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Wetting behaviour of SAC305 solder on different substrates in high vacuum and inert atmosphere

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Abstract The wettability between solder and substrate is a very important issue in reliability of soldering process. The contact angle θ is used to measure the degree of wetting. The contact angle of lead-free alloy Sn-3 % Ag-0.5 % Cu (wt%) was measured, as a function of temperature, for three different commercial surface finish substrates used in printed circuit boards (PCB): Sn, NiAu and Organic Solderability Preservative (OSP). The measurements were performed by the sessile drop method in two different atmospheres: high vacuum and inert gas. In high vacuum the results showed that on substrates of NiAu and OSP the solder started spreading suddenly at 225 °C and in Sn substrate the contact angle decreases slightly with temperature. In inert gas atmosphere the results showed different behaviours: the contact angle between molten solder in OSP and NiAu substrates is sensitive to temperature; and, in Sn substrate, the contact angle does not change with temperature. The NiAu and OSP substrates showed a better degree of wettability than Sn substrate, in inert atmosphere.

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1 Introduction

The soldering process consists in the formation of a joint between the molten solder and the metal surface. Thus, the ability of the molten solder to flow or spread on the substrate is important for the formation of a proper metallic bond [1–4]. For these reasons, the study of wetting behaviour is a crucial step in the characterization of solder alloys [2, 5–9].

The wettability is the ability of a liquid to spread over a solid surface [7, 10, 11] and refers to the capacity of the molten solder to react with a substrate, at the interface, to form a certain amount of intermetallic compound that acts as an adhesion layer to join the solder and the substrate [12]. The spreading of the solder over a metallic surface is a complex problem, that involves several physical and chemical processes [13]: the diffusion between the liquid and solid, and the nucleation followed by the formation and growth of the intermetallic compounds at the interface, which together determine the behaviour of the metal–metal system [2, 14].

Considering a liquid drop on a solid substrate, there are three surface tensions that need to be considered [15]: solid–liquid (σ_{sl}), liquid–gas (σ_{lg}) and solid–gas (σ_{sg}). Young's equation gives the relation between the equilibrium contact angle (θ) and the balance of surface tensions at the juncture [16]:

$\sigma_{sv} - \sigma_{sl} = \sigma_{lv} \cos\theta$

The derivation of Young's equation is made under the assumptions of the spreading of a non-reactive liquid on an ideal solid surface (physically and chemically inert, smooth, homogeneous and rigid) [7]. However, these conditions are rarely achieved in practical situations. Nevertheless Young's equation is a fundamental starting point for understanding of the complex field of wetting [7].

The extent of wetting is measured by the degree and rate of wetting. The degree of wetting is generally indicated by the contact angle that is formed at the interface between the surface and the liquid [1, 16–18]. It is optimized by minimizing the value of the contact angle [17]: $0° < \theta < 30°$—very good wetting, $30° < \theta < 40°$—good wetting, $40° < \theta < 55°$—acceptable wetting, $55° < \theta < 70°$—poor wetting, and >70°—very poor wetting, which corresponds to lower surface-interfacial energy [1].

There are several experimental techniques in the literature to evaluate the spreading and measure the equilibrium contact angle [19]. The conventional sessile drop method is probably the most used technique in the study of solid–liquid interfaces [1, 10, 20, 21], due to its simplicity and ease of use. This method is based on the analysis of the profile of the drop sitting on a substrate [22], as shown in Fig. 1.

Nowadays, in microelectronic industries, the SnAgCu based solder appears to be the most widely used lead-free alloy in replacement for traditional SnPb solder [24], because of its competitive price and apparently good mechanical properties [25]. Since the nature of the interactions depends on the composition of the solder and on the type of metallic substrate, the choice of the surface finish used in a PCB plays a crucial role [2]. There are several types of surface finish used in the PCBs, the most common are: OSP, NiAu and Immersion Tin. The pads from the PCB, to which the electronic components are soldered, are coated with this type of metallic material because of its affinity for soldering and essentially to improve the properties of reaction and wettability between solder/substrate. OSP surface finish is used mainly to prevent the oxidation of Cu and does not interfere metallurgical with the solder joints. PCB pad finish in NiAu has a Ni layer to prevent the diffusion of Cu into the solder, and the excessive growth of intermetallic compounds. The Ni layer has a thin coating of Au which improves the properties of wettability and protects the pads from corrosion. Sn is also a kind of used metal coating that improves the reaction of the substrate with the solder and prevent Cu from oxidation [23].

