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Microstructural Evolution of Intermetallic Compound Between SN100C Solder and ENIMAG Substrate During Isothermal Ageing

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Abstract

SN100C alloy emerges as a promising candidate when introducing lead-free solder alternatives due to its mechanical properties and solderability. The study aims to explore interfacial reactions between SN100C solder and ENIMAG surface finish. Methods involve preparing ENIMAG substrates, solder paste application, and reflow processes following JEDEC standards. Isothermal ageing treatments were conducted, and interfacial reactions were characterized using top surface and cross-section analyses via Optical Microscope (OM), Scanning Electron Microscope (SEM), and Energy Dispersive X-ray (EDX). Post-reflow, distinct IMC layers formed at the SN100C/ENIMAG solder joint's interface, evolving with ageing. Further analysis via cross-section confirms the initial formation of $(Ni, Cu)_3Sn_4$ layer followed by $(Cu, Ni)_6Sn_5$, each exhibiting varying appearance and thicknesses. Isothermal ageing increases IMC thickness, which is particularly influenced by Cu diffusion and exposure to high temperatures, and it facilitates the growth rate of IMCs.

1. Introduction

In electronic manufacturing, tin-lead solder has long been the go-to for connecting electronic components to circuit boards in various devices. However, with regulations like the European Restriction of Hazardous Substances (RoHS) and Waste from Electrical and Electronic Equipment (WEEE) becoming stricter, the industry has been pushed to find lead-free alternatives [1]. This journey involves thoroughly searching for reliable solder materials, carefully examining solder compositions, and deeply considering environmental impacts [2, 3].

Among various lead-free solder options, the Sn-Ag-Cu alloy stands out for its excellent mechanical properties and solderability [4]. This alloy melts and bonds solder to pads through controlled heating, creating essential connections between the solder and the copper substrate [5]. However, the road to perfecting lead-free soldering faces a significant challenge: the reflow processes. If not managed carefully, these processes can harm solder joint formation by causing brittle intermetallic compounds (IMCs) to form during multiple reflows [6].

To counter the excessive formation of IMCs, the use of Electroless Nickel/Immersion Gold (ENIG) surface finish has emerged as a top solution [7, 8]. ENIG offers outstanding solderability and the ability to limit unwanted IMC formation. It also acts as a protective barrier for the underlying copper substrate during packaging and assembly. However, concerns about the high gold content in ENIG and the persistent black pad issue have led to exploring alternative surface finishes, such as Electroless Nickel/Immersion Silver (ENIMAG). Supporters of ENIMAG highlight its performance similarities to ENIG while avoiding the cost implications of using gold. Thus, this study investigates the interfacial reaction between SN100C lead-free solder and ENIMAG surface finish during reflow processes and isothermal ageing, uncovering crucial insights into solder joint evolution and reliability.

2. Literature Review

The soldering alloy SN100C, which consists of tin, copper, and additional components, has gained popularity in the electronics industry for its good mechanical properties and environmental compliance. SN100C is known for its following elemental composition of 0.70% tin (Sn), 0.70% copper (Cu), and traces of other elements like 0.06% nickel (Ni) and 0.005% germanium (Ge). This solder material demonstrates excellent wetting properties, enabling it to form strong and reliable solder joints with various substrates including copper, gold, and nickel surfaces. This reliable wetting ability reduces the likelihood of solder defects such as solder bridging and non-wetting, thus enhancing the overall quality and integrity of soldered connections. In addition, this solder also ffers good thermal and mechanical properties with a melting temperature of approximately 227 Celsius. This moderate melting point allows for reliable soldering processes without subjecting electronic components to excessive heat, minimizing the risk of thermal damage. Coyle et al. [9] delve into SN100C's microstructural changes and phase transformations, highlighting its eutectic nature and mechanical properties. It is crucial for enhancing the reliability and longevity of solder joints, especially in applications subjected to thermal cycling stress.

Various methods like reflow soldering, wave soldering, and selective soldering are employed with SN100C. Belhadi et al. [10] scrutinize how reflow profile parameters affect solder joint quality, stressing the significance of temperature ramp rates and peak temperatures. Additionally, Cheng et al. [11] suggest a new flux formulation for wave soldering with SN100C, showing better-wetting properties and fewer defects. Moreover, SN100C is used in diverse electronic assemblies, from consumer electronics to automotive and aerospace sectors. Cheng et al. and Matkowski et al. [11, 12] assess the reliability of SN100C solder joints holds up under various stressors like thermal cycling, mechanical shock, and vibration, providing insights into its real-world performance. Overall, SN100C presents a promising alternative to lead-based solders, offering favourable mechanical properties, environmental compliance, and reliability in electronic assemblies.

