Sains Malaysiana 49(12)(2020): 3045-3054 http://dx.doi.org/10.17576/jsm-2020-4912-16

Effect of Isothermal Aging and Copper Substrate Roughness on the SAC305 Solder Joint Intermetallic Layer Growth of High Temperature Storage (HTS)

(Kesan Penuaan Isoterma dan Kekasaran Permukaan Substrat Kuprum ke atas Pertumbuhan Lapisan antara Logam Sambungan Pateri SAC305 pada Penyimpanan Suhu Tinggi (HTS))

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ABSTRACT

This study aims to evaluate the effect of copper (Cu) substrate surface roughness on the intermetallic compound (IMC) growth and interfacial reaction of SAC305 lead-free solder joint after undergone an aging process. Aging process was conducted using high temperature storage (HTS) at temperature of 150 °C and aging times of 200, 400, 600, 800, and 1000 h. IMC morphology and growth were examined using infinite focus microscope (IFM). Then, the SAC305 solder joint IMC growth kinetic was measured based on power law relationship and diffusion coefficient formula. It was noted that the morphology of IMC for the rougher Cu substrate has scallop-shaped and uniform layer as compared to that of smoother Cu substrate for the initial exposure to the HTS. In addition, Cu substrate with R_a of 579 nm is the turning point for the creation of Cu_6Sn_5 towards more Cu_3Sn of IMC. In addition, Cu substrate with R_a of 579 nm also acts as the turning point for the IMC growth of SAC305 solder joint on Cu substrate for the solid-state diffusion to be happened during 150 °C of aging from grain boundary dominant toward volume diffusion dominant.

Keywords: High temperature storage (HTS); IMC layer growth, IMC thickness; SAC305 solder; substrate roughness

ABSTRAK

Kajian ini bertujuan untuk menilai kesan kekasaran permukaan substrat kuprum (Cu) ke atas pertumbuhan lapisan sebatian antara logam (IMC) dan tindak balas antara muka sambungan pateri SAC305 selepas melalui proses penuaan. Proses penuaan dijalankan menggunakan penyimpanan suhu tinggi (HTS) pada suhu 150 °C dan masa penuaan 200, 400, 600, 800 dan 1000 jam. Morfologi dan pertumbuhan IMC dicerap menggunakan mikroskop fokus tak terjangka (IFM). Kemudian, pertumbuhan kinetik sambungan pateri SAC305 diukur berdasarkan hubungan hukum kuasa dan formula pekali resapan. Morfologi IMC untuk substrat Cu yang lebih kasar menunjukkan ia berbentuk kerangan dan lapisan yang seragam berbanding substrat Cu yang lebih licin untuk permulaan pendedahan kepada HTS. Sebagai tambahan, substrat Cu dengan 579 nm R_a merupakan titik permulaan kepada pertumbuhan IMC sambungan pateri SAC305 di atas substrat Cu untuk resapan keadaan pepejal berlaku semasa penuaan 150 °C daripada dominan sempadan terhadap dominan resapan isi padu.

Kata kunci: Kekasaran substrat; ketebalan IMC; penyimpanan suhu tinggi (HTS); pertumbuhan lapisan IMC; pateri SAC305

INTRODUCTION

Lead-free solder joints are widely used as the interconnection mean in the electronic industry. Substrate used as bond pad for the connection of lead-free solder joint are made of different type of metals and copper (Cu) is one of the widely utilised (Abu Bakar et al. 2016; Jalar et al. 2016; Jiang et al. 2019). The quality and the reliability of the connection of solder joints onto the different morphology of Cu substrates are evaluated by different type

of tests like wettability and microstructure evaluations (Ismail et al. 2018a, 2016a; Wang et al. 2017). Wettability is widely used test to measure the adhesion of the solder joint through the contact angle measurement taken right after the application of soldering of molten solder on the substrate (Bhat et al. 2014; Ismail et al. 2016b). However, the determination of solder joint wettability only explains the adhesion and spreading of molten solder through the measurement of the physical appearance.

