STRUCTURAL ANALYSIS ON NANOCOMPOSITES LEAD FREE SOLDER USING NANOINDENTATION

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ABSTRACT: As lead-based solder has been restricted, the microelectronics industry has been looking for lead-free solder like SAC. But there have been cases of solder joints failing, reducing product reliability because the formed intermetallic compound layer (IMC) is brittle. The thick IMC layer reduced the solder joint strength. This paper analyses real industrial package assemblies of nanocomposite lead-free solder joints. TiO_2, Fe_2O_3, and NiO nanoparticles with 0.05 weight percentage (wt.%) were mixed into 96.5% Sn-3.0% Ag-0.5% Cu (SAC305) solder paste using a mechanical stirrer to make nanocomposite lead-free solders. The effects of nanoparticles in miniaturized solder on joining quality in IMC layers and nanocomposite solders were studied using a scanning electron microscope (SEM) and a nanoindenter. Adding TiO_2, Fe_2O_3, and NiO nanoparticles changed the microstructure and reduced the IMC layer thickness by 29%-35%. The nanocomposite solder's hardness and elastic modulus are increased by 1%-11% and 8%-31%, respectively. In comparison to pure SAC305, the composition of SAC305 with NiO nanoparticles solder paste had the highest hardness, and with Fe2O3 had the highest elastic modulus. This proves that the incorporation of nanoparticles of TiO_2, Fe_2O_3, and NiO has enhanced the mechanical properties of pure SAC and increased the reliability of solder joints in miniaturised electronic packaging.

KEYWORDS: *Nanocomposite Lead Free Solder; Nanoindentation*

1.0 INTRODUCTION

Tin-lead (Sn-Pb) solder has been widely used in electronics over the last few decades due to its low melting temperature, low cost, good solderability, and satisfactory mechanical properties. However, due to human health and environmental concerns, lead (Pb) is being phased out of electronic products [1]. Pb content was limited to less than 1000ppm under the Restriction of Hazardous Substances Act (RoHS 1) [2]. Sn-Ag-Cu (SAC)-based materials are currently the most promising surface mount alloy solutions [2]. The SAC305 alloy, which contains 96.5% tin, 3.0% silver and 0.5% copper, is the preferred option for surface mounting technology (SMT) assembly. The melting point for the most widely used lead alloys, such as Sn-60 Pb-40, is 183°C, while the melting point of the most commonly used lead free alloy, SAC305, is 217°C to 219°C [3]. The melting point transition is crucial for the formation of intermetallic bonds during the reflow process. SAC mechanical properties such as melting temperature, hardness, wettability, and tensile stress are improved by incorporating nanocomposite materials such as Ag, Fe_2O_3, NiO and Al_2O_3 [4].

A thin IMC layer formed between the solder and the substrate is useful for joint bonding. However, the thicker IMC layer reduces joint strength due to low fracture strength and brittleness [5]. The presence of voids in the IMC layer causes the solder joint to fracture and fail. These voids act as crack initiators and propagate at random. IMC typically forms tin-based compounds such as Cu_6Sn_5 or Cu_3Sn during the reflow soldering process [6]. As time and temperature rise, the interface will first form a good Cu_6Sn_5 IMC layer and then gradually deteriorate into a weak brittle Cu_3Sn IMC layer. Chromik et al. [7] reported that Cu_6Sn_5 had the highest hardness compared to Cu_3Sn and Ag_3Sn, while Cu_3Sn was brittle whereas Ag_3Sn was ductile. On the other hand, Xiao and Yuang [8] found that the Cu_3Sn hardness and elastic modulus were higher than Cu_6Sn_5. Xu and Pang [9] and Rao et al. [10] found that Cu_3Sn elastic modulus and hardness exceeded Cu_6Sn_5. Yang et al. [11] found that the mechanical reactions of both Cu_6Sn_5 and Cu_3Sn depended largely on the strain rate during loading.

The hardness and elastic modulus of solder joint are measured by the Continuous Stiffness Measurement (CSM) nanoindentation technology. These mechanical properties are continuously updated from the beginning till the end of indentation [12]. Thermal energy causes Cu atoms to dissolve and diffuse with Sn atoms to create Cu_3Sn in a solder junction [13]. The aging temperature and strain rate rise when the solder joint's fracture mode changes from ductile to brittle.

