of low-angle grain boundaries (LAGBs) or subgrains, as shown in **Figure 9a**. When the temperature was further increased to 120 °C during the thermomechanical treatment, these subgrain boundaries were retarded by the $Cu_6Sn_5$ particles, and dislocations were continuously included in these boundaries. Finally, the LAGBs transformed into high-angle grain boundaries (HAGBs), i.e., new recrystallized grains were formed (**Figure 9b**). The new recrystallized grains resulted in a more refined microstructure, increasing the hardness of the Sn-0.7Cu solder alloy to 166 MPa. In their work on SAC305, Long et al. claimed that the rapid release of heat during the loading or compression process aided the refinement of grains.[23]

The Sn-0.7Cu solder alloy subjected to thermomechanical treatment with 80 % compression displayed the best stability among the samples, according to a micromechanical investigation utilizing the nanoindentation method. Consequently, the thermomechanical link with the micromechanical properties of the solder alloy is genuine, which is particularly beneficial for predicting its quality.

## 5 CONCLUSIONS

The relationship between the thermomechanical treatment and mechanical properties of the Sn-0.7Cu solder alloy was successfully investigated using the nanoindentation approach. The results showed that the hardness and reduced modulus of the control sample decreased by approximately 54 % and 66 %, respectively. The solder alloy with 80 % compression showed minor changes in the localized hardness and reduced modulus of approximately 14 % and 24 %, respectively. These smaller changes in the micromechanical properties were associated with the formation of new recrystallized grains. These results suggest that thermomechanical treatment can alter the microstructure and micromechanical properties of Sn-0.7Cu alloys and help predict the potential and performance of solder alloy materials in the field of electronic packaging.


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