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Statistical aspect on the measuring of intermetallic compound thickness of lead free solders

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Abstract: Intermetallic compound (IMC) growth is being studied in earnest in this past decade because of its significant effect the solder joint reliability. It appears that from numerous investigations conducted, excessive growth of IMC could lead to solder joint failure. Leading to this, many attempts has been made to determine the actual IMC thickness. However, precise and true representation of the growth in the actual 3D phenomenon from 2D cross-section investigations has remained unclear. This paper will focus on the measuring the IMC thickness using 3D surface profilometer (Alicona Focus G4). Lead free solder, Sn3.0Ag0.5Cu (SAC305) was soldered onto copper printed circuit board (Cu PCB). The samples were then subjected to thermal cycle (TC) storage process with temperature range from 0 °C to 100 °C for 200 cycles and up to 1000 cycles were completed. From the microstructural observation, IMC of SAC305 growth on Cu PCB below600TC is scallop-like in appearance. In contrast, for 800 and 1000 TC, the IMC appears to be thinner and taller with mixed scallop in shape. Statistical analysis of 300 points on IMC indicated different trend of average, mode and median in its thickness. The thickness of IMC can be represented by values of average, mode and median. However, due to nature of growth in IMC, whereas the irregularities of shape, hillocks and valleys are also being considered, it was recommended that IMC thickness is represented in the mode value instead of median and average value.

Key words: Intermetallic growth; Lead free solder; Thermal cycle test; Surface profilometer

1. Introduction

Recent development in electronic packaging has heightened the need for good solder joint reliability. Good solder joint is important in order for long lasting of life cycle of the components joining as well as the functionality of the device. Various factor affect the solder joint reliability such as IMC layer (Wei et al., 2013), void formation in the solder joint (Yunus et al., 2003), crack, solder joint quality and many more.

Concern arisen that an over growth of intermetallic compound (IMC) can be disadvantageous for solder joint reliability. It is known that, IMC grew at the interface between the solder and substrate and not only acts as the metallurgical bonding part but also mechanical interlocking between surfaces (Frear and Vianvo, 1994). However, Moreover, Meinshausen (2015) and Choi (1999) reported that IMC existence can have some detrimental effects on the joint reliability due to brittle nature.

There are number of studies reported about the IMC growth, which a variance of IMC morphologies formed during soldering and solidification process (Yang et al., 2014).It seems that the growth of IMNC has become quite a big challenge for researchers who needed to control or optimize a suitable thickness for a good solder joint. Normal practice used the average of IMC to represent the IMC thickness. The average thickness is calculated from the IMC cross-section profile. Some have used digital image analysis system to measure the average thickness by taking at least 20 to 40 points of uneven IMC thickness for each sample. Deillon et al. (2014) measured the average of IMC thickness using SEM micrograph. Oliver Krammer, (2014) used algorithm processed from the cross-section image captured using scanning electron microscope. Other researchers such as Ko et al. (2016), Hu et al. (2013) and Xu et al. (2014) determined the average of IMC thickness, by dividing the area of IMC region, by the length of the interface .Madeni et al. (2014) reported that the mean thickness can be used to represent the IMC thickness as well as considering the values of thinner and thicker layer IMC cross-section.

The accuracy of IMC thickness measurements is very crucial where previous researcher typically used the value for activation energy calculation using Arrhenius relationship (Saud and Jalar, 2010; Tan, 2015).

Indeed, the value of IMC thickness is used in the following equation to calculate the growth constant:

$$x = kt^{\frac{1}{2}} \quad (1)$$

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where x is IMC thickness, k is the growth constant and t the reaction time. In order to calculate the activation energy, the value of k (in equation 2) is used in the next equation:

$$k = A \exp\left(-\frac{Q}{RT}\right) \quad (2)$$

and the equation can be written as:

$$\ln k = \ln A - \frac{Q}{RT} \quad (3)$$

Where A is the growth constant (cm^2/s), Q is the activation energy, R is universal gas constant (8.314 J/mol K) and T is the absolute temperature (Kelvin).The activation energy is obtained from the slope of in (k) versus 1/T plot (Mayappan and Ahmad, 2010) using a linear regression analysis.

