PROJECTED AREA METHOD FOR THE EVALUATION OF CONTACT ANGLE ON SESSILE DROP TEST

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Abstract: *The sessile drop test is a well-known practice among materials scientists for evaluating surface/interface energies in many applications, from brazing processes to thin films technology. However, the evaluation of sessile drop results is mostly based in in-situ measurements or in cross-section analysis of samples, requiring complex experimental setup or extended sample preration work. This works provides a simplified method based in fundamental geometry that can be useful as a first approach for comparing contact angles and surface energies. An experimental validation of the proposed method was performed by sessile drop test for the copper – stainless steel system, which is widely used for brazing purposes, resulting in an experimental agreement higher than 83% in comparison with cross-section measurements.*

Keywords: *Sessile drop, Wettability, Surface engineering, Brazing.*

1. INTRODUCTION

As a widespread technique applied to surface characterization, sessile drop test (Bashforth and Adams, 1883) has supported the study and improvement of innumerous systems, from protective coating films (Panwar *et al*, 2003) to joining technology (Chen *et al*, 2006). Sessile drop test is commonly applied to brazing studies to measure the contact angle of a specific system in order to evaluate filler metal wettability (Funk and Udin, 1952) and consequently its adequacy for producing successful brazed joints (American Welding Society, 1991)

The evaluation of wettability of molten filler metal on base materials presents several challenges for brazing engineers due to the difficulties associated with contact angle measurement. In-situ measurement at brazing temperature requires complex instrumentation and has inherent high costs, while optical microscopy measurement of cross-sectioned samples is a time-consuming task due to the required sample preparation. In addition, the characteristic low contact angles required for brazing processes seriously compromises the acuity of those traditional methods.

Aiming the development of a simplified approach for contact angle evaluation, this work proposes a simplified geometric model and compares its results with the optical microscopy analysis of cross-sectioned samples for the evaluation of a copper-stainless steel brazing system.

2. EXPERIMENTAL PROCEDURE

2.1 Sessile Drop Experimental Setup

A typical brazing thermal cycle under pure hydrogen atmosphere was applied to 10 samples, as shown by the time-temperature profile illustrated in Fig. (1). Each sample consisted of AISI 304 stainless steel base metal with 50x50x3 mm, degreased and pickled in two stages with pure hydrochloric acid and with chromic acid, and a C11000 copper filler metal piece with approximately 50 mg freely placed on top of the base metal.

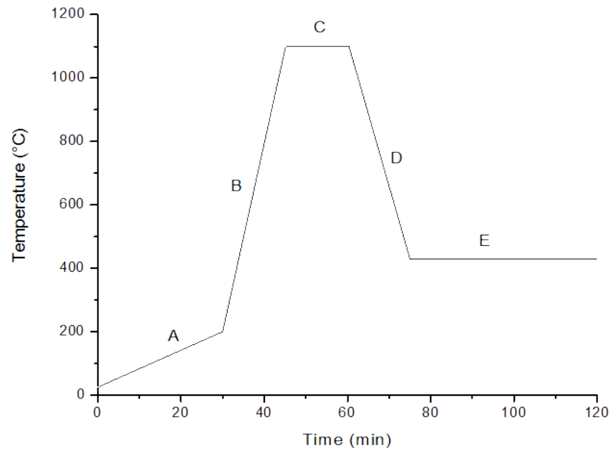

Figure 1: Temperature profile during sessile drop test

2.2 Simplified Method Application

The brazed samples were photographed using a digital camera (Nikon Coolpix 5200 5.1M) and the image scaling was performed digitally using an aligned caliper as reference. The drop projected area on top of the base metal was evaluated with ImageJ® software by color contrast for each sample.