Several researchers [4-13] investigate the effects of nanoparticle reinforcement's mechanical properties on SAC solder paste. However, the author is unaware of any studies that have used real industrial assembly packages to analyse the mechanical properties of nanoparticle reinforcement of SAC305 with TiO_2, Fe_2O_3, and NiO using nanoindentation. There is one study found on the hardness profiles of SAC305 with TiO_2 by using nanoindentation [14], but it has not used real industrial assembly packages. The author also found one study that used real industrial assembly packages showed in [15] however, did not focus on nanoindentation analysis.

The aim of this study was to evaluate the effect of nanoparticle reinforcement in solder paste on the microstructure and thickness of the IMC layer in solder joints, as well as the effect of nanoparticle reinforcement on the hardness and elastic modulus of the IMC layer and bulk solder. The real industrial assembly packages with nanocomposite lead-free solder joints samples were analysed by using a three-sided Berkovich pyramidal indenter for nanoindentation experiments. The composition of the bulk solder and indentation mark were analysed using the SEM for data analysis and the Energy Dispersion X-ray spectrum (EDX) for analysing the distribution of the compound in the solder joint.

2.0 METHODOLOGY

2.1 Sample Preparation

The industrial assembly packages used in this study was a 01005 capacitor with a size of 0.4 × 0.2mm mounted on a printed circuit board (PCB) via reflow soldering, as shown in Figure 1 [16]. TiO_2, Fe_2O_3 and NiO nanoparticles with particle sizes of 10nm are used. The particle size of SAC305 is between 15 and 25 μm as received. Prior to assembly, each type of nanoparticle with 0.05% weight is mixed with pure SAC305 solder paste using a mechanical stirrer at 300rpm for approximately 10 minutes to achieve homogeneity. Thus, three nanocomposite lead free solder mixtures are produced: SAC305-0.05wt% TiO_2, SAC305-0.05wt% Fe_2O_3, and SAC305-0.05wt% NiO.

The lead free solder paste was printed with a thickness of 0.127 mm. The assembly packages was placed on the printed lead free SAC305-nanocomposite solder paste using the component placement machine. The soldering was carried out in a complete convection reflow oven with a lead-free reflow profile. The reflowed PCBs were then washed with an aqueous cleaning machine for de-fluxing. The ready PCB with mounted capacitor was then cut into a small piece at the target area for cold mounting sample preparation, followed by a polishing process to produce a good surface finish for further characterization of the IMC layer, as shown in Figure 2.

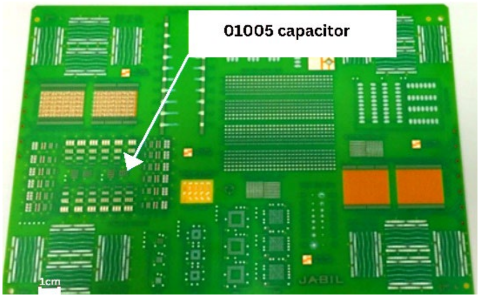


Figure 1: Industrial assembly packages that contain a 01005 capacitor mounted on printed circuit board (PCB) [16]

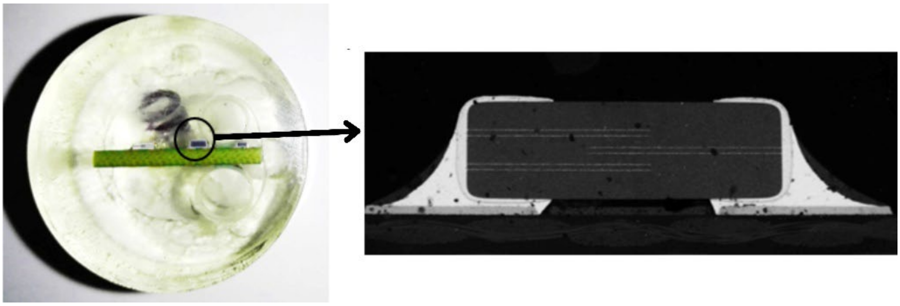

(a) (b)