In view of this, we are suggesting that the IMC measurements should use statistically calculation and to stress the importance in considering the shape of IMC. From the microscopic observations, we can observe that IMC has irregular growth with non-uniform shape with the presence of hillocks and valleys. By taking into account the shape of IMC, the mode data is more suitable in representing the IMC thickness. Therefore, additional type of data in terms of orientation of IMC growth should be included too instead of just average and median in representing the IMC thickness.

This paper will review a few normal practices of measuring the IMC thickness. This paper investigates the IMC thickness using proper statistical analysis on 2d cross section image and suggests the appropriate method for measuring the IMC thickness.

2. Materials and method

Printed circuit board (PCB) from Red Ring Solder (M) Sdn. Bhd. with Copper (Cu) substrate was used for this work (Fig. 1). The dimensions of the PCB Cu are 100 mm x 110 mm x 1 mm. The solder paste consist of 96.5% of tin (Sn), 3.0% of argentum (Ag) and 0.5% of Cu (SAC305). A stencil with thickness of 6 mils is used for solder printing on the PCB Cu. The printed PCB Cu was then placed in a relow oven (Madell Technology Corporation). The printed PCB Cu was reflowed for 8 second at 215 °C. After the reflow process, the samples were then cooled at room temperature. Next, the samples were cut into small pieces and consist of a few pitch of soldered sample using a diamond cutter blade machine (Isomet 1000, Buehler) (Fig. 1).

For thermal cycle (TC) test, the soldered samples were place into a thermal cycle chamber (Tenney, TPS). The thermal cycle test was done according to JEDEC standard (JESD22-A104D). This work chose a temperature range of 0 °C and 100 °C for TC test. The TC storage is 200, 400, 600, 800 and up to 1000 cycles. One complete cycle of thermal cycle test is from soaking at 100 °C for five minutes followed by rapid cooling to 0 °C. The process took 55 minutes. Next soaking at 0 °C for five minutes and continued by heating to 100 °C. One complete thermal cycle test takes 2 hour. The thermal cycle test profile details are shown in Fig. 2.

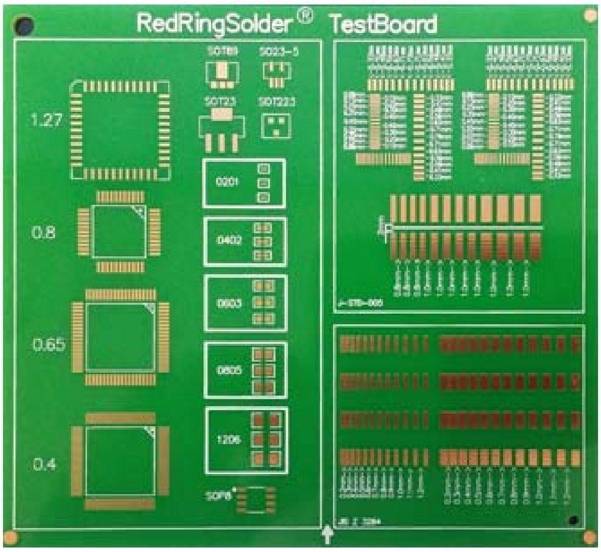

Fig. 1: Printed circuit board (PCB) from Red Ring Solder (M) Sdn. Bhd. with Copper (Cu) substrate

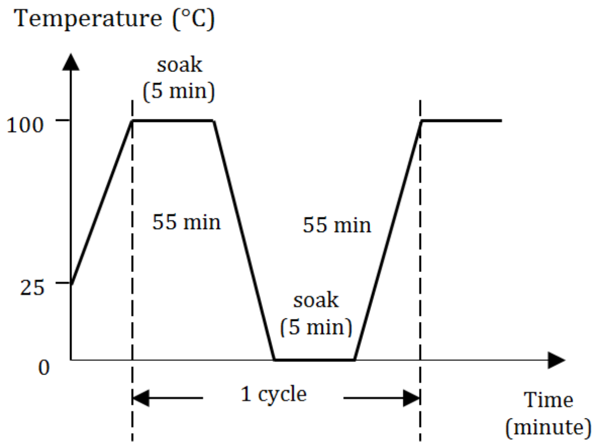

Fig. 2: Thermal cycle temperature profile range of 0 °C and 100 °C.

