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WETTING PROPERTIES OF Sn-Pb, Sn-Zn AND Sn-Zn-Bi LEAD-FREE SOLDERS

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Abstract. Three studies on Sn-40Pb, Sn-9Zn and Sn-8Zn-3Bi Pb-free solders were conducted. Spreading of the solders was investigated on 0.13 µm roughness Cu substrate at 250°C. Sn-40Pb has a contact angle of 6°, which is seven times better than Sn-9Zn and Sn-8Zn-3Bi solders. Wetting balance was used to study the wetting time, wetting force and surface tension at different temperatures using two different fluxes. For Sn-8Zn-3Bi solder, MHS (commercial name) flux gives higher wetting force and wetting time compared to HCl flux for increasing temperatures. The surface tension of Sn-40Pb solder decreases with increasing temperature whereas the surface tension of Sn-9Zn and Sn-8Zn-3Bi solders is not influenced by the temperature. However, the addition of 3% Bi reduces the surface tension of the Sn-Zn-Bi system. A study on the spreading of Sn-9Zn solder alloy on Cu with surface roughness between 0.33 and 1.53 µm was also conducted. Surface roughness below 0.62 µm does not contribute to the improvement of wetting but above 0.62 µm, there was an improvement in the contact angle and spreading area.

Keywords: Pb-free solder, spreading, roughness, wetting properties, surface tension

Abstrak. Tiga kajian terhadap Sn-40Pb, Sn-9Zn dan Sn-8Zn-3Bi pateri tanpa plumbum telah dijalankan. Penyebaran pateri-pateri ini dikaji terhadap substrat Cu yang mempunyai kekasaran 0.13 µm pada suhu 250°C. Sn-40Pb mempunyai sudut sentuhan bernilai 6°, iaitu tujuh kali lebih baik berbanding pateri-pateri Sn-9Zn dan Sn-8Zn-3Bi. Tetimbang basahan digunakan untuk mengkaji tempoh dan daya basahan serta ketegangan permukaan pada suhu yang berlainan menggunakan dua fluks yang berbeza. Untuk pateri Sn-8Zn-3Bi, fluks MHS (nama komersial) memberi daya dan tempoh basahan yang lebih tinggi berbanding dengan fluks HCl untuk suhu yang meningkat. Ketegangan permukaan untuk pateri Sn-40Pb berkurangan apabila suhu meningkat tetapi suhu tidak mempengaruhi ketegangan permukaan untuk pateri Sn-9Zn dan Sn-8Zn-3Bi. Walau bagaimanapun, tambahan 3% Bi mengurangkan ketegangan permukaan sistem Sn-Zn-Bi. Satu kajian dilakukan mengenai penyebaran pateri aloi Sn-9Zn ke atas permukaan Cu bagi kekasaran permukaan antara 0.33 dengan 1.53 µm. Didapati bahawa kekasaran permukaan di bawah 0.62 µm tidak membantu dalam peningkatan basahan, tetapi kekerasan melebihi 0.62 µm meningkatkan sudut basahan dan luas penyebaran.

Kata kunci: Pateri tanpa plumbum, penyebaran, kekasaran, sifat-sifat basahan, ketegangan permukaan

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1.0 INTRODUCTION

Lead (Pb) has been used for a wide variety of applications ranging from plumbing to electronics for many years. Pb, combined with tin (Sn), form a low-melting-point material that is easy to process, has good electrical conductivity, good reliability and low cost as soldering materials [1]. Furthermore, Sn-Pb solders have the advantage of mechanical properties and excellent wetting on copper. Unfortunately Pb and its compounds are toxic to the human body and cause serious environmental problems. So, many countries are going to ban this substance in the near future [2]. Thus, the development of Pb-free solders with the same properties as Pb-based solders is in progress [3].

For developing new Pb-free solders, various properties such as wettability, microstructure, mechanical properties, good manufacturability, affordable cost as well as the reliability of soldered joints must be taken into account [2]. The first consideration, in finding a suitable Pb-free solder is the melting temperature of the alloy must be near to the Sn-37Pb solder, i.e. 183°C [2,4].