Understanding the reactions happening at the interface between solder and different substrates such as Cu, Electroless Nickel/Immersion Gold (ENIG), Nickel, Immersion Silver, and Electroless Nickel/Immersion Silver (ENIMAG) is vital for dependable electronic packaging. How solder interacts with diverse substrates brings distinct challenges and opportunities, shaping joint reliability and performance. Research has extensively explored the formation of intermetallic compounds (IMCs), diffusion patterns, wetting properties, and how surface features affect solder adhesion across various substrate materials [13].

The interaction between solder and Cu substrates receives significant attention due to Cu's widespread use in electronic devices. Key challenges involve the formation of intermetallic compounds (IMCs), which can impact both mechanical and electrical properties. Gao et al. [14] delve into the kinetics of IMC formation, emphasizing the influence of temperature and time on interfacial reactions. Essentially, temperature and its distribution across the interface and the specific energy required for IMC formation dictate the speed and manner in which IMCs develop between solder and Cu substrates. Additionally, Jiang et al.'s research [15] highlights the importance of fine-tuning process parameters to establish sturdy solder-Cu interfaces. It proposes an innovative surface treatment method to curb IMC growth and enhance solder joint reliability.

ENIG coatings serve as protective layers on substrates, but their compatibility with solder materials is crucial for ensuring reliability. Tian et al. [16] explore diffusion behaviour at the ENIG-solder interface, revealing significant diffusion of Ni and Au into the solder matrix. Moreover, Lee et al. [17] investigate how ENIG surface characteristics influence solder-wetting properties, stressing the role of surface roughness in promoting solder adhesion. This underscores the intricate relationship between surface chemistry and solder behaviour in ENIG-based assemblies.

ENIMAG coatings blend the advantages of nickel and silver layers, presenting unique challenges and opportunities for solder interactions. Dele-Afolabi et al. [18] studied the impact of ENIMAG surface chemistry on solder wetting behaviour, showing improved wetting properties compared to individual nickel or silver coatings. Furthermore, ENIMAG coatings hold promise for enhancing solder-substrate interactions in advanced electronic applications by tailoring interfacial properties, thereby demonstrating enhanced solder joint reliability in harsh environments [18]. For instance, research by Zolhafizi et al. [19] indicates that employing ENIMAG substrate solder joints results in controlled IMC growth, reduced IMC thickness, and the formation of stable compounds, with Bi additions significantly altering IMC morphology and growth rate.

Reflow soldering stands as a widely embraced technique in electronic manufacturing, linking components to printed circuit boards (PCBs). During reflow, intermetallic compounds (IMCs) typically develop at the interface between solder and substrate, influencing the reliability of solder joints. Hence, the influence of reflow soldering on IMC formation and characteristics at solder-substrate interfaces holds considerable importance. The formation of IMCs occurs at the interface between solder and substrate materials, driven by atom diffusion and ensuing reactions amid soldering. Rasbudin et al. [20] delve into IMC formation kinetics and thermodynamics, spotlighting temperature, time, and alloy composition's roles in governing interfacial reactions to predict and regulate IMC growth during reflow soldering. Besides, reflow parameters, including temperature profiles, heating rates, and cooling rates, have a substantial influence on IMC formation and morphology. Yang et al. [21] explore reflow profiles' effects on IMC thickness, grain structure, and composition, underscoring the need to fine-tune soldering conditions to minimize IMC-related flaws. Moreover, Lee et al. [22] suggest a numerical approach linking reflow parameters with IMC growth kinetics, facilitating process optimization and quality control. A medley of strategies is employed to mitigate IMC formation and improve solder joint reliability during reflow soldering. Li et al. [23] explore solder alloy additives and surface coatings to suppress IMC growth, showcasing bolstered joint integrity and mechanical properties. Furthermore, Ismail et al. [13] probe solder flux chemistry's impact on IMC formation, shedding light on flux residues' role in spurring or hindering interfacial reactions. Given its substantial sway over intermetallic compound formation and traits at solder-substrate junctions, these inquiries underscore the necessity for preventive actions to tackle IMC-related challenges in reflow soldering.