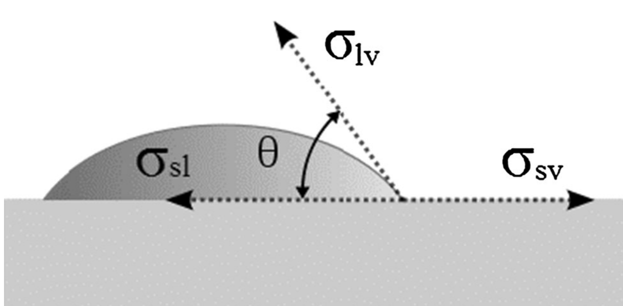

Fig. 1 Diagram of wetting angle (θ) and surface tension [23]

The measurement of the contact angle involves the control of a number of parameters that can affect the wetting behaviour of the solder. Temperature, atmosphere and substrate composition play the key roles to determine wettability, characteristics of spreading, and interfacial morphology [20, 26]. M. Arenas et al. [1] studied the contact angles of four Sn-based alloys measured with and without the use of fluxes, on Cu substrates. Measurements were performed using the sessile-drop method. Contact angles between $\theta = 10°$ and $\theta = 30°$ where obtained using RMA and RA fluxes. Higher values ($\theta = 30$–$40°$) were obtained for wetting under vacuum with no fluxes. S. Amore et al. [2] studied the behaviour of the Sn-Cu systems on two different metal substrate (Cu, Ni), under high vacuum conditions. The results shows a better wettability on the Cu-substrate than on Ni-substrate, $\theta = 40°$ and $\theta = 90°$ respectively, for temperatures up to 300 °C.

Zang et al. [17] investigated the wetting behaviour of Sn-3.0Ag-0.5Cu solder on Cu, Ni substrates with temperatures, under an Ar atmosphere. At 250 °C the contact angle is about $\theta = 29°$ on Cu substrate and $\theta = 37°$ on Ni substrate.

Several methods to approximate surface tension (σ) from droplet shape and size, based on the Laplace equation have been developed in past [27, 28]. Bashforth and Adams [28] generated sessile drop profiles for different values of surface tension and radius of curvature at the apex of the drop. Bashforth-Adams tables, become one of popular axisymmetric drop shape analysis (ADSA) to determine interfacial tension and contact angle from the actual experiment profile. However, use of these tables is limited to drops of a certain size and shape range with contact angle greater than 90°.

Malcolm and Elliott [27] used semiempirical equation to estimate surface tension from total height (H) and maximum diameter (D) of a sessile drop, which led to the development of axisymmetric drop shape analysis—height and diameter (ADSA-HD). The advantage of this approach has been applied to sessile drops of any contact angle [27]. Moreover, Rio and Neumann [29] developed a new computer software called Axisymmetric Liquid–Fluid Interface (ALFI) to overcome the deficiencies of the numerical schemes, which using more efficient algorithms. It performs theoretical Young–Laplace curves by integrating the

Laplace equation for recognized values of surface tension and curvature at the apex, which automates and extends the procedure of Bashforth and Adams.

The aim of this study is to understand the wetting behaviour of the Sn-3.0Ag-0.5Cu (SAC305) solder by measuring the contact angle on different substrates by the sessile drop method. The effect of the atmosphere and temperature of measurement will be also examined.

2 Experimental procedures

The lead-free alloy Sn-3.0 %Ag-0.5 %Cu (wt%) was used for this investigation. This alloy was provided by the company Stannol in form of solid wire with 3 mm of diameter. The samples of solder used for wetting experiments were cut from the solid wire with mass around 0.3–0.35 g. Before the tests the samples were cleaned with alcohol in an ultrasonic bath during 10 min.

The wetting behaviour of this solder was analysed on three different substrates obtained from three printed circuit boards (PCB) with surface pad finish of Sn, OSP and NiAu. Each PCB consists of a base of FR4, a layer of copper and a certain surface finish. In Table 1 it is presented the composition the three different boards. The PCB with surface finish in Sn have a different number of layers. The PCB is composed of two layers of Cu (bottom and top surface) with a Sn finish layer over it.

The PCBs were cut into pieces of 20 mm × 20 mm, to use as substrates in the experiments. After cleaning the samples it was applied a flux on the substrate surface, right before starting the experiments. The type of flux used was Cobar 94QMB (REL0) applied in the liquid form. This flux promotes heat transfer to the solder joint, improves adhesion of the solder on the surface (increase wettability) and prevents oxidation of the metal surfaces during soldering.