Figure 2: (a) Cold mounting sample and (b) SEM image of solder joint

2.2 Nanocomposite Lead Free Solder Joint Characterization

The IMC layer hardness was measured with a NanoTest Vantage, Micro Materials equipped with a three-sided Berkovich pyramid tip and a maximum load of 10 mN, a loading rate of 0.5 mN/s, and a dwell time of 60 s [17-19]. These parameters are determined by the effect of loading rate on creep characteristic and indentation size at each maximum load. The SEM is used to observe IMC microstructure and indentation marks on samples as shown in Figure 3. The scanning is done at 15 kV to determine the indentation mark and the microstructure of the IMC layer. The solder joint's area of interest is captured, and an EDX analysis is performed on specific regions to analyse its composition. The indentation parameters are 0.1 mN limit stop load and 0.02 mN image load.

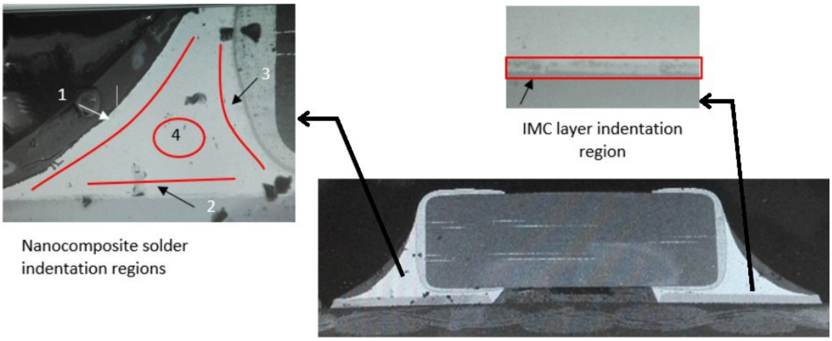


Figure 3: Indentation regions for IMC layer and nanocomposite solder

2.3 Nanoindentation Testing

Oliver and Pharr's method is widely used in nanoindentation to determine the hardness and elastic modulus. Figure 4 shows the load-displacement curve, which gives the maximum load (P_{max}), maximum displacement (h_{max}) and elastic unloading stiffness or contact stiffness (S).

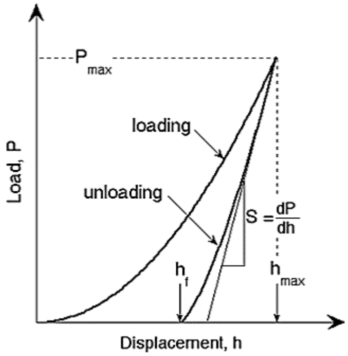


Figure 4: Load-displacement curve of identation [18]

The unloading processes shown in Figure 4 are used to calculate hardness and elastic modulus. The Berkovich indenter is used at an angle of $\phi = 70.3°$. Assuming the pile-up is negligible, the elastic model shows the sink-in amount, h_s is given in Equation (1), where ϵ (Geometry constant of the indenter) = 0.75 for the Berkovich paraboloid indenter. The contact depth (h_c) is used to calculate the contact area ($A = F(h_c)$). Equation (2) gives the value of h_c. However, the projected contact area is obtained from Fischer – Cripps by Equation (3).

$$h_s = \epsilon \frac{P_{max}}{s} \tag{1}$$

$$h_c = h_{max} - \epsilon \frac{P_{max}}{s} \tag{2}$$

$$A = 24.49 h_c^2 \tag{3}$$

The hardness, elastic modulus and stiffness of the projected area can be calculated using Equations (4)-(6), respectively.

$$H = \frac{P_{max}}{A} \tag{4}$$

$$S = \beta \frac{2}{\sqrt{\pi}} E_{eff} \sqrt{A} \tag{5}$$

$$\frac{1}{E_{eff}} = \frac{1-v^2}{E} + \frac{1-v^2}{E_1} \tag{6}$$

The value of β = 1.034 is used as a constant. For Berkovich diamond indenter, E_1= 1141 GPa and v_1 = 0.07. An assumption of ν = 0.3 is used to calculate the elastic modulus of samples whose elastic modulus has not been measured.