The isothermal ageing process emerges as a pivotal phase in understanding the long-term reliability of solder joints in electronic devices. This process involves exposing solder joints to extended periods at heightened temperatures, triggering the growth and metamorphosis of IMCs. Kelly et al. [24] delve into the kinetics and mechanisms governing IMC transformation during isothermal ageing, shedding light on the roles of diffusion, migration along grain boundaries, and coarsening processes. Understanding the temporal evolution of IMCs is crucial for predicting solder joint reliability throughout prolonged service lifetimes. The duration of isothermal ageing profoundly alters the microstructure and morphology of IMCs at solder-substrate interfaces. Li et al. [25] investigate how ageing duration affects IMC thickness, grain dimensions, and phase constitution, unveiling nonlinear growth patterns and phase shifts over time. Furthermore, Sharma et al. [26] analyze the correlation between ageing duration and IMC mechanical properties, showcasing changes in hardness and fracture toughness with prolonged ageing. The evolution of IMCs during isothermal ageing bears direct consequences on the reliability and performance of solder joints in electronic assemblies. Andersson et al. [27] assess the impact of ageing-induced IMC growth on solder joint mechanical properties, thermal conductivity, and fatigue resistance, highlighting the degradation mechanisms and modes of failure linked to extended ageing. Yet, another study suggests that isothermal ageing might hinder the strength of joints, as demonstrated by Zhang et al. research [28], which investigates solder joint reliability under such conditions, revealing that joints formed through reflow soldering tend to lose more strength compared to those aged for 21–28 days. These studies stress the necessity of factoring in ageing effects when evaluating solder joint reliability under real-world operating conditions.

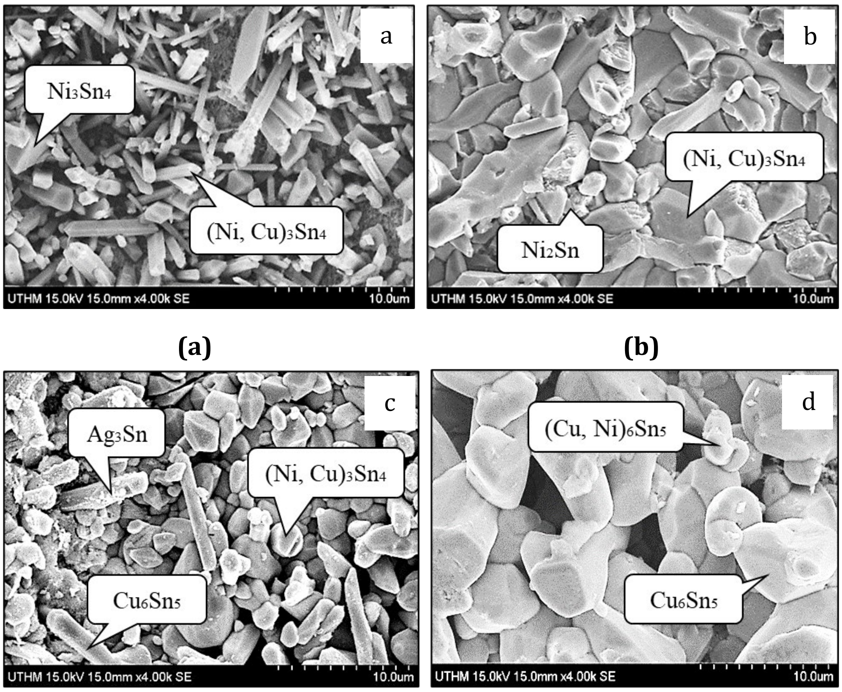
3. Materials and Methods

The copper substrates with dimensions of 30 mm x 30 mm x 0.15 mm were used as the base for applying ENIMAG coatings. Before implementing the electroless nickel and immersion silver process, the Cu substrate underwent pre-treatment [29]. Subsequently, the substrates were immersed in a plating bath containing electroless nickel and immersion silver solution. The plating bath temperature for the substrates was kept at 90℃ and 40℃ for 60 and 8 minutes, respectively [29]. The ENIMAG substrate was prepared by applying flux on the top surface and then arranging solder paste on top of the flux/substrate. The solder paste used was SN100C, composed of Sn-0.7Cu-0.05Ni-0.009Ge. The packages were reflowed at 230℃ using a Memmert furnace for 20 minutes and then cooled to room temperature. The solder joint formation process followed the Joint Electron Device Engineering Council (JEDEC) standard of JESD22-B102E. After that, the packages were subjected to isothermal ageing treatment at 150℃ for a different ageing duration from 250 up to 1000 hours, following the high-temperature storage test outlined in the JEDEC standard (JESD22-A103C). Two types of characterization analyses were conducted: top surface and cross-section. The packages were mounted in epoxy resin for cross-section analysis, followed by metallographic grinding and polishing techniques to obtain the desired results. As for the top surface analysis, the packages were etched in a solder removal solution to expose the interfacial microstructure. The characterization of interfacial reaction after reflows and isothermal ageing process at the solder joint interface will be carried out by 3D morphology and cross-sectional examination of intermetallic was determined by selectively etching solution and using the optical microscope (OM), scanning electron microscope (SEM) and energy dispersive x-ray (EDX).