Although several commercial and experimental Pb-free solder alloys exist, none complies all the standards requirement. Current processing equipment and condition (involving fluxes) have been optimized for Sn-Pb solder alloys. So, the development of proper alloy for the new solder system, with suitable fluxes and assembly processes for Pb-free solders, is also needed [5].

Among the Sn-based solder alloys, the Sn-9Zn has been considered as a suitable candidate because of its low melting temperature of 198°C [6]. However, since Zn-containing alloys have the problems of oxidation and corrosion, new Sn-Zn based alloys with the addition of Bi might be a potential candidate [2].

In the electronics industry, how well the liquid solder wets the substrate is a topic of fundamental interest. In addition, surface characteristic, for example the surface roughness influences the wetting behaviour. The substrate surface roughness will influence the surface energy and the wetting behaviour of the reacting liquid/solid interface. By roughing the surface, additional surface area is introduced and this can be regarded as effectively causing an increase in its surface energy. Wenzel [7] found out that the experimental contact angle decreases with the roughness when the contact angle is less than 90°. So, the influence of surface roughness on the spreading of liquid solder is investigated in this paper. In our previous research [8], it was found that Sn-40Pb and Sn-8Zn-3Bi solders did not show any improvement in wetting force but Sn-9Zn solder shows some improvement in wetting force for increasing dwell time.

There are two main methods to conduct wettability test. One is spreading (reflow) method: putting a solder disc on a substrate, so as to achieve soldering by heating the substrate above the solder melting temperature. Another is the dipping method by which the solder joints were fabricated by dipping the substrate into the liquid solder. In this paper, Sn-9Zn and Sn-8Zn-3Bi lead-free solders were studied and Sn-40Pb solder was used as reference. The spreading method (reflow) was used to study the contact angle on clean Cu Substrate for the three solders. The contact angle and spreading area were investigated for Sn-9Zn solder after spreading on different Cu surface roughness. The dipping method was used to study the wetting properties at different temperatures using two different fluxes.

2.0 THEORY

2.1 Spreading of Solder on Base Metal (Reflow Method)

Solder spreading on a substrate is fundamental for the development of new solders. It is a complex phenomenon involving joint configuration, flux selection, substrate finishing and many other factors [9]. When the solder melts, the increase in contact area indicates the wetting behavior of solder [10]. The extent of wetting is measured by the contact angle that is formed at the juncture of a solid and liquid (molten solder), as shown in Figure 1.

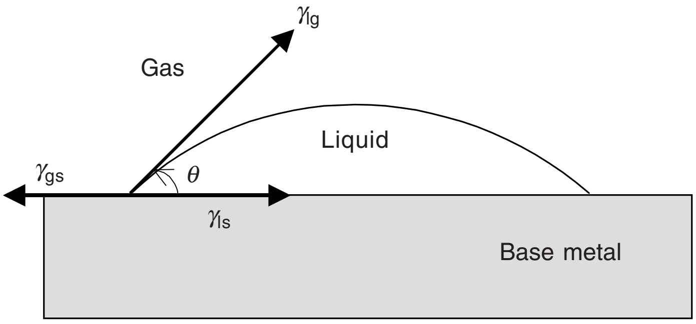


Figure 1 Schematic diagram of thermodynamic equilibrium in wetting

The contact angle is determined from the balance of surface tension at the junction. For a non-reacting system, the contact angle (θ_Y) is predicted by Young's equation:

$$\gamma_{gs} = \gamma_{ls} + \gamma_{ls} \cos\theta_Y \tag{1}$$

$$\cos\theta_Y = \frac{\gamma_{gs} - \gamma_{ls}}{\gamma_{lg}} \tag{2}$$

where γ_{gs} is the surface tension of the solid in the particular environment, γ_{ls} is the surface tension between the solid and the liquid, and γ_{lg} is the surface tension of the solid in the same environment. When good wetting occurs, the wetting angle should be small (i.e. γ_{ls} and γ_{lg} smaller and γ_{gs} larger).