After the thermal cycle test, the sample was prepared for metallographic cross-section. Sample clip is used to grip the sample as shown in Fig. 3. The sample was put into a mounting cup. A mixture of epoxy resin powder (VersoCit 2-powder) with resin hardener liquid (VersoCit 2-liquid) with ratio of 2:1 is used for cold mounting process. Then, the mixture was stir for 30 second and pour into the mounting cup.

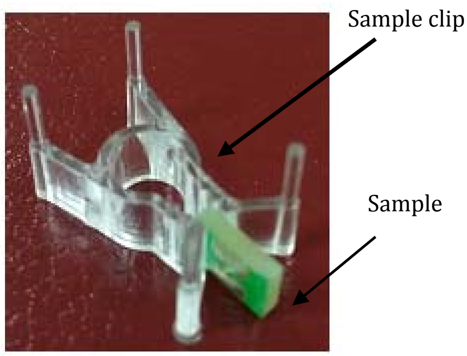

Fig. 3: Sample clip grip the soldered sample

The cold mounted samples are shown in Fig. 4. Next, the mounted sample was grounded starting from low grit or coarse silicon carbide (SiC), 400 grit followed by finer or higher SiC grit 800, 1000 and 1200-grit. Following is polishing process using diamonds spray of 1 μm.

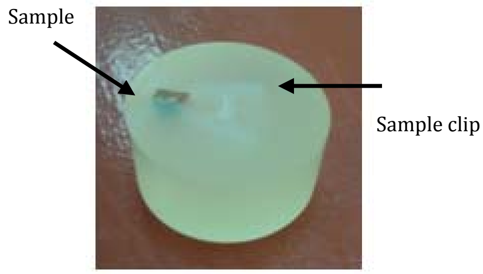

Fig. 4: Sample in cold mounted using epoxy resin

The microstructure of the solder cross-section and the intermetallic compound (IMC) thicknesses were observed using 3D surface profilometer (Alicona Focus G4). In order to measure IMC thickness of the cross-section, the appropriate IMC growth and shape was chosen before selecting point of the measurement. The single measurement point was made using perpendicular length of scale line of a point between top border of IMC and bottom border IMC or top border of Cu substrate. Then the process was repeated for at least for 300 different measurement points to provide 300 different points IMC thickness measurement values.

Afterward, the IMC thickness data was analyzed to obtain average, mode and median values. The average value of IMC thickness in this work is obtained by dividing the sum of 300 measurement points of IMC thickness to 300. While, the median of IMC thickness is the middle value of the 300 measurement points value which is listed in ascending numerical order. The mode value of IMC thickness in this work is referred to the value that is repeated more often than any other values within the 300 measurement points taken.

3. Results and discussion

Fig. 5 shows the micrograph of intermetallic compound (IMC) growth of lead free solder, SAC305 on the Cu substrate for 0 cycle (TC) test (Fig. 5a) and after subjected to 200, 400, 600, 800 and 1000 cycles (Fig. 5b-5f).

Some shape irregularities of IMC morphology are observed to be grown between the Cu and solder interface. Scallop-liked IMC shapes can be seen formed on the solder and Cu interface for 0 TC sample as shown in Fig. 5a. Similar morphology was observed grown for 200 TC (Fig. 5b), 400 TC (Fig. 5c) and 600 TC (Fig. 5d). While a thinner and taller mixed of scallop-like shape was found for 800 and 1000 TC. The inset of Fig. 1f showing that the taller IMC shape was observed compared to 800 TC (inset Fig. 5e). Xu et al. (2014) investigate the interfacial IMC growth of carbon nanotubes reinforced SAC305. They reported that the scallop-like morphology of IMC were grown and develop more during the solid state thermal aging. It is observed that, the coverage of IMC growth was enlarged with the increasing of TC. This is due to coarsening of IMC, which dissolve from Cu substrate to solder alloy boundaries by Ostwald ripening (Maleki et al. 2013).