The wetting experiments were performed by the sessile drop method. It was used two different atmospheres in this investigation: high vacuum and inert atmosphere (using Nitrogen).

Table 1 Composition the three different boards used in the wettability testes

Layers	PCB		
	OSP	NiAu	Sn
Layer of FR4	1600 μm	1600 μm	1600 μm
Layer of Cu	35 μm	35 μm	18 μm (×2)
Surface finish	1.9 μm de Sn	2 μm de Ni	2 μm Sn (×2)
	0.1 μm de OSP	0.5 μm de Au	

In this study, the aim was to determine the contact angle of the solder as function of temperature, so it was placed a portion of solid solder on the substrate and, then, the test was started. With increasing temperature over time the drop profile was dynamically registered by a digital camera, by taking photographs at various temperatures. From the images, it is possible to calculate the contact angle at the desired temperature (Fig. 2). The estimated error associated with the angle measurements was approximately ±3°. Each test made in high vacuum ($\sim 10^{-5}$ mbar) had a duration of 1 day to ensure a good vacuum and a slow and steady pre-heating in high vacuum. The processing conditions were as follows: vacuum at room temperature for 7 h; pre-heating in a high vacuum from room temperature to 170 °C for 9 h (to facilitate stabilization of the oven) followed by the wetting measurements temperature cycle.

A platform was used to ensure the horizontality of the sample during the test (Fig. 3). Furthermore, because of its geometry, it allows placing a thermocouple below the sample, ensuring a more accurate reading of the temperature.

The wetting experiments performed in an inert atmosphere had the same initial processing conditions (for furnace chamber cleaning). However, before beginning the temperature cycle, the secondary vacuum pump was turned off and nitrogen was introduced at an initial pressure of 100 mbar.

Determination of the surface tension is based on the ADSA-HD analysis given by Rio and Neumann [29]. First, the profile line of droplet was extracted from experiment. Then, required coordinate information (x, z, Θ) was input to the ADSA-HD. The apex curvature (b), the capillary parameter (c) or surface tension (σ) is computed by numerical integration of the Laplace equation with starting from suitable initial assumptions:

$$\frac{dx}{d\theta} = \cos\theta\left(2b + cz - \frac{\sin\theta}{x}\right)^{-1} \tag{1}$$

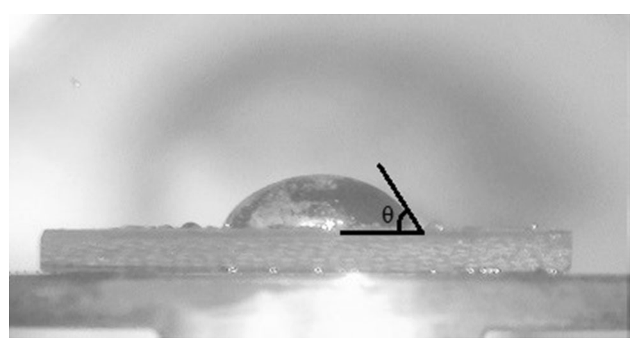

Fig. 2 Measurement of the solder droplet contact angle

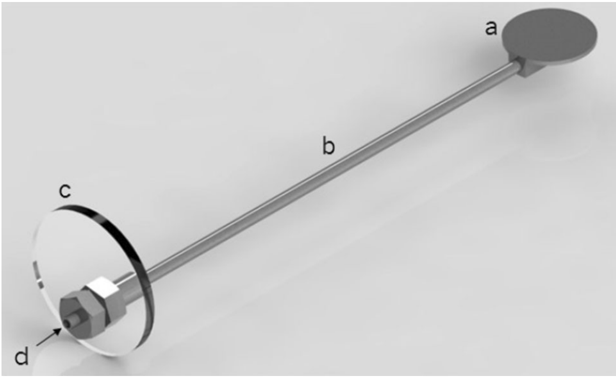

Fig. 3 Support platform: *a* base; *b* hollow stainless steel tube; *c* glass window; *d* input to the thermocouple

$$\frac{dz}{d\theta} = \sin\theta\left(2b + cz - \frac{\sin\theta}{x}\right)^{-1} \quad (2)$$

$$\frac{db}{d\theta} = 0 \quad (3)$$

$$\frac{dc}{d\theta} = 0 \quad (4)$$

$$x(0) = z(0) = 0, \quad x(\theta_1) = R, \quad z(\theta_2) = H \quad (5)$$

where R = D/2, c = (Δρ)g/σ.