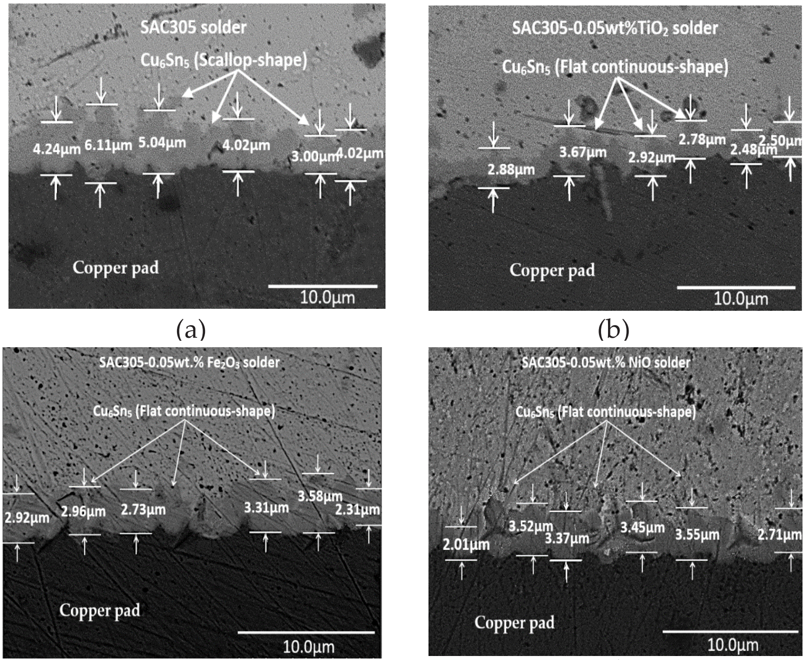
3.0 RESULTS AND DISCUSSION

3.1 IMC Layer Thickness and its Microstructure Analysis

The brittleness of the IMC layer, especially when it is thicker, contributes to the failure of solder joints. Thus, it's crucial to analyse the formation of the IMC layer thickness between the copper pad and the bulk solder in the real industrial assembly packages. The microstructure of the IMC layer and its thickness is observed using SEM as depicted in Figure 5. It is assumed that CU_6Sn_5 is the only IMC layer present due to the fact that only a single IMC layer is visible via SEM in Figure 5. This assumption is then proved by the EDX result in Figure 6, where only the elements of copper (59.06%) and tin (40.02%) are available to show the Cu_6Sn_5 IMC layer is visible between the copper pad and the bulk solder. The scallop-shape pattern of IMCs was observed in pure SAC solder with thicknesses ranging from 3.0 to 6.5 µm. In contrast, a different shape of IMC was found in all nanocomposite solders, with a flat continuous shape identified, as shown in Figure 5(b)-(d) which results in stronger solder joint. The IMC layer thickness for SAC305-0.05wt% TiO_2 is between 2.48 and 3.67µm as shown in Figure 5(b), while the IMC layer thickness for SAC305-0.05wt% Fe_2O_3 is between 2.31 and 3.58µm, as shown in Figure 5(c). In the SAC305–0.05wt% NiO IMC layer, the thickness ranges from 2.01 to 3.55µm. All three reinforced nanocomposite solders show that the IMC layer has a thinner thickness and flat continuous-shape compared to pure SAC305.

Table 1 shows that the thickness of the Cu_6Sn_5 IMC layer has decreased due to the reinforcement of nanoparticles in SAC solder. The nanoparticles addition into bulk solder refined the Ag_3Sn intermetallic compound. The Cu atom from the substrate is dissolved and diffused into the Sn-rich solder during the reflow soldering process, and the Cu_6Sn_5 IMC layer is formed when the copper and tin reactions occur [15].

The thickness of the Cu_6Sn_5 layer increases with the depth of continuous diffusion of Cu. It has a higher surface energy than larger compounds like Ag_3Sn, which appear to disperse with a compound to form a lower surface energy compound. The refined Ag_3Sn is then absorbed to the surface of the IMC layer, acting as a diffusion barrier to reduce the diffusion of Sn in nanocomposite solder and Cu from copper substrate. Since the IMC layer thickness depends on the depth of the Cu atom being diffused, the IMC layer growth is retained and eventually the IMC layer of nanocomposite solder becomes smaller than the pure SAC305 as shown in Table 1.