This shows that non-uniform shape and microstructure of IMC has grown on the Cu substrate after subjected to TC. Based on this irregularities IMC shapes, we may possibly consider few types of data that can be used to represent the IMC thickness such as the average, mean, median or mode.

Fig. 6 shows the 2D schematic illustration of measuring point of IMC thicknesses. The Figs describe how the 300 points of IMC thicknesses are measured. The measurement point was made using perpendicular length of scale line from one point to another point. The measured points are narrowly close with each other points. All the data from the 300 points was taken and analyzed. The type of data such as average, mean, median, mode and range are analyzed in order to choose the suitable data to represent the IMC thickness.

Previous researcher determined the thickness of IMC by measuring the height of IMC directly at different position points. Schreck et al. (2013) used image analysis software Olympus Analysis to calculate the surface integral divided by the image detail width to determine the IMC thickness. They reported that their approach of evaluating the IMC thickness is considerably to be more accurate as the IMC were captured in totally, not only at different points.

However, in our approach, we have considered factors like where should we determine the IMC thickness and is it suitable to measure from the edge or middle of the solder joint? The other factor is how the way we cut the sample to get the solder joint cross-section can effect in measurement practice, question such as is. Is it valid to measure from the edge or we should measure the IMC thickness at the center of the solder joint has arisen. All these factors were considered because we knew that the IMC growth is uneven at the substrate surface. As its growth is uneven, we will obviously obtained different values of IMC thickness for the same sample. This led to the question of the accuracy of this kind of measuring technique. Therefore, we are suggesting a more precise method to determine the IMC values by considering the 3D measurements growth phenomenon of IMCs.

This method employs the refinement in measurement by statistical analysis of 300 measured points. In this work, we have carefully considered the precision and accuracy of data taking and analysis. Even though, previous works done only employed 6 to 7 data points and even in statistical analysis, 30 points are considered to be sufficient. But due to the nature of IMC growth, which consists of hillocks and valleys, a more refine measurement and data taking need to be applied and we have measured 300 points spread out on all parts of the IMCs.