4. Results and Discussions

4.1 Top View Analysis

In reflow soldering processes, the Cu atoms within the Electroless Nickel/Immersion Silver (ENIMAG) coated substrate dissolve into the molten solder, initiating a reaction and resulting in the formation of solid intermetallic compounds (IMCs) at the interface. Figure 1 presents the top surface analysis, revealing the overall IMC morphology of the SN100C/ENIMAG solder joints post-reflowed and aged durations ranging from 250 to 1000 hours. Following the post-reflow, the immersion silver layer dissolves into the molten SN100C solder paste, exposing the nickel layer to the solder and forming a thin needle-like scallop Ni_3Sn_4 IMC. The Ni_3Sn_4 IMC layer appears thin and refined, influenced by the specific alloying elements present in SN100C and their interaction with the ENIMAG substrate which is consistent with findings from other research by Azman et al. [30] where both $(Ni, Cu)_3Sn_4$ and Ni_3Sn_4 exhibit comparable morphology due to the same interaction between the exposed nickel layer from Electroless Nickel/Immersion Gold (ENIG) surface finish and SN100C. This interaction fosters the growth of a finely grained and thin Ni_3Sn_4 IMC layer during soldering. After being exposed to ageing for 250 hours, a rod-like $(Ni, Cu)_3Sn_4$ IMC emerges at the interface due to the heating during the reflow process, which promotes the diffusion of nickel and copper atoms. Furthermore, the SN100C solder alloy comprises elements such as Sn, Ag and Cu elements, with nickel playing a key role in the formation of $(Ni, Cu)_3Sn_4$ IMC, influencing the kinetics and thermodynamics growth of IMC formation and affecting the morphology and distribution of the IMC layers. For instance, the rod-like structure of $(Ni, Cu)_3Sn_4$ IMC suggests directional growth likely influenced by its crystal structure and solidification conditions [31, 32].