2.2 Dipping Test Using Wetting Balance

The dipping test using wetting balance measures the forces imposed by the molten solder on the test specimen as the specimen is dipped into and held in the solder bath. The wetting force is measured as a function of time and is plotted. Three of the most commonly applied wetting indices are the wetting time (T_o), the maximum wetting force (F_{max}) and the withdrawal force (F_w). A typical wetting curve is shown in Figure 2 [11]. The wetting time, T_o, is the moment when the wetting force is equal to buoyancy force. When the substrate was dipped into the molten solder, initially the force is negative. This indicates that the solder has not yet begun to wet the specimen and, in fact shows a buoyancy effect. The force exerted by the solder approaches zero as the solder begins to wet the specimen. The T_o is the time to cross the zero axis of wetting force. This point indicates the transition from not-wetting ($F < 0$) to wetting ($F > 0$). The maximum wetting force, F_{max}, can be obtained when the meniscus shows a stable state after complete immersion and the measured force remains constant.

In a static equilibrium conditions, the wetting force, F, can be expressed as follows [12]:

$$F = P\gamma\cos\theta - \rho g V \tag{3}$$

where P is the perimeter of the specimen, γ is the surface tension of the solder in contact with the flux, θ is the contact angle, ρ is the density of the solder, g is the gravity acceleration constant and V is an immersed volume.

The steep rise to the withdrawal force curve means increase of buoyancy force by withdrawal process, and the top point shows that sliding solder along the side of the sample meets the bottom corner, and contact angle falls down to zero. Subsequently,

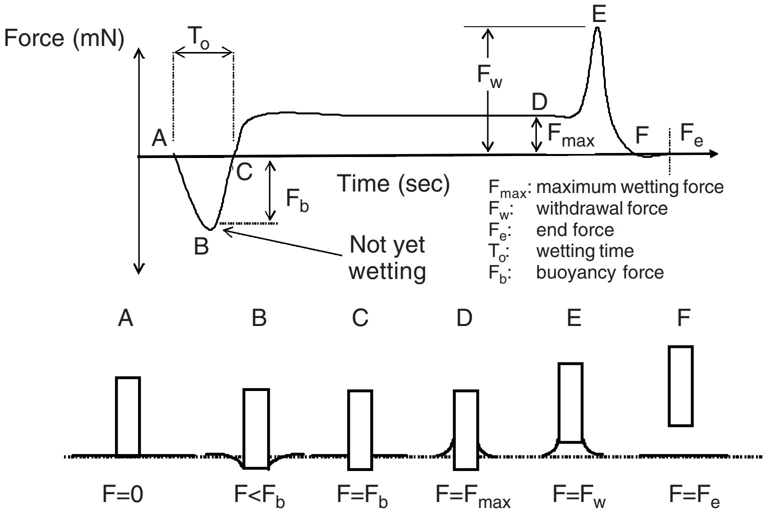


Figure 2 A typical wetting curve and wettability indices [11]

the interface between the solder and the sample changes from side edge to the bottom edge of the sample, and test ends when necking of the solder, leads to the detachment between the solder and substrate. If we assume that static equilibrium is applied to the above theory, the force at the top point of the withdrawal force curve can be expressed as follows:

$$F_{\text{withdrawal}} = P\gamma \tag{4}$$

Because of zero contact angle and buoyancy forces, the surface tension is the only variable for determining the wetting force.

3.0 MATERIALS AND METHODS

Alloys of Sn-40Pb, Sn-9Zn and Sn-8Zn-3Bi were used in the present work. Sn-9Zn alloy were prepared using Sn bar and Zn granules. Sn was melted in an alumina crucible at 500°C and followed by addition of Zn granules and the mixture was stirred to homogenize and was let to cool in the furnace. Sn-40Pb is commercially available solder and Sn-8Zn-3Bi are supplied by Nihon Almit Co. Ltd., Japan.

3.1 Preparation of Solder Disc and Spreading Test

Sn-40Pb, Sn-9Zn and Sn-8Zn-3Bi solders were remelted to 400°C and poured into a mold to make a disc with 6 cm in diameter and 3 mm in thickness. Then the disc was polished until the thickness is 2 mm. By using a puncher, the disc was sliced into 5 mm in diameter cylinder with mass around 0.4 g.