The algorithm in this case is initialized using an elliptical fit that satisfies the boundary conditions as shown in Eq. (5), and the capillary parameter is initialized with a rough estimate which around 10.

Based on Butler's model, the surface is considered as an additional thermodynamic phase, in equilibrium with the bulk. The surface tension of binary and ternary liquid alloys, assuming the regular solution model, can be calculated as [30]

$$\sigma = \sigma_i + \frac{RT}{S_i} In \frac{X_i^s}{X_i^b} + \frac{1}{S_i}.\left[G_i^{xs,s}\left(T, X^s_{j(j=2,3)}\right) - G_i^{xs,b}\left(T, X^b_{j(j=2,3)}\right)\right], \quad i = 1,2,3. \quad (6)$$

where R, T, σ_i, S_i are the gas constant, absolute temperature, the surface tension of pure components and the surface area, respectively.$G_i^{xs,s}\left(T, X^s_{j(j=2,3)}\right)$ and $G_i^{xs,b}$ $(T, X^b_{j(j=2,3)})$ are the partial excess Gibbs energies of a component i in the surface phase and in the bulk phase, respectively. Both free energies are given as functions of T and composition of the surface and bulk phase.

The surface area of component i is calculated from Avogadro's number, the atomic mass and the density data [31] as follows:

$$S_i = 1.091 N_o \left(\frac{M_i}{\rho_i}\right)^{2/3} \quad (7)$$

The excess energy term of a component i can be derived from the standard thermodynamic relation, in the form:

$$G_i^{xs} = G^{xs} + \sum_{j=1}^{n} (\delta_{ij} - X_j) \frac{\partial G^{xs}}{\partial X_j} \quad (8)$$

where δ_{ij} is Kronecker's symbol.

Assuming that the free energy of the alloy is always proportional to the number of interactive contacts between neighbouring atoms, can be related to the respective co-ordination numbers in the surface layer and the bulk phase as:

$$G_i^{xs,s}\left(T, X^s_{j(j=2,3)}\right) = \beta G_i^{xs,b}\left(T, X^b_{j(j=2,3)}\right) \quad (9)$$

where β is the ratio between the two coordination numbers, i.e. a parameter describing the reduced coordination in the liquid phase. In present paper, β was assumed as 0.75.

The excess Gibbs energies of the ternary subsystems of the Ag–Cu–Sn system is calculated combining the corresponding values of the Ag–Cu, Ag–Sn and the Cu–Sn [5] binary subsystems with an additional ternary contribution in the form:

$$^{xs}G_L^{A-B-C} = \sum_{i,j} x_i.x_j. \sum_{v=0}^{n} (A_v + B_v.T).\left(x_i - x_j\right)^v + x_1 x_2 x_3 L_{123} \quad (10)$$

In present paper, the program named SURDAT [31] was used for surface tension calculations based on Butler's equation. The SURDAT program contains a set of data of surface tension and density in an extensive range of temperatures and concentrations. Surface tension calculated from Butler's equation was used to validate the surface tension determined from the sessile drop method.

3 Results and discussion

Figure 4 shows the results of contact angle measurements of the tests between lead-free solder SAC 305 and different substrates, using a high vacuum atmosphere. The results shown that the contact angle is sensitive the temperature.

In NiAu and OSP substrates, the contact angle of the solder starts from the initial value of about $\theta = 110°$ and $\theta = 117°$, respectively. It decreases slightly with the temperature increase, up to 225 °C. Above this value, the contact angle quickly decreases from the equilibrium value, for values around $\theta = 20°$ and $\theta = 32°$ in NiAu and OSP substrates.

P. Protsenko et al. [32] studied the role of intermetallics in wetting in metallic systems. These authors explained that in low temperature, the oxide films on the liquid or the solid can be a fundamental factor to brake or stop the