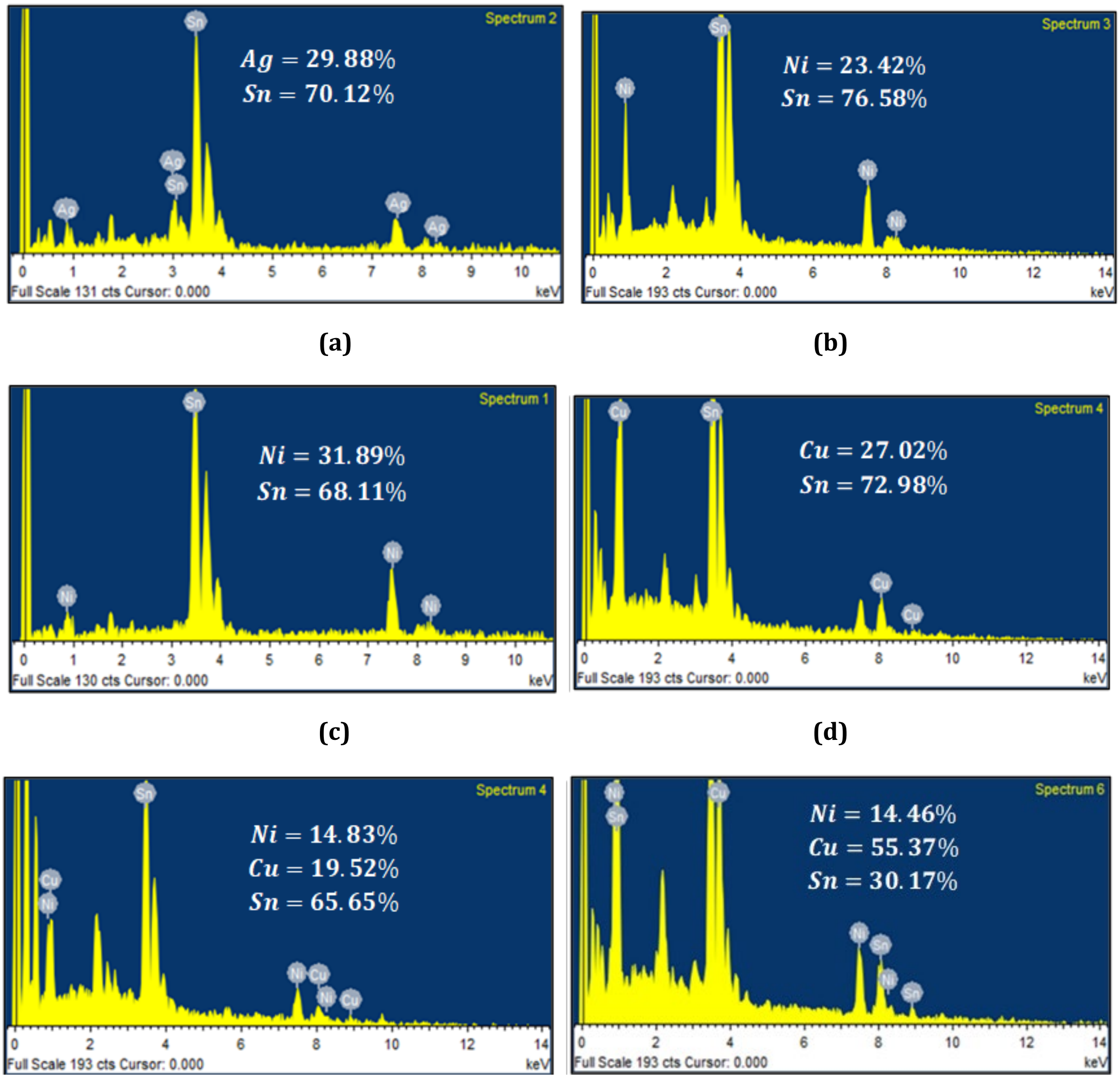
(a) **(b)**

(c) **(d)**

Fig. 1 *Scanning electron microscope view of SN100C/ENIMAG solder joint with magnification 4000x; (a) 0 hour; (b) 250 hours; (c) 500 hours; (d) 1000 hours of isothermal ageing*

Ni_2Sn IMC is also observed at the SN100C/ENIMAG solder joint's interface, representing a specific intermetallic compound with a 2:1 ratio of nickel to tin atoms in a metallurgical system. Subsequent ageing for 500 hours reveals the formation of a chunk-like Cu_6Sn_5 IMC at the SN100C/ENIMAG solder joint's interface despite the solder's low Cu content compared to Sn, Ag, and Ni. In addition, the presence of rough rod-like shaped Ag_3Sn IMC is also observed at the interface. This stems from the solder's local composition near the interface, which becomes enriched in Ag due to the specific reaction forming binary or ternary IMC. However, the rapid growth of Ag_3Sn within the liquid phase has the potential to influence mechanical behaviour and decrease the fatigue life of the solder joint. This observation aligns with research conducted by Faizal et al. [33], indicating that a significant Ag_3Sn IMC structure contributes to solder joint failure and cracking and initiates and propagates along the interface between Ag_3Sn and solder. Following 1000 hours of ageing, a stable ternary IMC with a prismatic-shaped $(Cu, Ni)_6Sn_5$ structure appears at the interface due to the diffusion of Ni from the solder alloy towards the Cu substrate. This reaction at the interface enables Sn, Cu, and Ni atoms to diffuse through the existing IMC layer and form $(Cu, Ni)_6Sn_5$ [34].

Conversely, if the solder material lacked additional Ni, $(Cu, Ni)_6Sn_5$ would not form, and the Cu_6Sn_5 IMC would remain stable. However, the SN100C solder contains sufficient Ni to reach the substrate, allowing $(Cu, Ni)_6Sn_5$ to dominate the interface. Furthermore, the similarity in atomic sizes between Cu and Ni facilitates Ni atoms replacing Cu atoms during reflow and ageing, increasing atomic concentration and providing more nucleation sites for $(Cu, Ni)_6Sn_5$, thereby explaining the smaller grain size compared to Cu_6Sn_5 [35]. Both $(Ni, Cu)_3Sn_4$ and $(Cu, Ni)_6Sn_5$ are recognized as stable forms in the Cu-Sn-Ni ternary system and tend to be smaller than Ni_3Sn_4 and Cu_6Sn_5 compounds. This observation aligns with Tseng et al. [32]. Energy Dispersive X-ray (EDX) spectrum analysis in Figure 2 confirmed the presence of Ag_3Sn, Ni_2Sn, Ni_3Sn_4, Cu_6Sn_5, $(Ni, Cu)_3Sn_4$, and $(Cu, Ni)_6Sn_5$ IMCs formed at SN100C/ENIMAG solder joint's interface.