The spreading test was done using Solder Checker Instrument model SAT-5100, Rhesca. The Cu substrate used was 30 mm × 30 mm with 0.4 mm in thickness. The solder disc was placed on a clean Cu substrate and it was positioned on top of the Sn-37Pb solder bath for 20 seconds. HCl based flux was used in this experiment and the temperature of the solder bath was 250°C (Figure 3). Each test was repeated twice

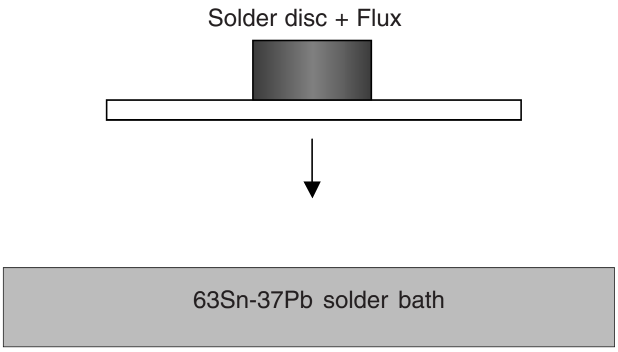
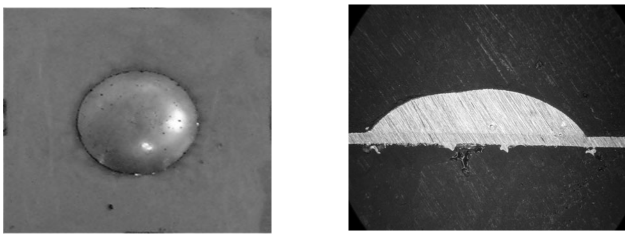


Figure 3 Schematic diagram of the spread test



(a) (b)

Figure 4 Schematic diagram of the spread test

(which gives eight contact angles). After the test, flux was cleaned using hot water. The samples were cross sectioned by a low speed diamond cutter and mounted using epoxy. The mounted samples were polished with silicon carbide coated papers to smooth the surface. The cross sectioned samples were photographed as shown in Figure 4. The contact angles were measured using Image J software.

3.2 Dipping Test

The dipping test was done using the Solder Checker Instrument model SAT-5100 Rhesca. Cu substrate with (10.0 ± 0.5) mm in width and 0.4 mm in thickness were used. Sn-40Pb, Sn-9Zn and Sn-8Zn-3Bi solders were tested. Prior to wetting investigation, the specimens were dipped in HCl based flux for 10 seconds. For Sn-8Zn-3Bi solder the test was repeated using MHS flux. This flux is an organic rasin-based with small addition of surface active agents. The wetting balance investigation was performed at an immersion rate of 5 mm/s, immersion depth of 5 mm and immersion time of 20 seconds with temperature varying from 220 to 330°C.

3.3 Preparation of Surface Roughness and Spreading Test

The spreading test was repeated for Sn-9Zn solder using different surface roughness on Cu substrate. Sand blasting technique was used for preparation of the rough surface. Various sizes of sand were sieved and listed in Table 1. A commercially available sand blaster was used to blast the sand on the Cu substrate. The Cu substrate used was 30 mm × 30 mm with 0.4 mm in thickness. It was placed 5 cm from the blaster. The blasting was done in a zigzag direction for 30 seconds. For each range of sand sizes, the blasting was repeated twice. The roughened Cu substrates were ultrasonically cleaned with water. Then, the surface roughness was measured using Surface Measuring Instrument model Surftest SV-400, Mitutoyo. The scanning was done in three different parallel directions and the average value was calculated and listed in Table 1. The Ra mode was used by which it is the arithmetical mean of the areas between the surface profiles and the mean line area of the evolution. The spreading area and the contact angle were measured using Image J.