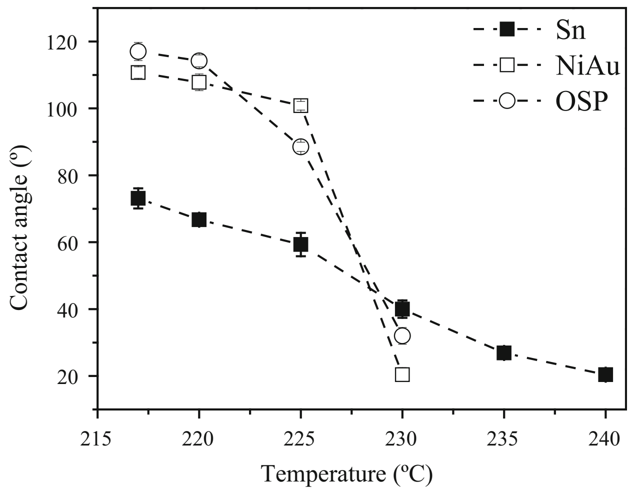

Fig. 4 Contact angle as function of temperature, of the solder SAC305 in different substrates, in high vacuum

Table 2 Contact angle (°) as function of temperature, of the solder SAC305 in different substrates, in high vacuum

Substrate	Temperature of the sample (°C)					
	217	220	225	230	235	240
Sn	73	67	59	40	27	20
NiAu	111	108	101	20	–	–
OSP	117	114	89	32	–	–

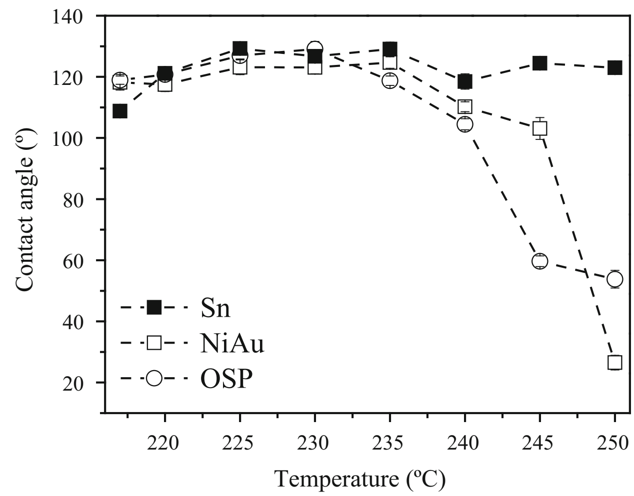

Fig. 6 Contact angle as function of temperature, of the solder SAC305 in different substrates, in inert atmosphere

wetting process. Due to the nature of Sn, this type of lead-free alloys can form a protective oxide layer when heated to a liquid form [33]. However, with increasing temperature this protective layer breaks and allows full spreading (Fig. 5). Furthermore, with increasing temperature, the wetting is strongly improved by the intermetallic formation [32, 34].

A different behaviour was observed on Sn substrate. In this last case, the initial contact angle at 217 °C is $\theta = 73°$. At the melting point of the solder the contact angle is lower than in previous cases mainly due to faster solder-Sn reaction, promoting wettability and spreading between liquid and solid. With increasing of temperature, the contact angle decreased slowly. At 240 °C the contact angle is $\theta = 20°$, above this temperature the solder spreads completely (Table 2). A good wetting result was achieved when the oxides were completely removed [35].

Figure 6 shows the results of contact angle measurements of the tests between lead-free solder SAC 305 and different substrates, using an inert atmosphere. The initial value the angle contact is very similar in the three substrates, around $\theta = 130°$ and 110°. In the test performed on Sn substrate, this value does not change with increasing temperature. In NiAu and OSP substrates, the contact angle remains almost constant until the temperature of 235 °C. Above this temperature, the contact angle starts to decrease. At 250 °C, the contact angle between molten solder and NiAu substrate is about 27°, representing a degree of very good wetting. While in OSP substrate, at this temperature the contact angle is around $\theta = 54°$, indicating a degree of acceptable wetting (Table 3).

The results obtained in this case are quite different in comparison with those obtained in vacuum atmosphere. This could be explained by the higher possibility of oxidation that can occur during the test of wettability, since the atmosphere is not perfectly inert [10]. For this reason, in NiAu and OSP substrates, due to the eventual stronger film of oxidation, the solder only starts to spread at a higher temperature, compared with vacuum results, resulting in

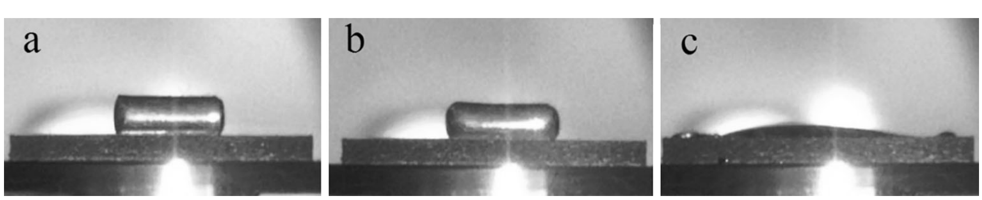

Fig. 5 Evolution of the wettability of the solder on the substrate NiAu at: **a** 200 °C; **b** 217 °C; **c** 230 °C in vacuum atmosphere