(a) **(b)**

(c) **(d)**

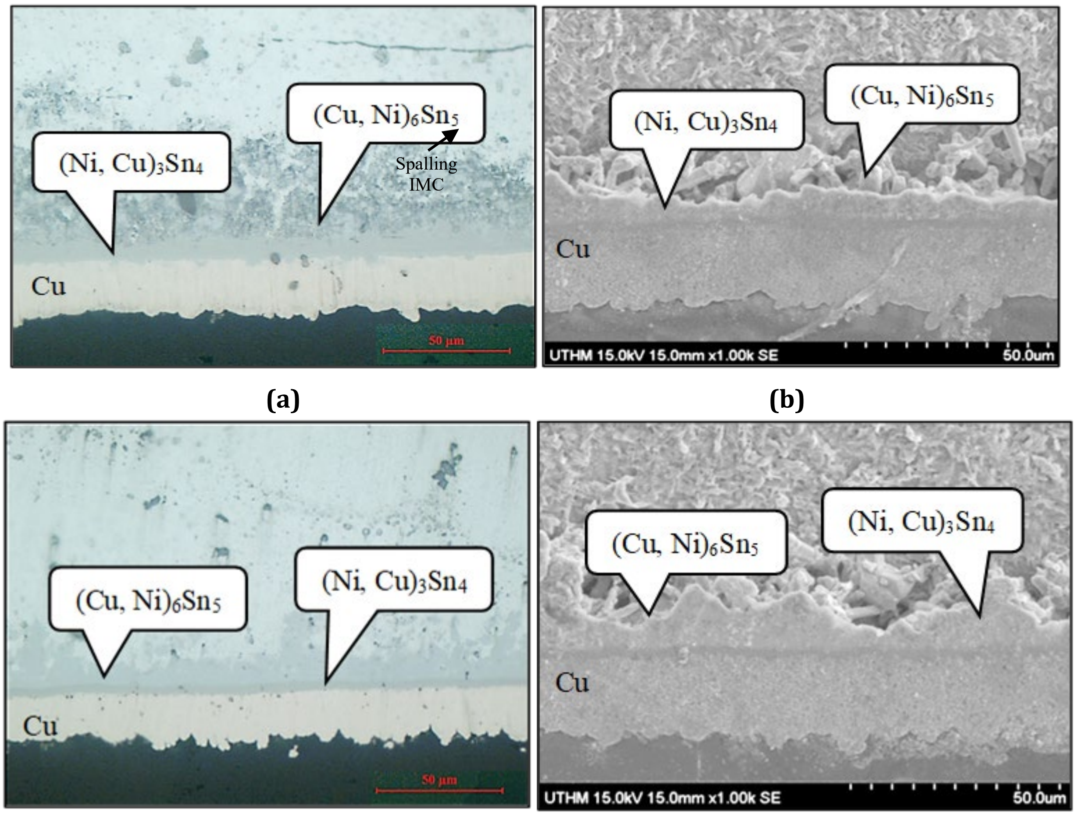
(e) **(f)**

Fig. 2 *Energy dispersive X-ray analysis for the IMC formation at the SN100C/ENIMAG solder joint; (a)* Ag_3Sn*; (b)* Ni_2Sn*; (c)* Ni_3Sn_4*; (d)* Cu_6Sn_5*; (e)* $(Ni, Cu)_3Sn_4$*; (f)* $(Cu, Ni)_6Sn_5$

4.2 Cross-Sectional Analysis

Figure 3 presents the cross-sectional view of the SN100C/ENIMAG solder joint, examined using an optical microscope (OM) and a scanning electron microscope (SEM). In line with the findings from the top surface analysis, the solder joints revealed that the initial layer formed post-reflow was the $(Ni, Cu)_3Sn_4$ intermetallic compound (IMC), with $(Cu, Ni)_6Sn_5$ growing beneath it after a specific ageing duration. The $(Ni, Cu)_3Sn_4$ layer, positioned between the $(Cu, Ni)_6Sn_5$ and the substrate, displayed a dark-grey phase, while the $(Cu, Ni)_6Sn_5$ layer, developed between the $(Ni, Cu)_3Sn_4$ and solder on the interface, exhibited a white-grey phase. Both IMCs demonstrated a non-uniform layer on the interface, notably with the $(Cu, Ni)_6Sn_5$ layer appearing thick and irregular. At the same time $(Ni, Cu)_3Sn_4$ had a thinner and more uniform layer formed through solid-state diffusion. Moreover, the $(Ni, Cu)_3Sn_4$ showed an increase in thickness with prolonged exposure to ageing treatment, particularly in the presence of $(Cu, Ni)_6Sn_5$. In solid-state reactions, these layers continued to grow under continuous heat exposure, driven by concentration gradients of Sn, Cu, and Ni through interdiffusion processes, corroborating findings by Hu et al. [36]. Additionally, the formation of spalling IMC at the interface was observed, as depicted in Figures 3 (a, e, and g). According to research by Zhang et al. [37], spalling in lead-free interfacial reactions is attributed to elevated interfacial energies at the interfaces between the thick compound layer and the substrate. This heightened interfacial energy results in internal stress buildup during the reflow process, leading to the spalling of the IMC as a mechanism to alleviate the induced stress.