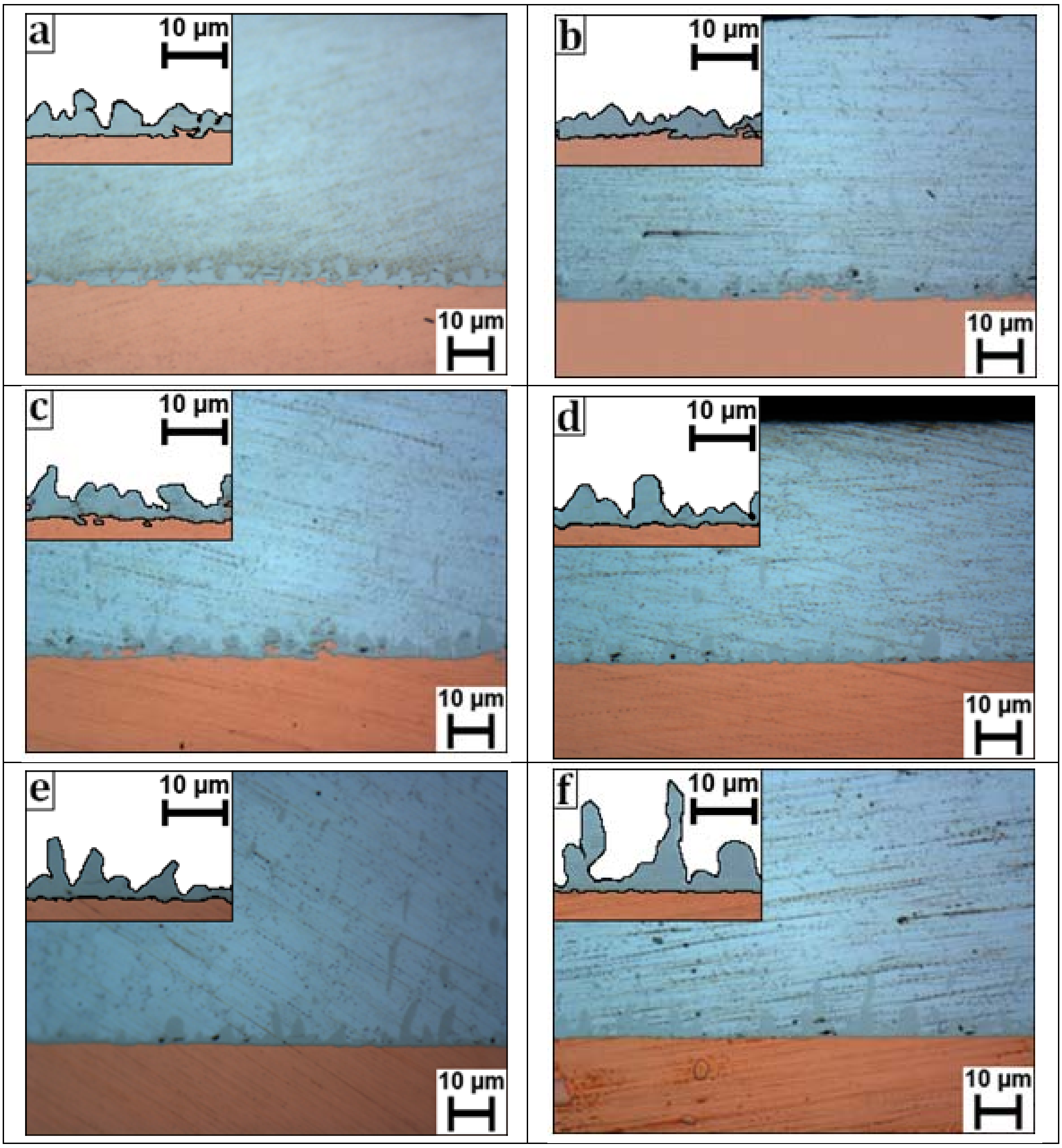

Fig. 5: Intermetallic growth of SAC305 on the Cu substrate for: (a) 0, (b) 200, (c) 400, (d) 600, (e) 800 and (f) 1000 cycles.

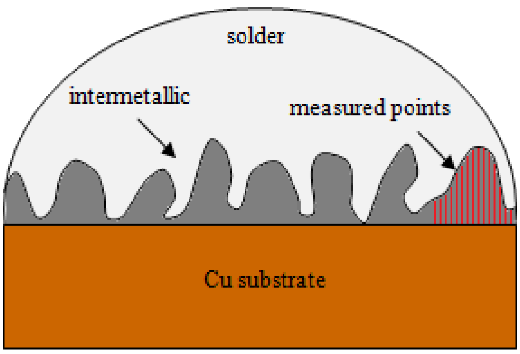

Fig. 6: Schematic illustration of measuring point of IMC thickness

Fig. 7 shows the average data of IMC on Cu substrate subjected to different thermal cycle (TC) test; the average data showing an increasing trend of IMC thickness from 3.95 µm for soldered sample without TC. The average thickness increased to 4.90 µm for 200 TC, slightly increased to 5.07 µm for 400 TC, 7.89 µm for 600 TC, 8.18 µm for 800 TC and 8.22 µm 1000 TC.