Table 1 Sand size and surface roughness

Sand size (μm)	**Surface roughness, R_a (μm)**
Smooth surface (No blasting)	0.13
< 45	0.33
63 to 75	0.62
75 to 90	0.90
90 to106	0.93
106 to 125	1.21
> 125	1.53

4.0 RESULTS AND DISCUSSION

4.1 Spreading Test

Figure 5 shows the variations in contact angles on a smooth Cu substrate for Sn-40Pb, Sn-9Zn and Sn-8Zn-3Bi solders. Sn-40Pb solder has a very low contact angle of 6° compared to Sn-9Zn and Sn-8Zn-3Bi solders which have 39.7° and 40.9°, respectively. In this case, the addition of Bi increases the contact angle, hence reduces the wettability.

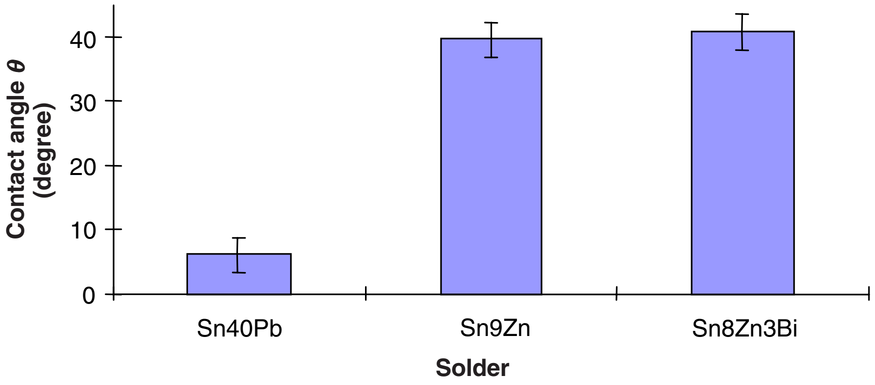


Figure 5 Contact angle on Cu substrate without sand blasting

4.2 Dipping Test and the Effect of Temperature

Figure 6 shows that the wetting time decreases as solder temperature increases. This is due to better removable of flux from the Cu surface. The HCl flux evaporates more easily and reduces preheating time and the potential for oxidation. For Sn-8Zn-3Bi solder, MHS flux gives higher wetting time compared to HCl based flux. This shows the reaction kinetics and the evaporations of the flux from the Cu surface is slower.

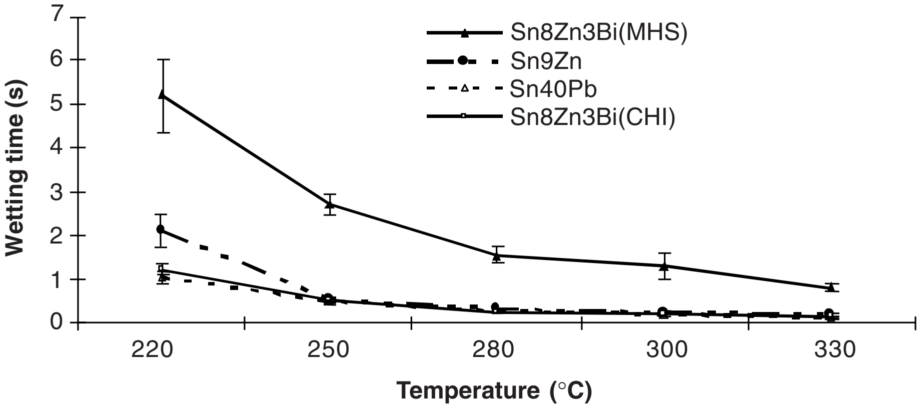


Figure 6 Wetting time at different temperatures using HCl and MHS fluxes

The MHS flux increases preheating time which could increase the potential for oxidation. For example, a wetting time of 0.5 second is needed at 250°C for all the three solders when using HCl flux. Nevertheless, applying MHS flux gives rise to a wetting time of 5.5 seconds. Wetting is governed by the interfacial interaction between the deposit and the solders. Thus, the thickness of the deposit is not expected to have any significant effect on the wetting time. The wetting time results show that the reaction temperature significantly enhances the wetting rate. The MHS flux degrades the wetting kinetics as evidenced by the increasing wetting time for Sn-8Zn-3Bi solder.