Figure 4 compares the average IMC thickness for SN100C solder joint with ENIMAG surface finish after isothermal ageing. The IMC thickness is determined where the average thickness for each ageing duration is 3.08μm, 3.12μm, 3.19μm, and 3.25μm, respectively. After analysis, it becomes evident that the IMC thickness tends to rise proportionally with the ageing duration. For instance, when comparing the IMC thickness after 250 and 500 hours of ageing, there is approximately a 13% rise in thickness. Similarly, there is an approximately 36% increase in IMC thickness between the ageing durations of 500 and 1000 hours. This trend suggests that prolonged isothermal ageing gradually thickens IMC layers at the SN100C/ENIMAG solder joint interface. In simpler terms, the thickness of the IMC layer increases during thermal ageing by considering factors like the diffusion coefficient and how long the ageing process lasts. Various factors determine this coefficient, including the pre-exponential diffusion constant, apparent activation energy, gas constant, and temperature [38–40]. Therefore, based on Figure 4, it is evident that as the duration of time progresses at 150℃, the average thickness of the IMC layer increases in proportion to the ageing duration. Furthermore, increasing IMC growth during ageing increases grain boundaries and smaller IMC grain sizes. This phenomenon facilitates easier and quicker diffusion processes. As the duration of isothermal ageing prolongs, the thickness of both solder joint layers increases, aligning with previous research, such as that conducted by Xiong et al. [41], highlighting a gradual increase in IMC layers with extended ageing duration. Furthermore, while the solder joints exhibit a proportional relationship between IMC thickness and ageing duration, it is evident that the IMC thickness of SN100C/ENIMAG solder exhibits a thin IMC layer. This can be reasoned to the presence of silver in ENIMAG, which acts as a barrier, slowing down the interfacial reaction and reducing the thickness of the IMC layer formed between the solder and substrate. By altering the reaction rates, silver can also influence the kinetics of IMC formation reactions and potentially inhibit excessive growth, particularly at elevated temperatures [42].



(a) **(b)**

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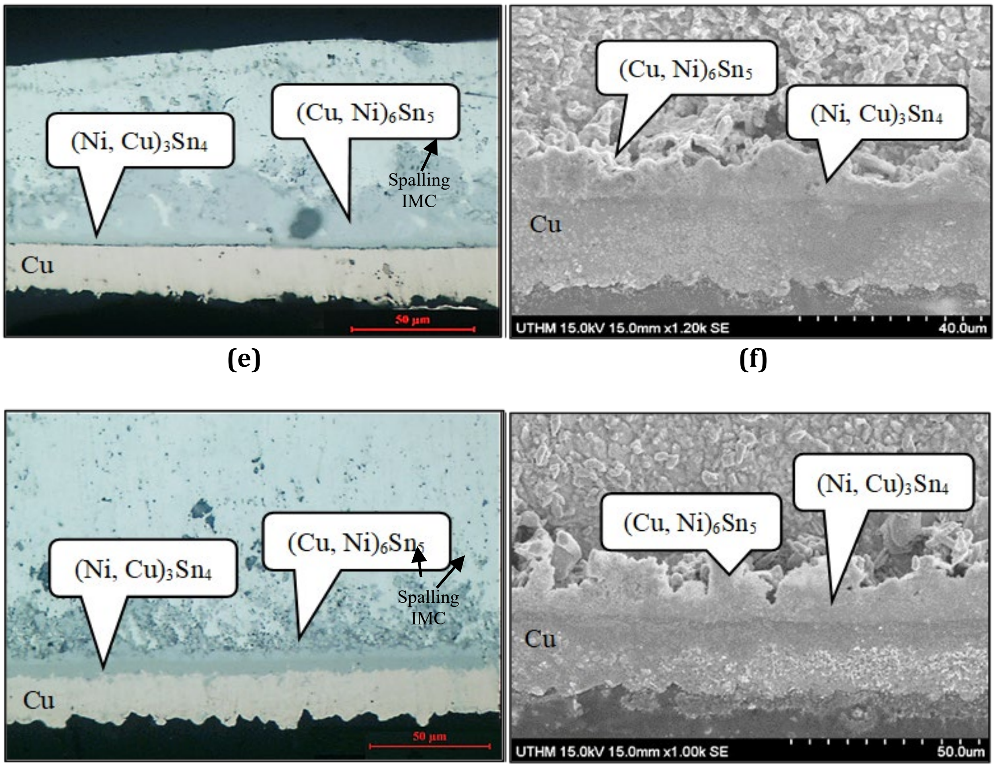