(a) (b)

(c) (d)

Figure 5: IMC layers of (a) pure SAC305, (b) SAC305-0.05wt% TiO_2, (c) SAC305-0.05wt% Fe_2O_3 and (d) SAC305-0.05wt% NiO

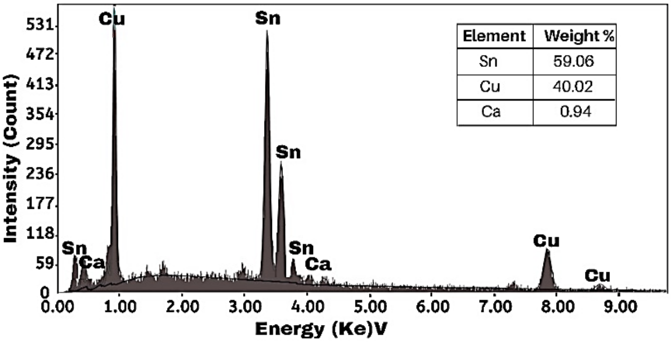


Figure 6: EDX result of IMC layer

The 0.05wt% TiO_2 nanoparticles addition in solder resulted in a 35% reduction in thickness when compared to pure SAC305 solder. Despite the fact that TiO_2 nanoparticles have the greatest thickness reduction, their Cu_6Sn_5 layer thickness is only 2.9-3.1 m, that is due to similar nanoparticle sizes. Other researchers [14, 20-21] reported that TiO_2 nanoparticles inhibited the growth rate of the Cu_6Sn_5 layer and refined Ag_3Sn in composite solder. The reaction with nanoparticle TiO_2 will generate a stable compound with lower surface energy than the entangled compounds. Therefore, Ag_3Sn in solder reacts with TiO_2 to refine the compound's size and spacing. Similar to TiO_2, the addition of Fe_2O_3 nanoparticles made it possible to sustain the growth of the IMC layer [22]. When a small amount of Fe_2O_3 nanoparticles were added to SAC305 solder, the nanoparticles tended to adsorb to the surface of Cu substrates, thereby reducing surface energy. The small size of nanoparticles that adsorb on the IMC layer, which has a low surface energy, has a greater effect on inhibiting the formation of interfacial IMC. The thickness of the IMC layer was reduced by about 32% when Fe_2O_3 was added, and it was reduced by about 29% when NiO was added, when compared to pure SAC305. The addition of NiO nanoparticles to the solder paste reduced the thickness of the IMC layer because NiO nanoparticles precipitated on the Cu_6Sn_5 surface, acting as a barrier to prevent the IMC layer from growing [23]. This lower thickness of the IMC layer is comparable to the findings of this study.

Table 1: Values of mean thickness of Cu_6Sn_5 IMC layer & percentage of reduced thickness between different nanocomposites and pure SAC305

Type of material	SAC305	SAC305–0.05wt%TiO_2	SAC305 – 0.05wt%Fe_2O_3	SAC305 – 0.05wt%NiO
Mean thickness, μm	4.405	2.872	2.968	3.102
Reduced thickness, %		34.81	32.61	29.59

3.2 IMC Layer Hardness and Elastic Modulus Analysis

The relationship between the IMC layers' indent positions, hardness, and elastic modulus is also investigated. When indent positions are moved from the Cu/IMC boundary to the nanocomposite solder, as seen in Figure 7, the size of the indent increases. The Cu/IMC boundary indent is the smallest (2.06 m), whereas the IMC/solder boundary indent is the largest at 5.73 m. During the transition from Cu/IMC boundary to nanocomposite solder, the material's hardness and elastic modulus decrease as the contact area increases, as seen in Figure 8. The maximum difference in hardness and elastic modulus is 2 GPa and 20 GPa respectively. This is due to a large amount of Cu accumulating at the Cu/IMC boundary. The high concentration of Cu_6Sn_5 at Cu/IMC increases its resistance to deformation, resulting in a higher elastic modulus and hardness.