Temperature variation also affects the wetting force, surface tension of solder and contact angle, and has a practical importance because it helps us to determine the optimum temperature condition in the soldering system. The interaction between molten solder and Cu substrate is a complex phenomenon. The types of interaction and the physical force, such as interfacial tension, between the molten solder and Cu substrate govern the wetting force [13]. Maximum wetting force (F_{max}) and withdrawal force (F_w) are shown in Figures 7 and 8. For Sn-40Pb (HCl flux) solder, F_{max} increases slightly as temperature increases while F_w decreases. This counteraction can be explained qualitatively using Equations (3) and (4). According to Equation (3), temperature variation affects the surface tension, the contact angle, and density of the solder, which in turn affect the wetting force. Density of the solder is a variable that can change the buoyancy force as temperature varies, but change of density due to temperature change is very small compared with the values of wetting force. Therefore, the buoyancy effect as a result of density change is negligible in this case [14]. Generally, the surface tension and contact angle between sample and solder decrease as temperature increases. From Equation (3), decrease in the surface tension reduces the wetting force and on the other hand, decrease of contact angle increases the wetting force because of the cosine value. Therefore, increase of F_{max} as the result of temperature increase means that the effect of contact angle is stronger than that of the surface tension.

In the case of withdrawal force (F_w), only the surface tension can change the value of F_w in Equation (4). According to Equation (4), the value of the surface tension can be directly obtained by dividing the maximum withdrawal force by the sample perimeter. The values of withdrawal force from Figure 8 are used with Equation (4) to calculate the surface tension of solders as various temperatures and it is plotted in Figure 9. The fact that F_w tends to decrease as the temperature goes up implies that its surface tension is inversely proportional to temperature. This argument is in agreement with Figure 9 for Sn-40Pb solder.

In the case of Sn-9Zn (HCl flux) solder, F_{max} increases slightly as the temperature increases, and this result is the same as in Sn-40Pb solder. However, variation of F_w under different temperature conditions is very small as can be seen from Figure 8. If we adopt the same theory for Sn-40Pb, it may be concluded that effect of contact angle is much stronger than that of surface tension. F_w however, remains constant under different temperature conditions. This fact shows good agreement with the almost constant values in the surface tension under different temperature conditions for Sn-9Zn solder.

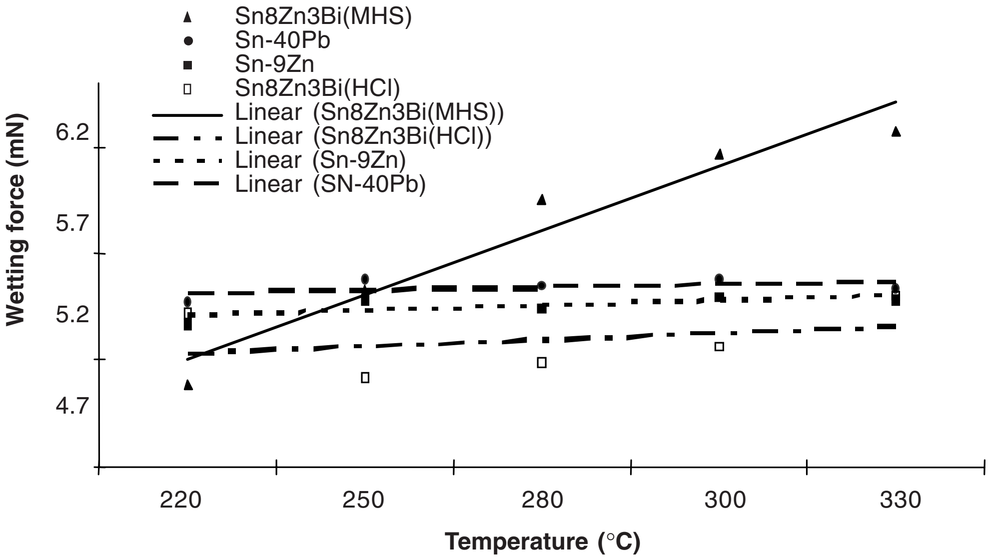


Figure 7 Maximum wetting force at different temperatures using HCl and MHS fluxes

Sn-8Zn-3Bi solder gives similar results as Sn-9Zn solder when HCl flux was used. On the other hand, the wetting force of Sn-8Zn-3Bi increases in proportion to temperature when MHS flux was used. Although the above theory applies here, the flux plays a very important role in increasing the wetting force and provides wetting enhancement functions [15]. Sn-8Zn-3Bi solder has a lower surface tension than Sn-9Zn solder which is due to the addition of Bi to the system.

