

Title	Conditions for the Measurement of Surface Tension of Solders with a Wetting Balance Tester(Materials, Metallurgy & Weldability)
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Citation	Transactions of JWRI. 1997, 26(1), p. 81-84
Version Type	VoR
URL	https://doi.org/10.18910/11738
rights	
Note	

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Conditions for the Measurement of Surface Tension of Solders with a Wetting Balance Tester[†]

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Abstract

A method for measuring the surface tension of solder alloys has been developed by using a wetting balance tester and the measuring conditions have been examined. A non-wetting rod was immersed into a molten solder bath at a fixed speed, and the surface tension was calculated by analyzing the measured force-immersion depth relationship. The theoretical model is based on the hypothesis that the meniscus configuration of molten solder gradually varies depending on the depth of the immersing rod and its radius, and the configuration shows a stable form when the depth reaches a critical value. After exceeding the critical depth, the force increases in proportion to the immersion depth of rod. Therefore, the surface tension can be measured by analyzing the force-time curve produced. It was found that the most suitable non-wetting material for measurement was Al_2O_3 . It is important for obtaining an accurate surface tension to adopt a low immersion rate and to dry the rod after fluxing. Soldering flux reduced the surface tension of Sn-Pb solder and lead-free solders.

KEY WORDS: (Surface tension) (Wetting balance tester) (Tin-lead eutectic solder) (Lead-free solder)

1. Introduction

In the modern electronics industry, packages and pitches of conductor and solder land are becoming smaller and smaller due to the miniaturization and reduction of components^{1), 2)}. Consequently, the parts to be soldered become minute and miniaturized and suffer problems such as the displacement of minute chip components and tomb stones, and furthermore, solder bridging to short-circuit the neighboring conductors on a printed circuit board. These problems are associated with the surface tension of molten solder alloy³⁾. To prevent these defects, the application of activated fluxes, soldering in an inert environment, etc. are proposed⁴⁾ and seem to be effective because of the improvement of wettability and reduced contact angle due to the decrease of surface tension of the molten solder.

Furthermore, the use of lead is going to be regulated in the electronics industry because of its toxicity⁵⁾. Therefore, researches to develop new lead-free solders which do not contain lead have been

pursued⁶⁾. However, the wettability of lead-free solder is usually inferior to that of the Sn-Pb system solder⁷⁾. The deterioration in the wettability of lead-free solder has the possibility of increasing soldering defects. However the physical properties which are related to wettability, especially surface tension of lead-free solders, has not been sufficiently clarified. Therefore, if there is a simple accurate method to measure the surface tension of solder alloys, the method will be useful to develop new lead-free solders with improved wettability. In the present work, a simple method to measure the surface tension of solder alloys has been developed by using a wetting balance tester and the experimental conditions have been examined.

2. Experimental method

2.1 Principle

When a rod is immersed into a liquid which does not wet it, the liquid surface is pushed down and the liquid surface configuration is determined by the balance between gravity and surface tension according

[†] Received on May 19, 1997

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Transactions of JWRI is published by Joining and Welding Research Institute of Osaka University, Ibaraki, Osaka 567, Japan.

to the immersion depth of the rod. In the case of a non-wetting substance, it receives a reaction force which is proportional to the volume of the liquid excluded. By using this principle, the surface tension can be measured.

When a non-wetting rod is vertically immersed into the molten solder bath with a fixed temperature at a fixed speed, the meniscus configuration formed on the rod shows a stable size if the rod is immersed to a certain depth. Until the non-wetting rod is immersed to the depth where the meniscus configuration shows a stable size, the volume of liquid excluded by the non-wetting rod increases according to the immersion depth of the rod so that the reaction force acting on the rod increases correspondingly. Assuming that this reaction force can be measured by the increase of both contact angle and buoyancy, the relation among them will be as the following equation.

$$f = -\pi D \gamma \cos \theta + \rho g h A \quad (1)$$

where, f is the reaction force to act on the rod and the direction to push up the rod is "positive" (+) in this test. D , A , θ and h refer to the diameter, sectional area of the immersed rod, contact angle and depth of immersion, respectively. g is gravity. In the case of no wetting between the rod and liquid, the reaction force in equation (1) can be expressed as equation (2), because as the test piece is lowered into the molten solder by keeping the same contact angle of π rad ($\cos \theta = -1$) after it reaches π rad., only buoyancy increases in proportion to the depth of rod.

$$f = -\pi D \gamma + \rho g h A \quad (2)$$

By measuring the reaction force above, both surface tension and density of the molten solder can be obtained.