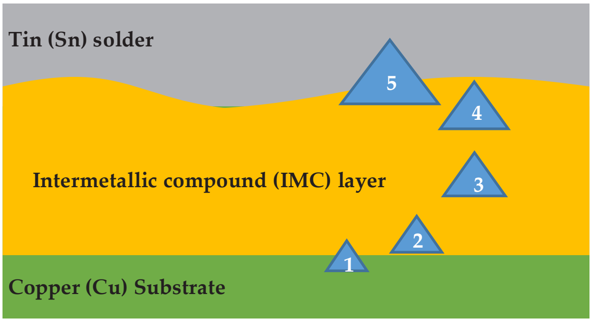


Figure 7: Indent size increases when Cu/IMC boundary is transferred to nanocomposite solder (indent position 1= Cu/IMC boundary; 2=close to Cu/IMC; 3=middle of IMC; 4=close to IMC/solder; 5= IMC/solder boundary)

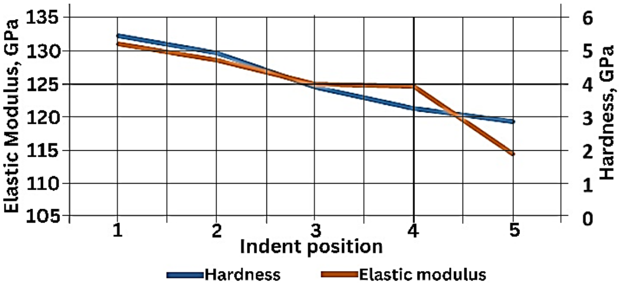
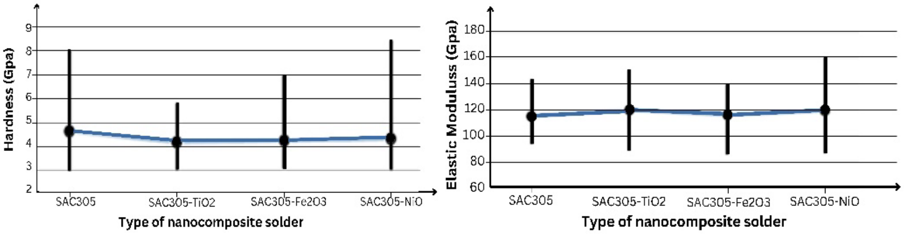


Figure 8: Hardness & elastic modulus based on indent position (indent position 1=Cu/IMC boundary; 2=close to Cu/IMC; 3=middle of IMC; 4=close to IMC/solder; 5=IMC/solder boundary)

Furthermore, the effect of nanoparticle reinforcement on the hardness and elastic modulus of the IMC layer is observed. Four different samples were tested for nanoindentation along the Cu_6Sn_5 layer. Figures 9(a) and 9(b) show the hardness and elastic modulus, respectively.



(a) (b)

Figure 9: Graphs of IMC layer: (a) hardness and (b) elastic modulus

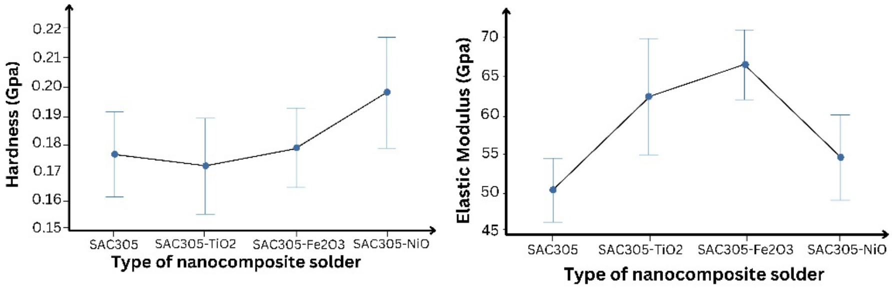
From Figure 9(a), the hardness of pure SAC305, SAC305-0.05wt% TiO_2, SAC305-0.05wt% Fe_2O_3 and SAC305-0.05wt% NiO is 3-8 GPa, 3-5.5 GPa, 3-7 GPa and 3-8.5 GPa, respectively. Elastic modulus for pure SAC305 is 90-140 GPa, 90-150 GPa for SAC305-0.05wt% TiO_2, 80-140 GPa for SAC305-0.05wt% Fe_2O_3 and 90-160 GPa for SAC305-0.05wt% NiO. Figure 9(a) clearly shows that SAC305–0.05wt% TiO_2 has the lowest hardness, which due to the deviated indenter from the defined position. The indentation is done on the upper part of the IMC layer, which is slightly deviated from the defined position. The difference in hardness and elastic modulus ranges is plotted, and their percentage differences are less than 10%, indicating that nanoparticle reinforcement has no significant effects on the hardness and elastic modulus of the IMC layer. Although the Cu_6Sn_5 IMC layer increases in thickness during thermal ageing, the elastic modulus and hardness did not change significantly due to measurement error. Due to differences in sample preparation, test procedure and data treatment, the values of hardness and elastic modulus vary significantly amongst different scientific literatures [8-14].