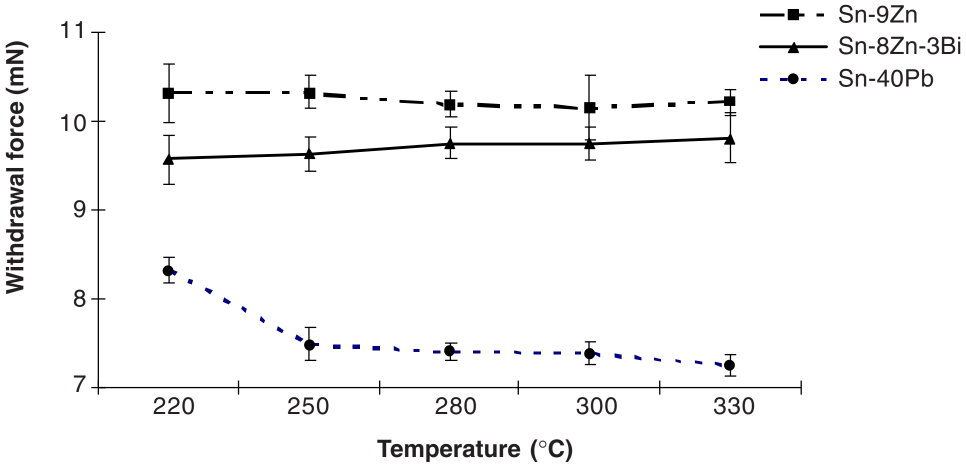


Figure 8 Withdrawal force at different temperatures using HCl flux

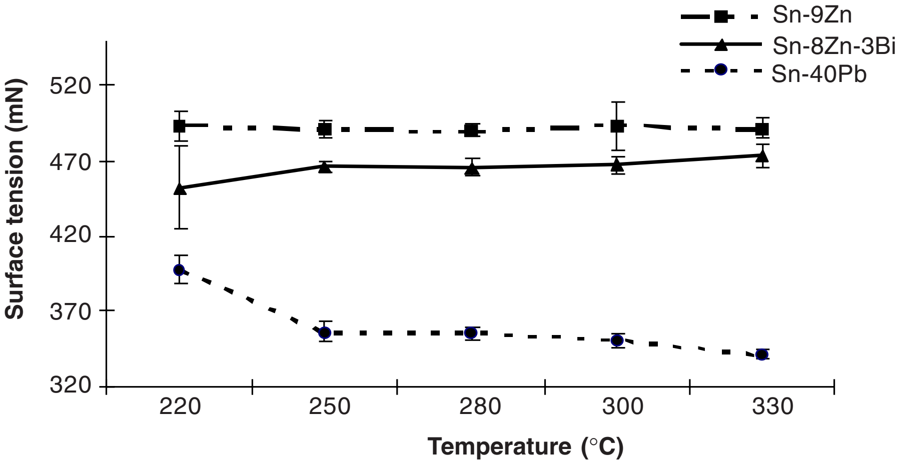


Figure 9 Surface tension at different temperatures using HCl flux

4.3 Roughness Test

Table 1 shows the variations in surface roughness on the Cu substrate for different sand sizes used. The roughness increases when the sand size increases. Figure 6 represents the correlation between surface roughness, contact angle and spreading area for Sn-9Zn solder. The smooth surface has the lowest contact angle of 38.5°. The increase in roughness increases the contact angle but when the roughness exceeds 0.62 mm, the contact angles begin to decrease. Wenzel [7] proposed an equation (Equation 5) that describes the contact angle between liquid and rough surface, θ_w:

$$\cos\theta_w = \bar{r}\cos\theta_Y \tag{5}$$

where $\bar{r}$ is the average roughness ratio, defined as the factor by which roughness increases the solid-liquid interfacial area. The derivation of this equation was similar to that of Young's equation, except that the apparent area of the solid surface was multiplied by $\bar{r}$. Thus, the Wenzel's equation is supposed to give the relationship between apparent and intrinsic contact angles (in the absence of line tension effect) [16]. In general, the results of Figure 10 obey Wenzel's equation when the surface roughness is above 0.62 µm where the contact angle decreases with the increase of surface roughness.