Table 3 Contact angle (°) as function of temperature, of the solder SAC305 in different substrates, in inert atmosphere

Substrate	Temperature of the sample (°C)							
	217	220	225	230	235	240	245	250
Sn	109	121	129	127	129	119	125	123
NiAu	118	118	123	123	125	110	103	27
OSP	119	121	127	129	119	105	60	54

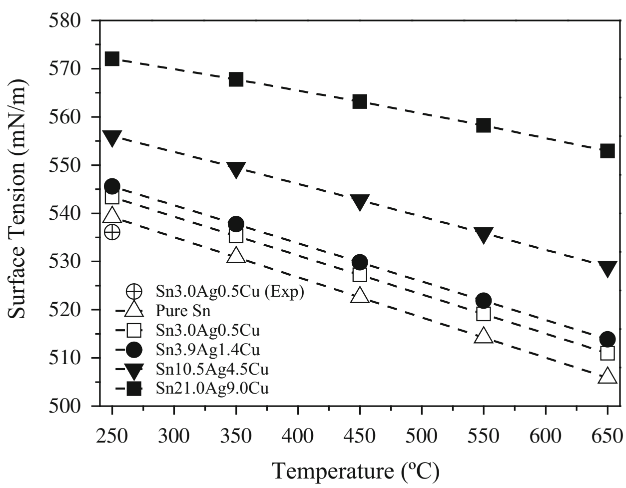

Fig. 7 Surface tension for various tins concentrations based on Butler model

higher values of contact angles. In Sn substrate, after the solder starts to melt, the contact angle does not change much with temperature. The surface conditions of the PCB is also an important aspect that affects the wettability behaviour of the solder. Au and OSP layers protect the pads from surface oxidation, however, the PCB with surface finish in Sn tends to oxidize when exposed to air and depending on the time of exposure to air, this oxide layer may vary [29]. This layer of oxidation acts also as a barrier to the wetting process. The oxide layer formed around the solder associated to the oxidation on the surface of Sn substrate, resulted in a strong barrier that prevented the solder from spreading and react properly with the substrate, corresponding to very high values of contact angle.

A comparison between the contact angle values obtained in the literature for Sn-based solder are quite disparate and hard to compare, because of the different conditions used during testing.

Figure 7 shows the surface tension comparison between experimental and modelling.

The surface tension measurements were carried out on the Sn3.9Ag1.4Cu, Sn3.0Ag0.5Cu, Sn10.5Ag4.5Cu, Sn21.0Ag9.0Cu liquid alloys and pure tin between their melting point, T_m, and 650 °C, as shown in Fig. 7.

Table 4 Experimental values of the surface tension compare with predicted by the butler model

Experiment		Butler model	Error (%)
c (cm^{-2}) [29]	σ (mN/m)	σ (mN/m)	
13.45	537.54	543.35	1.1

The experimental values of the surface tension of SAC305 on NiAu substrate at 250 °C are shown in Table 4 and compare with predicted by the Butler Model. Taking into account systematic errors of measurement procedure and the accuracy and repeatability of profile acquisitions, the overall or total error involved in the surface tension value is estimated to be less than 5 %.

4 Conclusions

The contact angle of lead-free alloy SAC305 in function of temperature was measured using three different substrates, by the sessile drop method. The measurements were directly made on PCB pieces with different surface finishes. The process of measuring wettability on this type of solder is relatively complicated due to the strong oxidation film formed by tin, when heated to liquid state. In high vacuum, on substrates of NiAu and OSP the solder started spreading suddenly at 225 °C to a low value of contact angle, thus showing a very good wettability. On Sn substrates the contact angle is lower at low temperatures, and gradually decreases with increasing temperature. In nitrogen atmosphere, the contact angle was lower for NiAu and OSP substrates and in Sn, due to oxidation, the solder didn't spread as predicted, so it was not possible to obtain good values for the contact angle. Since the PCB pad with Sn and OSP are similar, in terms of surface metal finish, can be concluded that the existence of the OSP film prevents the oxidation of the Sn surface and improves the wettability of the solder.

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