2.2 Materials

The solder alloys used for measurement were Sn-37Pb alloy and Sn-3.5Ag-5Bi alloy, one of the candidate lead-free solder substitutes for Sn-37Pb alloys. Alumina, stainless steel, Teflon, vitreous silica, carbon and Pyrex glass rods were used in order to select an immersing material which would be non-wetting with the molten solder. The size of the rods used was 100mm length and 3mm diameter. The measurement was carried out in an air atmosphere without using any flux and also with a flux to investigate its effect on the surface tension of molten solder. The fluxes used were rosin (R) and activated flux (RA) with 0.2 % chlorine. Isopropyl alcohol was used as a solvent to adjust the solid content to 35%. Flux was coated onto the rod by immersing into flux by a constant depth.

2.3 Method for measurement of surface tension

The detailed illustration of the measuring method for both surface tension and density in accordance with the principles above is shown in Figure 1. Using a wetting balance tester it is possible to measure the variation of the force acting on a test piece as time passes. The curve of the time-force relation when the rod test piece is immersed into the molten solder at a fixed speed has been measured with a computer system. The sampling intervals for measurement were 0.01 s. The time-force curve can be converted into a force-immersion distance curve under the condition of a fixed immersion speed and this is shown in Fig. 1(a). After obtaining the straight line part of the force-immersion distance curve from sampled data by the least involution, the surface tension can be calculated from the intercept ($\pi D \gamma$) of this equation and the density from its gradient ($\rho g h A$). The measurement of Sn-Pb and Sn-Ag-Bi solders was carried out at the temperatures of 503 K and 513 K.

3. Results and Discussion

For the exact measurement of the surface tension in this research, it is important and indispensable that the immersion test piece does not become wet with molten solder, in other words, the contact angle θ should be π rad. To achieve this purpose, an immersing material which never becomes wet with molten solder was selected. The measurement was carried out using both Sn-37Pb and Sn-3.5Ag-5Bi solders at the immersion speed of 0.1mm/s without using any flux and

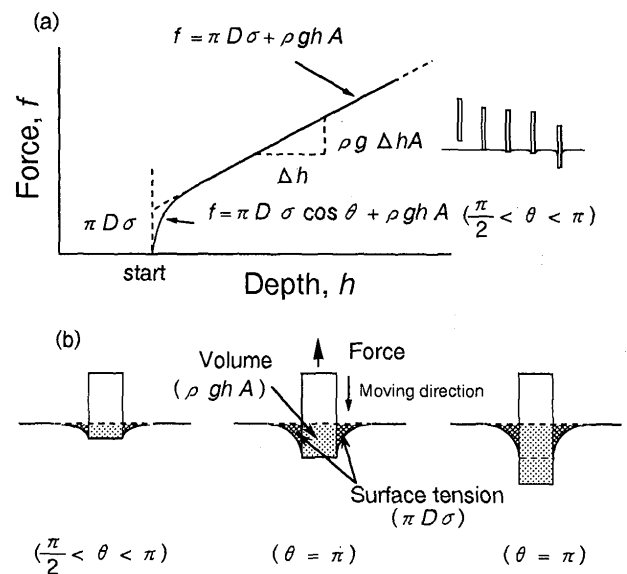


Fig. 1 Method for measurement of surface tension and density of solder, (a) force distance curve measured by wetting balance method, (b) illustration of immersion of non-wetting rod into molten solder.