The proposed method is based on fundamental geometry and assumes that any sessile drop can be considered as a spherical cap, as shown in Fig. (2a), what is good approximation when the drop is small and the surface tension dominates over the gravity (Erbil, 2006). From this assumption, it is possible to evaluate the contact angle, when α<<90°, as function of the drop total volume and projected area on the base metal. The total volume of a spherical cap can be achieved by integral calculus as demonstrated elsewhere (Lennart and Westergren, 1990) and results in Eq. (1):

$$V = \frac{\pi h^2 (3R - h)}{3} \tag{1}$$

where **R** represents the sphere radius and **h** the drop height, as noted in Fig. (2b). The value of **V,** as drop total volume, can be obtained by the relationship of filler metal mass and density. **R** and **h** variables can be correlated by a pythagoric relationship on cross section, also illustrated in Fig. (2b) and presented in Eq. (2):

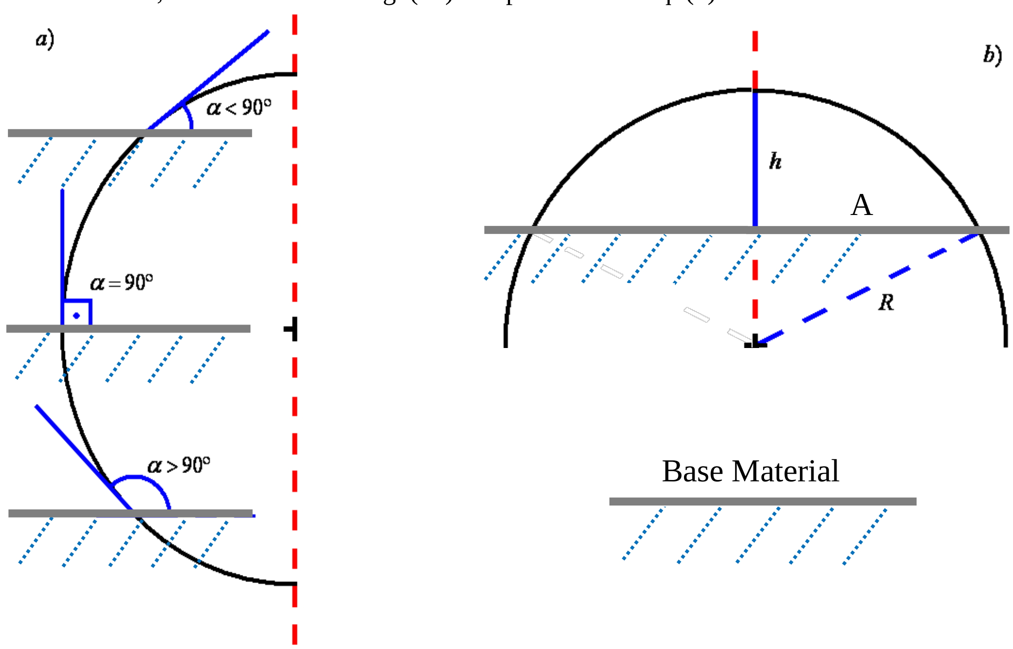

Figure 2: a) Illustration of contact angles for sessile drops as a spherical cap and b) geometrical parameters applied on proposed method.

$$R^2 = A^2 + (R-h)^2 \quad (2)$$

where **A** represents the projected spherical cap radius.

Equation (1) and (2) constitutes a two variable system that is easily solved by numeric evaluation. With the calculated value of R it is possible to evaluate the curvature of the spherical surface at a given point, as shown in Fig. (3).

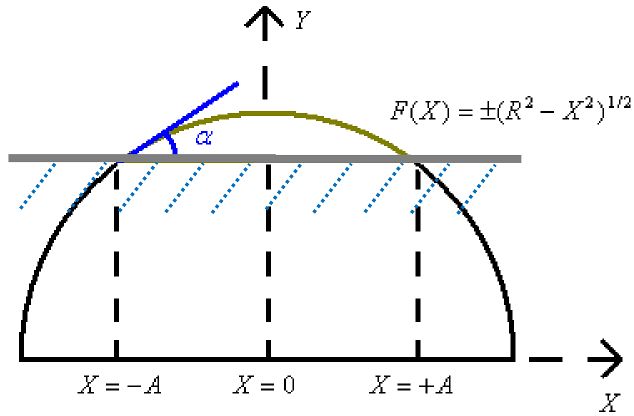

Figure 3: Illustration of curvature function and derivative position for proposed model.