3.3 Nanocomposite Solder's Hardness & Elastic Modulus Analysis

Figure 10 depicts the nanocomposite solder's hardness and elastic modulus. The hardness of the reinforced solder is higher than the pure SAC305, except for TiO_2 which has a hardness of 2.26% lower than the pure SAC305. These results contradict the findings of previous studies in this field, which proposed increasing hardness by reinforcing low concentration of TiO_2 nanoparticles [19-20]. This reflects the effect of surface roughness on sample hardness and elastic modulus.

Furthermore, the addition of 0.15wt.% TiO_2 nanoparticles into solder resulted in higher hardness as reported in [15] whereas the current studies use a lower TiO_2 composition (0.05 wt.%). The highest hardness is achieved by a NiO nanoparticle reinforced solder of 0.198 GPa.

Meanwhile, the elastic modulus of three reinforced solders is higher than the pure SAC305, and the elastic modulus of Fe_2O_3 nanoparticle reinforced solder is the highest, at 66 GPa, among others. Nanoparticles added to bulk solder refine the Ag_3Sn aggregation distributed in the solder. The Ag_3Sn dispersion and embedded nanoparticles have been fixed to the solder grain surface, preventing grain slipping and increasing hardness over pure SAC305. In addition to NiO nanoparticles, a Sn–Ni physical bond is formed between β-Sn and Ni nano-elements [23]. It is strong enough to resist deformation on the sample surface when compared to the reaction between molten solder and inert ceramic nanoparticles, giving it the highest hardness among the others. In addition, the line curves for hardness and elastic modulus are different in Figures 10(a) and 10(b). Theoretically, the connection between hardness and elastic modulus is generally proportionate. In Figures 10(a) and 10(b), it is depicted in different ways, which is rather intriguing. Further examination of the material surface is conducted to assess the effect of material surface roughness on the contact between the indenter and the surface, and hence the contact compliance. The difference between actual partial contact and ideal perfect contact has led to an inaccurate penetrated depth being obtained and then affected the contact area whereby hardness and elastic modulus change also.



(a) (b)

Figure 10: Graphs of nanocomposite solder: (a) hardness and (b) elastic modulus

4.0 CONCLUSION

The objectives of the study were achieved with the addition of the TiO_2, Fe_2O_3, and NiO nanoparticles to the pure SAC305 solder paste for industrial assembly packages. The microstructure, thickness, hardness, and elastic modulus of the Cu_6Sn_5 IMC layer and nanocomposite solder were improved compared to those of pure SAC305. The Cu_6Sn_5 layer's microstructure pattern changes from elongated-scallop shape (pure SAC305) to flat continuous shape (nanocomposite solder) which results in stronger solder joint. The addition of TiO_2, Fe_2O_3, and NiO nanoparticles changed the microstructure of SAC305 solder and decreased the IMC layer thickness by 29% to 35% due to the presence of the refined Ag_3Sn growth barrier. It is good to have a thinner IMC layer as the brittle behavior of the IMC layer is prone to failure, especially when the IMC layer is thicker. The Cu_6Sn_5 IMC layer's hardness and elastic modulus data show that different nanocomposite solders have similar values, implying that adding nanoparticles has no effect on the mechanical properties of the Cu_6Sn_5 IMC layer. This finding contrasts with the overall performance of the nanocomposite solder, where NiO reinforcement has 11% higher hardness and Fe_2O_3 has 31% higher elastic modulus than pure SAC305. It is also worth noting that the indentation position influences the hardness and elastic modulus of the Cu_6Sn_5 IMC layer.

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