Microstructure examination and intermetallic compounds growth kinetic measurement at the interfaces of solder joints and Cu substrate provide more detail analysis regarding the quality and reliability of solder joints on different morphology of Cu substrates (Bhat et al. 2014; Ismail et al. 2018b, 2016a). This is because the evaluation based on microstructure and growth kinetic measurement are taken at micrometre scale and at the cross-sectional view that show the internal structure of solder joints with Cu substrate. Several studies have been conducted to evaluate the microstructure evolution and intermetallic compound (IMC) growth kinetic at the interfaces of lead-free solder with Cu substrates. Lee and Ahmad Azmin (2013) reported that SnAgCu (SAC) solder is the most popular type of lead-free solder that has been used as eco-friendly product. They noted that there was two IMC layers commonly found at the interface of SAC solder and Cu substrate which are Cu₆Sn₅ and Cu₃Sn. Both IMCs were growing with the increasing soldering reflow temperature and time. Mo et al. (2015) found that the type of substrate have a significant effect on the IMC microstructure changes. It was noted that the Cu and Cu_cSn_s reaction were much faster on the copper-plated substrate compared to that of copper-rolled substrate. They also observed that the increase of dwell time of reflow time has increased the thickness of IMCs. Hu et al. (2016) conducted the isothermal aging at range 150 to 180 °C for 486 h on the Sn3.0Ag0.5Cu solder joint with the Cu substrate. They noted the increment of IMC thickness during aging were following parabolic relationship with time. In addition, they reported that the formation of IMC is mostly controlled by diffusion mechanism. Li et al. (2015) reported that the thickness of Cu₂Sn in in the SAC305 solder joint and Cu substrate system is much thinner compared to that of pure Sn and Cu system. This is due the inhibiting effect of Ag and Cu on the diffusion phenomenon of Sn atoms into the Cu substrate.

However, there are quite limited sources that discussing the effect of the Cu surface roughness towards the growth kinetic of solder joints IMC after the soldering and aging processes. Bhat et al. (2014) carried out a study on the effect of reflow temperatures and substrate surface roughness on the interfacial reaction of SAC387 solder joint on the Cu substrate. They noted that the morphology or shape of IMCs transformed from long to short needles with the increment of Cu substrate surface roughness. They also stated that the shear strength of SAC37 solder joint on rougher Cu substrate has higher value compared to that of smoother Cu substrate. However, smoother Cu substrate is preferred due to joint failure mode predominantly occurred in solder matrix as compared that of rougher Cu substrate where the joint failure mode happened at the interfaces of SAC387 joint and Cu substrate.

Although the study about microstructure and mechanical performances of SAC305 solder joint on the Cu substrate have been conducted quite intensively, but the relationship between Cu substrate surface roughness towards the reliability and IMC growth kinetic of SAC305 solder joints are still lacking and comprehensive study is critically needed. In the current study, Cu substrates with different surface roughness were prepared through wet grinding process on different grits of silicon carbide (SiC) abrasive papers. This has created the Cu substrates with different average surface roughness (R_{1}) of 567, 505, 477, 338, 311, and 172 nm. SAC305 solder joint was hand soldered on the Cu substrates with different average surface roughness (R_{i}) . Reliability test or aging was carried out the SAC305 solder joints with Cu substrate using high temperature storage (HTS). The shape or morphology changes and IMC growth were evaluated on the cross-sectional view of interface area between SAC305 solder joint and Cu substrate. IMC growth kinetic was measured based on the empirical power law relationship and diffusion coefficient formula to evaluate the SAC305 solder joints and Cu substrate interfacial reaction.

EXPERIMENTAL WORKS

Different surface roughness of Cu substrate with size of $25 \times 15 \times 2.5$ mm were prepared by wet grinding the Cu substrates on the 240, 400, 600, 800, 1000, and 1200 grits of silicon carbide, SiC abrasive papers. The Cu substrates were then cleaned with ethanol and deionized (DI) water. Infinite focus microscope (IFM) made of Alicona, was used to evaluate the surface roughness of Cu substrates that have gone through the wet grinding process. The average roughness (R_a) measurement was obtained using IFM based on the following formula (Anon 2020):

$$R_{a} = \frac{1}{L} \int_{0}^{L} |Z(x)| dx \tag{1}$$

where *L* is the evaluation length and Z(x) is the profile height function. SAC305 lead-free solder wire with diameter of 1.0 mm was used as solder material for current study. Hand soldering with applied temperature of 300 °C was conducted to solder the SAC305 lead-free solder wire onto the six different sample of Cu substrates that have been grounded with six different grit of abrasive papers. This has created six different samples of SAC305 solder joints on Cu substrates.