Table 1 Evaluation of immersion materials for measurement of surface tension with the obtained surface tension and density.

Materials	Flux	Sn-37Pb (503 K)			Sn-3.5Ag-5Bi (513 K)		
		Undesirable wetting*	Surface tension (N/m)	Density (kg/m ³)	Undesirable wetting*	Surface tension (N/m)	Density (kg/m ³)
SUS 304	No flux	×	0.569	8020	×	0.513	7870
	R flux	○	0.399	8520	○	0.415	7370
	RA flux	×	0.362	8730	○	0.391	7580
TEFLON	No flux	×	0.508	9140	×	0.510	8900
	R flux	○	0.411	8910	○	0.421	8060
	RA flux	○	0.407	8940	○	0.432	7820
SiO ₂	No flux	×	0.550	8200	×	0.580	7110
	R flux	○	0.403	8110	×	0.400	7370
	RA flux	×	0.362	8130	×	0.422	7040
Carbon	No flux	○	0.494	8070	○	0.452	7960
	R flux	○	0.477	8430	×	0.416	7800
	RA flux	○	0.421	8860	×	0.415	7660
Pyrex glass	No flux	×	0.518	8050	×	0.511	7680
	R flux	○	0.364	8430	×	0.382	7870
	RA flux	○	0.332	8100	×	0.369	7620
Al ₂ O ₃	No flux	○	0.506	8430	○	0.542	7430
	R flux	○	0.384	8170	○	0.411	7350
	RA flux	○	0.379	8350	○	0.407	7580

(* : ○; Absent, ×; Present)

Table 2 Surface tension measured by previous work.

Solder	Temperature (K)	Flux	Surface tension (N/m)	ref.
Sn-37Pb	553	-	0.490	8)
Sn-40Pb	533	-	0.481	9)
Sn-40Pb	523	R flux	0.410	10)
		R+0.2%Cl(RA)	0.380	
Sn-37Pb	503	(Calculation)	0.459	11)
Sn-3.5Ag-5Bi	513	(Calculation)	0.501	

with flux RA. The results are shown in **Table 1**. The calculated values of surface tension calculated by using thermodynamics from other references are shown in **Table 2**. In Table 1, the mark ○ means non-wetting with molten solder and mark × means the adhesion of molten solder to the test piece. In the case of stainless steel, Teflon, vitreous silica and Pyrex glass rods for both Sn-37Pb and Sn-3.5Ag-5Bi solders without any flux used and also for Sn-3.5Ag-5Bi solder partly with flux, they are all wetted by molten solder during their measurement so that a desirable force-immersion depth curve can not be obtained. The surface tension values obtained are larger than those shown in Table 2. Furthermore, a carbon rod with flux is wet with molten solder. As distinct from the case of other materials, the surface tension without flux becomes larger than the surface tension with flux. However, for an alumina rod among the materials used this time, it is neither wet nor adhered to by molten solder and a desirable force-immersion depth curve is obtained. Comparing the

measured values with those in Table 2, the measured values in the case of an alumina rod well conform to those in the references so that the surface tension can be satisfactorily measured with an alumina rod. Therefore, an alumina test piece is used for the measurement of surface tension hereafter.

For the sake of checking the effect of immersion conditions of an alumina rod test piece, the measured data obtained using Sn-37Pb solder with flux RA by changing an immersion speed are shown in **Figure 2**. The flux was dried with a hot air dryer prior to the measurement in the case of the immersion speed of 0.1mm/s but not dried in the case of that of 2mm/s. As it can be seen in Fig. 2(a), when the immersion speed is fast without drying of the flux, a rapid decrease of reaction force at a certain time was noticed. This is believed to be caused by the evaporation of the isopropyl alcohol into the atmosphere as bubbles accompanied the immersion of a test piece. Since the dispersion of both surface tension and density values is large, even if measured in such a way, the measurement was also carried out in the condition with an immersion speed of 0.1 mm/s and with isopropyl alcohol evaporated using a hot air dryer after the supply of the fixed amount of flux. The data measured in this test condition are shown in Fig. 2(b). With these conditions, it becomes possible to obtain stable data to avoid the rapid fluctuations of force caused by the evaporation of isopropyl alcohol. It can be concluded