The median data of IMC on Cu substrate subjected to different thermal cycle (TC) test is shown in Fig. 8.Similar trend of median data with the average data, the IMC thickness is increased from 3.93 µm for 0 TC sample, 4.55 µm for 200 TC, 5.16 µm for 400 TC, 7.62 µm for 600 TC, 7.86 µm for 800 TC and 8.48 µm for 1000 TC. The median values for 400 TC and 1000 TC are slightly higher compared to average data.

Next is the mode data of IMC on Cu substrate exposed to different thermal cycle (TC) test as shown in Fig. 9.It is apparent from mode and average data where the IMC thickness for 0 TC sample is 3.93 µm, decreased to 3.67 µm for 200 TC and increased to 4.30 µm for 400 TC, 6.39 µm for 600 TC, 6.63 µm for 800 TC and 9.46 µm for 1000 TC. Higher value of mode data is observed for IMC thickness for 1000 TC.

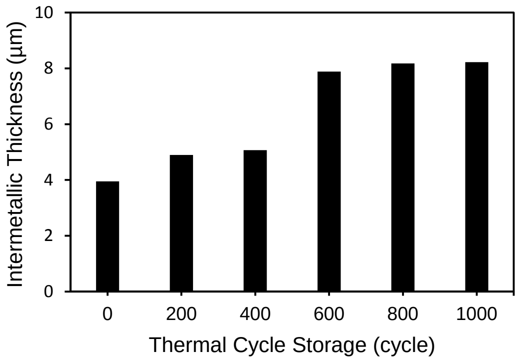

Fig. 7: Average data of IMC thickness on Cu substrate subjected to different thermal cycle test

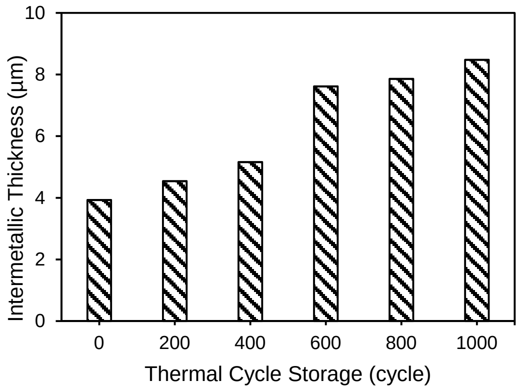

Fig. 8: Median data of IMC thickness on Cu substrate subjected to different thermal cycle test

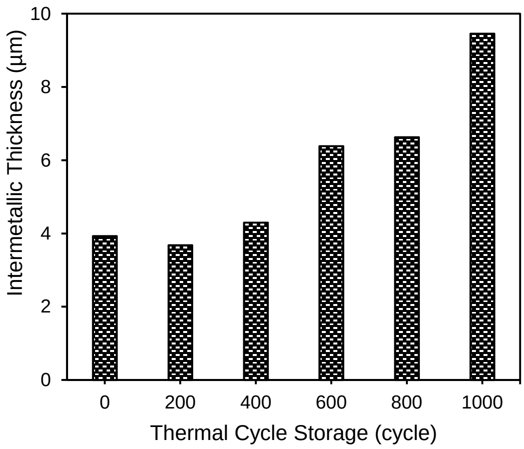

Fig. 9: Mode data of IMC thickness on Cu substrate subjected to different thermal cycle test

From the all IMC thickness data variation, it is clearly observed that different data are obtained. All of these types of data should be considered to determine the IMC thickness especially when the IMC growth structure is uneven or heterogeneous shape type. From all the data, the mode data more likely represent the IMC thicknesses as it represent the shape of IMC and often appear in most of the 300 measurements points taken.

4. Conclusion

Due to the variations of shape in 3D growth for IMC, conventional thickness measurements may not be able to give correct and precise value. A reliable thickness measurement method is crucial because the measured values will be used for activation energy calculation using Arrhenius relationship. Therefore, we have employed a statistical method of 300 measured points-values to ensure precise mode analysis. From this method, the thickness of IMC are represented by all three statistical values are average, median and mode. However, due to nature of IMC shapes (hillocks and valleys) that are being considered, the recommended thickness measurement is using mode value rather that median and average.

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