High temperature storage (HTS) test was carried out on the six samples of SAC305 solder joints with Cu substrate using Memmert universal oven based on JESD22-A103C standard with applied temperature of 150 °C. Five HTS aging times of 200, 400, 600, 800, and 1000 h have been chosen to be applied on the six samples of SAC305 solder joints with Cu substrate. This has produced six SAC305 solder joints with Cu substrate for each five HTS aging time.

After SAC305 solder joints with Cu substrate was tested with HTS test, the sample was prepared for microstructure examination using metallography procedure by first resin was mounted the samples. When the samples were cured, wet grinding was carried out with 600, 800, 1200, and 2000 grits of abrasive papers. Then, the samples were polished with 1 and 0.25 µm diamond suspensions on silk cloth. SAC305 solder joints with Cu substrate was immersed into an etchant solution of 5% hydrochloric acid (HCL) and 95% methanol for 10 s, then the sample was rinsed with deionized (DI) water to show the microstructure. Microstructure examination of cross sectioned of SAC305 solder joints with Cu substrate was performed using IFM. The measurement of intermetallic compounds (IMC) thickness was taken by gathering 100 vertical thickness readings using IFM.

RESULTS AND DISCUSSION

Figure 1 shows the surface morphology of Cu substrate grounded by SiC abrasive papers with grits of 240, 400, 600, 800, 1000, and 1200. Figure 2 illustrates the depth versus path length profiles of Cu substrate grounded by SiC abrasive grits of 240, 400, ,600, 800, 1000, and 1200. As shown in Figure 1(a), the surface shows rougher and the depth versus path length profiles curve was less shallow as in Figure 2(a) when the decreasing the number of SiC abrasive paper grits. When the increasing number of SiC abrasive paper grits as shown in Figure 1(d), the surface morphology shows smoother and the depth versus path length profiles curve becomes shallower which confirms that the surface roughness is reduced. While in Figure 3 exhibits variation of Cu substrate, R_a towards SiC abrasive paper grits. In Figure 3, it is shown that the trend of R_{a} reduction is not quite in the linear behaviour where the SiC abrasive paper with grit 240 produces the highest R_{\perp} of 567 nm and reduces gradually for grits 400 and 600 with R_a of 505 and 477 nm, respectively. For SiC abrasive paper with grit of 800, the R_a value is reduced quite abruptly with value of 338 nm before gradually decreases to 311 nm for grit of 1000. Then, the R_a is reduced quite significantly again with value of 172 nm for 1200 grit. This indicates that the R_v value is not changed in the linear relationship as compared to that of the increment of grit values of SiC abrasive papers that have applied where the value is increased with similar difference of grit of 200 except for the grit 240. From Figure 3, it can be identified that the changes of R_a of Cu substrate after grounded by six difference grits of SiC abrasive paper have three segments. First segment is for the Cu substrate with R_a of 567, 505 and 477 nm where the decrement trend is in gradual manner, followed by second segment that start with abrupt decrement towards R_a of 338 nm and then gradual decrement with R_a of 311 nm. Third segment is represented with abrupt reduction towards 172 nm of R_a. Thus, the application of SiC abrasive papers with grits of 240, 400, 600, 800, 1000, and 1200 produce Cu substrates with R_{a} that can divided into three segments that is represented by the reduction manner in either gradually or abruptly changes.