(e) **(f)**

(g) **(h)**

Fig. 3 *Cross-sectional view of SN100C/ENIMAG solder joint under OM and SEM with magnification 50μm and 1000x, respectively; (a) 0 hour; (b) 250 hours; (c) 500 hours; (d) 1000 hours of isothermal ageing*

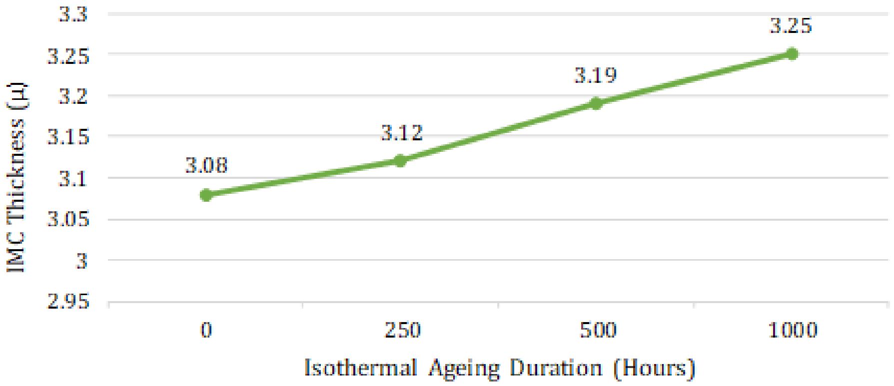

Fig. 4 *IMC thickness versus isothermal ageing SN100C/ENIMAG solder joint durations*

5. Conclusion

From the study, some conclusions can be made:

- Post-reflow analysis showed a thin needle-like Ni_3Sn_4 intermetallic compound (IMC) at the interface of the SN100C/ENIMAG solder joint. Meanwhile, subsequent isothermal ageing induces the formation of Cu_6Sn_5, $(Ni, Cu)_3Sn_4$, and $(Cu, Ni)_6Sn_5$ IMCs, with Cu diffusion notably affecting $(Cu, Ni)_6Sn_5$ formation. Both $(Ni, Cu)_3Sn_4$ and $(Cu, Ni)_6Sn_5$ are stable forms in the Cu-Sn-Ni ternary system.
- Cross-sectional analysis using microscopy confirmed $(Ni, Cu)_3Sn_4$ as the initial layer in both solder joints, with $(Cu, Ni)_6Sn_5$ forming later. These IMCs display different appearances and thicknesses, where $(Ni, Cu)_3Sn_4$ layer exhibits a dark-grey phase, while $(Cu, Ni)_6Sn_5$ displays a white-grey phase, with both IMCs showing non-uniform layers on the interface. $(Cu, Ni)_6Sn_5$ layers appear thick and irregular, while $(Ni, Cu)_3Sn_4$ layers are thinner and more uniform, formed through solid-state diffusion.
- Prolonged exposure to ageing treatments increases the thickness of $(Ni, Cu)_3Sn_4$, especially in the presence of $(Cu, Ni)_6Sn_5$, indicating continuous growth driven by interdiffusion processes. Spalling IMC at interfaces was also observed, likely due to stress during reflow.

- The IMC thickness increased with more exposure to high temperatures, showing a proportional relationship with isothermal ageing duration. The IMC thickness for SN100C/ENIMAG were 3.08µm, 3.12µm, 3.19µm, and 3.25µm, respectively.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm their contribution to the paper as follows: ***study conception and design:*** *Dayang Izzah Nabilah, Saliza Azlina;* ***data collection:*** *Dayang Izzah Nabilah;* ***analysis and interpretation of results:*** *Dayang Izzah Nabilah, Saliza Azlina;* ***draft manuscript preparation:*** *A.A Dayang Izzah Nabilah, O. Saliza Azlina, N. Pavithiran and Y. Kozutsumi. All authors reviewed the results and approved the final version of the manuscript.*

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