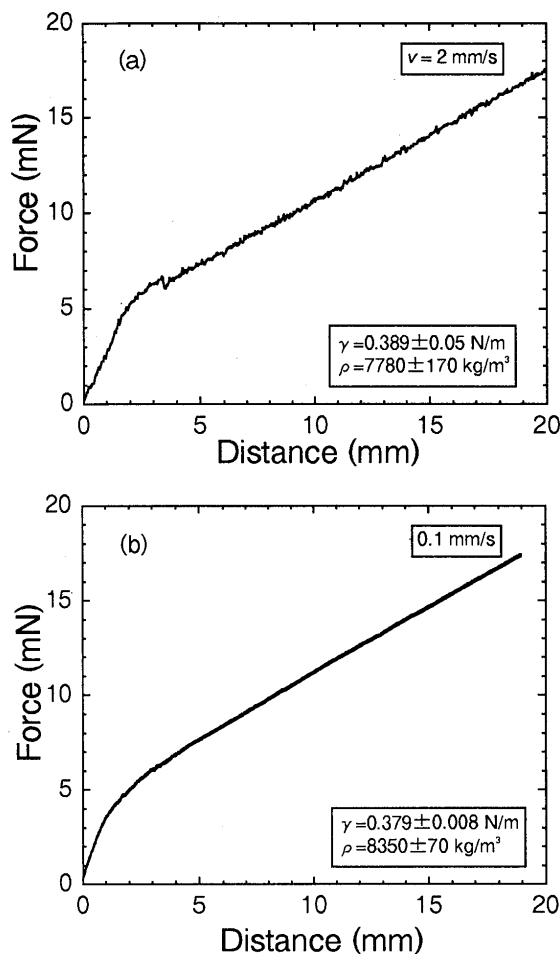


Fig. 2 Effect of immersion conditions on force-distance curve obtained by Sn-37Pb solder with RA flux at 503 K; (a): 2 mm/s without drying flux, (b): 0.1 mm/s after drying flux.

that the variation of force seen in Fig. 2(a) is clearly related to the generation of gas bubbles stemming from the evaporation of the solvent in the flux. The dispersion of surface tension values is then smaller as a whole compared with those in Fig. 2(a) which shows the stable results of measurement. It is understood then that the important test conditions are to sufficiently dry the flux and to use an immersion speed slower than 0.1 mm/s.

On the other hand, an increase of density is noticed by making the immersion speed slower. This seems to be caused by the increase of volume of molten solder to be excluded due to the oxidation of solder surfaces. The density increases as compared with that at room temperature and increases further because of the use of flux. This seems to be caused by the invasion of flux into the gaps between the rod and molten solder and a reconsideration of the measuring method of density is required hereafter.

4. Conclusions

By immersing non-wetting rods into molten solder at a fixed speed and measuring the reaction force acting on the rod, a method to measure both surface tension and density has been developed and the measured results with a wetting balance tester will be summarized as follows.

- (1) An alumina rod is the most appropriate as a non-wetting material among those used for measurement in this research. Furthermore, when using flux, stable results of measurement could be obtained by drying the flux and making the immersion speed slower.
- (2) The measurement of surface tension of both Sn-37Pb and Sn-3.5Ag-5Bi solders is possible and the surface tension decreases in accordance with the efficiency of the flux if it is used. Furthermore, the measured values well conform to those in the references and it has been found that this method can easily be used for measuring surface tension.
- (3) The densities measured by this method show larger values than those at room temperature and shows further larger values using flux. This seems to be caused by the invasion of flux into the gaps between the rod and molten solder and it has been found that a reconsideration of the measuring method of density is required.

Acknowledgment

The authors would like to express their hearty thanks to Shorai Foundation for Science and Technology for the financial support of this work.

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