FIGURE 1. Surface morphology of Cu substrate grounded by SiC abrasive papers with grits of (a) 240, (b) 400, (c) 600, (d) 800, (e) 1000, and (f) 1200



FIGURE 2. Depth versus path length profiles of Cu substrate grounded by SiC abrasive papers grits of (a) 240, (b) 400, (c) 600, (d) 800, (e) 1000, and (f) 1200



FIGURE 3. Variation of Cu substrate average surface roughness, R_a towards SiC abrasive grits

Figure 4 shows the micrograph of the cross sectioned at the area of IMC of SAC305 solder joints aged with 150 $^{\circ}$ C and aging time of 1000 h. Figure 5 indicates the variation of IMC average thickness towards aging

time. In Figure 4, it is noted that there two differences IMC created next to the interface between SAC305 solder joint and Cu substrate namely Cu_6Sn_5 and Cu_3Sn which are assigned with dotted red and black lines, respectively.

Several studies have identified the whitish and greyish layers that created at the interface of SAC305 solder joints with the Cu substrate were Cu₆Sn₅ and Cu₃Sn, respectively (Lee & Kim 2014; Xiao et al. 2013). Bhat et al. (2014) reported that the IMC created after reflow soldering for the case of SAC305 solder joints on the Cu substrate have a shape of needle especially for the Cu₆Sn₅ and the shape of IMC transformed from long to short needles or scallop-shaped with the increment of Cu substrate surface roughness. This is attributed to the penetration of molten solder atom occurred in higher degree on the

rough surface as compared to that of smooth surface. Satyanarayan and Prabhu (2013) also noted that higher level of asperities possessed by rough surface increase the capillary action for solidification of molten solder on the rough Cu substrate surface. Therefore, for the as soldered or initial exposure to the HTS, the IMC of SAC305 solder joints on Cu substrate with rougher surface have scallopshaped and uniform IMC, particularly Cu₆Sn₅ as compared to that of the smoother Cu substrate surface where it has quite non-uniform IMC as shown in Figure 4.



FIGURE 4. Micrographs of the cross sectioned at the area of IMC of SAC305 solder joints aged with 1000 h and Cu substrate surface roughness of a) 806, b) 600, c) 579, d) 557, e) 490, and f) 340 nm

Figure 5 shows the variation of IMC average thickness towards aging time. As mentioned earlier, the measurement of IMC average thickness was taken from 100 readings of vertical thickness. In Figure 5, it is noted that the IMC average thickness has inversely proportionate relation with the Cu substrate R_a and directly proportionate relation with the aging time. In

addition, it is shown that the trend of Figure 5 cannot be divided into three segments of abrupt, gradual and higher slope decrements of Cu substrate R_a as indicated in Figure 3. Furthermore, Cu substrate with smoothest R_a of 340 nm have the highest and abruptly increment of average thickness especially for the cases of 200, 600, and 800 h of aging time. Therefore, the increment of IMC average

thickness is not in linear manner with decreasing Cu substrate R_a and increasing aging time which also cannot

be divided into three segments as noted in the variation of Cu substrate R_a towards SiC abrasive grits.



FIGURE 5. Variation of IMC average thickness towards aging time

In order to evaluate the IMC growth rate behaviour and mechanism, time exponent, n is obtained based on the following empirical power-law relationship (Wang et al. 2018):

$$\mathbf{X} - \mathbf{X}_{0} = \mathbf{k}t^{n} \tag{2}$$

where x is the total thickness of the reaction layer at time t; x_o is the initial thickness; k is the growth rate constant; and n is the time exponent. To obtain the time exponent, n linear regression analysis on the logarithmic expression of empirical power-law relationship is carried as follows:

$$\log(x - x_0) = \log k + n \log t \tag{3}$$

Then, the graph of $log(x-x_a)$ vs log t is plotted as illustrated in Figure 6 where the R^2 and the slope of the graph are obtained. Figures 7 and 8 exhibit the variation of R^2 towards Cu substrate R_a and variation of time exponent, n towards Cu substrate R_a , respectively. In Figure 7, it is noted that R^2 has values ranging from 0.79 until 0.99 which mean the growth of SAC305 solder joints IMC obey the parabolic law. From Figure 7, it is shown that time exponent, n values vary across different Cu substrate R_a values with most of it has n value of more than 0.5 that represents the diffusion-controlled IMC growth mechanism except for the Cu substrate with R_a of 477 nm where the n is equals with 0.46 which represents the chemical reaction-controlled IMC growth mechanism. This means that SAC305 solder joint on Cu substrate with R_{a} of 477 nm has the lowest diffusivity rate compared to the rest of Cu substrates. As mentioned earlier, the measurement of IMC growth for the current study includes both Cu₂Sn and Cu₆Sn₅ IMCs. Therefore, both changes in term of IMC growth are attributed by both IMCs. The *n* values are decreased for the roughest Cu substrate with R_a of 567 until 477 nm. As mentioned earlier, the rougher Cu substrates have scallop shapes of IMCs particularly Cu₆Sn₅ with the existence of grooves or asperities between each grain of IMC. The existence of groves between the Cu₆Sn₅ grains is known to provide the channels for diffusion between Cu atom from Cu substrate into Sn atom in SAC305 solder to be happened. As the Cu substrate become smoother particularly at R_a of 477 nm, the creation of Cu₂Sn₅ becoming more into planar shape that has lesser groves or channels that hinder the diffusion of Cu atom from Cu substrate directly into Sn atom of SAC305 solder. That is why the *n* for Cu substrate with R_a of 477 nm has lowest value of 0.456 that represent lower diffusivity process of chemical reaction mechanism (Mo et al. 2015). From Figure 8, it is noted that the *n* is starts to increase with the smother Cu substrate with R_a of 338 and 311 nm. This increase of diffusivity might be due to the increase of solidstate diffusion between Cu atom from SAC305 solder with Sn atom from Cu₆Sn₅ which eventually crates the Cu₃Sn that located in between Cu₆Sn₅ and Cu substrate. According to Liu et al. (2018), the increase growth rate of Cu_3Sn is attributed by the shape of Cu_6Sn_5 , where the reduction of the channels with planar shape of Cu_6Sn_5 has increased the possibility of Cu atom from Cu substrate to diffuse into Cu_6Sn_5 IMC and create Cu_3Sn by the reaction of Cu_6Sn_5 + 9Cu 5Cu₃Sn. In addition, Tang et al. (2018) reported that the creation of Cu_3Sn is becoming more apparent for the lead-free solder that have gone through solid-state aging process. Hence, the Cu substrate with R_a of 477 nm is the turning point for the creation of Cu₆Sn₅ towards more Cu₃Sn due to the closing of the channel of planar shape Cu₆Sn₅ that increase the possibility of Cu atom from Cu substrate to diffuse into Cu₆Sn₅ IMC and create Cu₃Sn.



FIGURE 6. Plot of $log(x-x_o)$ versus log t to determine the R^2 and time exponent, n with different Cu substrates R_a of 806, 600, 579, 557, 490, and 340 nm



FIGURE 7. Variation of R^2 towards Cu substrate R_a



FIGURE 8. Variation of time exponent, towards Cu substrate R_a

In order to further analyse the solid-state diffusion behaviour that happened during HTS or aging of SAC305 solder joints with different roughness of Cu substrates, the diffusion coefficient is measured by plotting the IMC thickness versus squared of hour as shown in Figure 9. The plotting of Figure 9 is based on the following layergrowth coefficient or diffusion coefficient formula (Wu et al. 1993; Yu et al. 2005):

$$\boldsymbol{d} = \boldsymbol{d}_0 + \sqrt{Kt_a} \tag{4}$$

where d_0 is the initial thickness of IMC layer K is the layer-growth coefficient that is related to the diffusion coefficient of atomic elements of IMCs and t is time. In Figure 10, it is observed that the diffusion coefficient is decreased gradually for the Cu substrates with R_a of 567, 505, and 477 nm which has the similar reduction trend of n towards Cu substrate R_a as shown in Figure 8. However, the reduction trend of Figure 10 is quite gradual and has least changes as compared to that of Figure 8. According to Li et al. (2015) the diffusion coefficient of Cu in Sn during aging with 150 °C was 2.05×10^{-11} m²/s and this value is not that far with current diffusion coefficient with range of 9.726×10^{-10} until 2.562×10^{-9} m²/s. Like that of Figure 8, the diffusion coefficient of Cu substrate with R_a of 477 nm is the turning point for the diffusion coefficient to increase quite significantly with the decrease of Cu substrate R_{1} of 338, 311, and 172 nm. This might be because the grain boundary diffusion is the dominant factor for the IMC growth in the rougher surface of Cu substrates with R_a of 567, 505, and 477 nm. Whereas, in the smoother surface of Cu substrates with R_a of 338, 311, and 172 nm, the volume diffusion might be the predominant for the IMC growth (Rabiatul Adawiyah & Saliza Azlina 2018). Thus, Cu substrate with R_a of 477 nm also acts as a turning point for the IMC growth of SAC305 solder joint on Cu substrate for the solid-state diffusion to be happened during 150 °C of aging from grain boundary dominant toward volume diffusion dominant.