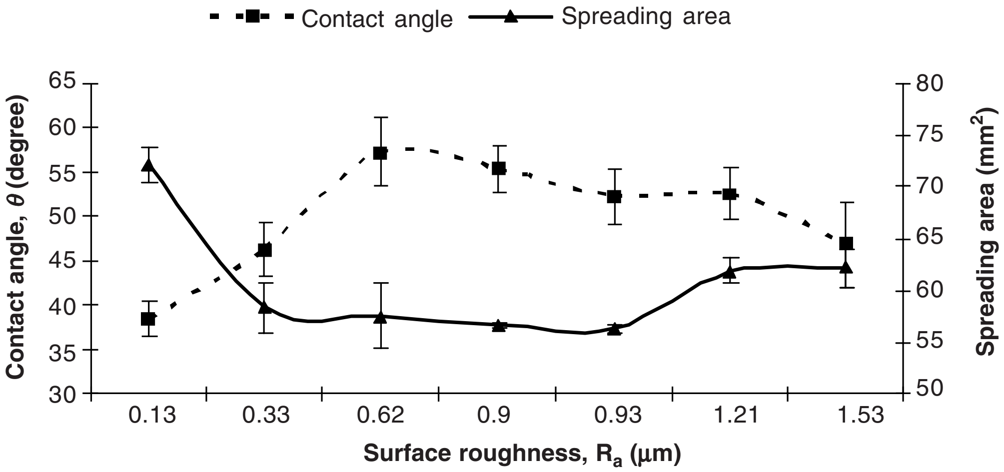


Figure 10 Contact angle and spreading area for Sn-9Zn on rough Cu surface

Over the years, a lot of efforts were made to understand the effect of surface roughness on wetting [17-21]. However, for reacting system, controversy still exists and the effect of surface roughness on dynamic wetting is not well-clarified yet. Furthermore, it has long been known in the solder industry that partial substrate dissolution occurs during solder wetting on metal substrate and intermetallic formation is almost always observed in practical solder-substrate systems. In addition, the reactive surfaces present on the liquid solder and metal substrate are highly susceptible to contamination through adsorption, reaction and diffusion processes. In fact, the primary purpose of the fluxing agent commonly used in soldering processes is to remove existing surface oxides and prevent their recurrence until after the formation of the solder joint is completed [22].

5.0 CONCLUSION

At 250°C, Sn-40Pb solder has a very low contact angle of 6° compared to Sn-9Zn and Sn-8Zn-3Bi solders which have 39.7° and 40.9°, respectively. For the three solders used, wetting time decreases when the reaction temperature increases. For Sn-8Zn-3Bi solder, the MHS flux gives higher wetting time compared to HCl based flux. The HCl flux does not improve the wetting force at higher temperatures. Whereas, the wetting force for Sn-8Zn-3Bi solder increases in proportion with temperature when MHS flux was used. For Sn-9Zn and Sn-8Zn-3Bi solders variation of the surface tension under different temperatures is very small but on the other hand the surface tension of Sn-40Pb decreases with increase in temperature. The surface tension of Sn-9Zn solder is too large to provide a good wetting property and the addition of 3% Bi reduces the surface tension of Sn-Zn-Bi system. The experimental results indicated that flux affects the wetting kinetics of the solders on Cu substrate. It significantly affects the wetting time and wetting force of the Sn-8Zn-3Bi solder. For Sn-9Zn solder, surface roughness below 0.62 mm does not contribute to the improvements of wetting. But above 0.62 mm, there was an improvement in the contact angle and spreading area.

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