FIGURE 9. Plot of IMC thickness versus time squared to determine the slope or diffusion coefficient with different Cu substrates R_a of 567, 505, 477, 338, 311, and 172 nm



FIGURE 10. Variation of diffusion coefficient versus Cu substrate R

Several studies have identified that the crystallography planes play a role in determining the IMC growth direction. Single crystal and polycrystalline Cu substrate have shown significant effect on the IMC nucleation and growth (Yang et al. 2017; Zhu et al. 2019). For the current analysis, polycrystalline Cu have been used as the substrate. This means, the IMC nucleation and growth are in the random locations and directions, respectively. However, the main finding of Cu substrate with R_a of 477 nm is that it acts as a turning point for the IMC creation from Cu₆Sn₅ towards Cu₃Sn and abrupt changes of diffusion coefficient of SAC305 solder joint signifies that Cu substrate surface roughness can be used as an IMC growth control mechanism. This is because the combined effects of different IMC nucleation locations and crystallographic directional growth occurred in polycrystalline Cu substrate can be controlled at certain value of surface roughness as has been shown in the current study. Therefore, the procedure introduces in the current study is the important steps for those who want to determine the suitable Cu substrate surface roughness to control the IMC growth of SAC305 solder joints.

CONCLUSION

The evaluation of the interfacial reaction is crucially needed to provide a more comprehensive analysis regarding the effect of Cu substrate surface roughness on the quality and reliability of SAC305 solder joint. It is noted that, the application of SiC abrasive papers with grits of 240, 400, 600, 800, 1000, and 1200 produce Cu substrates with Rthat can divided into three segments that are represented by the reduction manners in either gradually or abruptly changes. The use of variation of SiC abrasive paper grit is to indicate the surface roughness of SAC305 with Cu substrate. The higher number of SiC abrasive paper grit reduces the surface roughness value obtained by SAC305 with Cu substrate. For the as soldered or initial exposure to the HTS, the IMC of SAC305 solder joints on Cu substrate with the rougher surface have scallop-shaped and uniform IMC, particularly Cu₆Sn₅ as compared to that of the smoother Cu substrate surface where it has quite non-uniform IMC. The increment of IMC average thickness is not in linear manner with decreasing Cu substrate R_a and increasing aging time which also cannot be divided into three segments as noted in the variation of Cu substrate R_a towards SiC abrasive grits. Cu substrate with R_a of 477 nm is the turning point for the creation of Cu₆Sn₅ towards more Cu₃Sn due to the closing of the channel of planar shape Cu₆Sn₅ that increase the possibility of Cu atom from Cu substrate to diffuse into Cu₆Sn₅ IMC and create Cu_3Sn . In addition, Cu substrate with R_a of 477 nm also acts as the turning point for the IMC growth of SAC305 solder joint on Cu substrate for the solid-state diffusion to be happened during 150 °C of aging from grain boundary

dominant toward volume diffusion dominant. Thus, the procedure introduces in the current study is the important early step for those who want to obtain the suitable Cu substrate surface roughness to control the IMC growth of SAC305 solder joints.

ACKNOWLEDGEMENTS

This work is supported by the Ministry of Education, Malaysia under Fundamental Research Grant Scheme (FRGS/1/2019/STG07/UKM/03/1), Universiti Kebangsaan Malaysia (UKM) under Geran Universiti Penyelidikan (GUP-2018-079) and Redring Solder (M) Sdn. Bhd. for research materials and collaboration work.

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Received: 13 August 2020 Accepted: 27 August 2020