For the derivative took at x = ±A, the contact angle α is evaluated considering a spherical cap model, which is given by Eq. (3):

$$\alpha = arctg\left[\frac{A}{\sqrt{(R^2 - A^2)}}\right] \quad (3)$$

2.3 c) Model Evaluation

The sessile drop samples were also evaluated using standard cross-sectioning. The samples were sectioned using a precision saw (Buehler IsoMet 4000) followed by metallographic preparation and optical microscopy (Leica DMLM 100x) observation. The obtained images were digitally analyzed using ImageJ® software.

3. RESULTS AND DISCUSSION

Figure (4a) shows a photography taken after the sessile drop test and Fig. (4b) presents an example of micrographic image detail obtained after metallographic preparation for contact angle measurement.

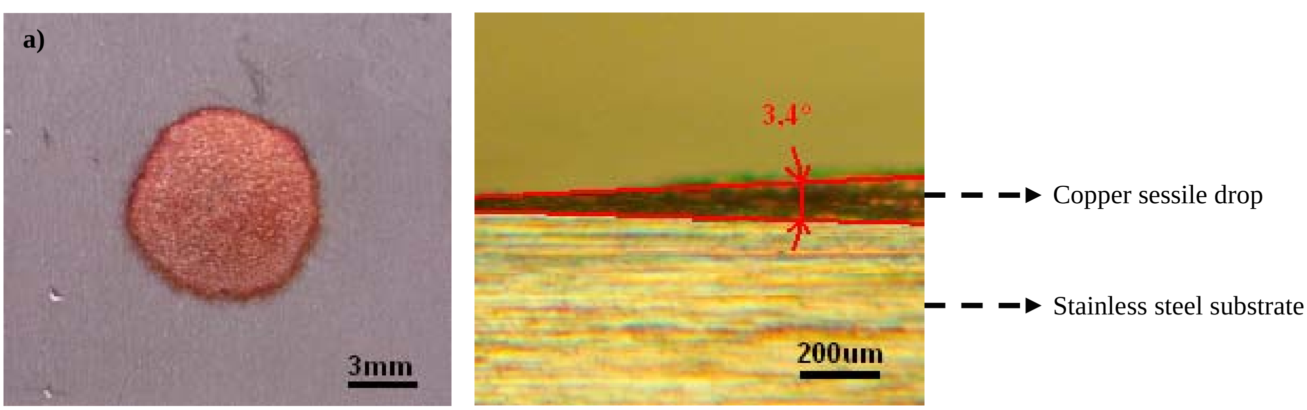

Figure 4: a) Top view of an obtained sessile drop and b) cross-section image with measured contact angle.

Contact angle measurement results by the cross-section and by the projected area method for the copper-stainless steel system under pure hydrogen atmosphere are listed in Tab. (1):

Table 1: Contact angle in degrees measured by cross-section and calculated by the proposed method

Sample number	Cross Section Test (degrees)	Projected Area Method (degrees)
1	2,9	2,7
2	4,7	4,2
3	1,9	2,4
4	3,1	2,8
5	1,9	2,5
6	2,8	2,4
7	3,4	2,4
8	2,3	2,2
9	3,5	4,1
10	2,6	2,3

Results listed in Tab. (1) indicate a direct correlation of the cross section and project area method results, indicating that the latter can be applied as a qualitative tool for comparing contact angles, achieving a concordance value of approximately 83,5% when compared with conventional cross section method.

Table (2) presents a statistical comparison of the results of both standard and proposed procedures, which are listed in Tab. (1).

Table 2: Statistical comparison of the standard sessile drop test and the proposed method

Result	Cross Section Test	Projected Area Method
Mean Value (degrees)	2,9	2,8
Max Value (degrees)	4,7	4,2
Min Value (degrees)	1,9	2,2
Variance (degrees^2)	0,78	0,61
Standard Deviation (degrees)	0,88	0,78
Value for 95% Confidence Interval (degrees)	2,9 ± 1,8	2,8 ± 1,6

Statistical comparison of the results obtained using the conventional cross section and the projected area method indicates the quantitative equivalence of both methods to measure the contact angle within a 95% confidence interval.

As the projected area method is based on geometrical evaluation of a sessile drop which can be virtually extended to any case that the drop shape can be assumed as a spherical cap. Therefore, there are restrictions regarding the drop mass and angle range evaluated. A correction on total volume of the drop V must be performed in systems that the filler material is partially consumed during the brazing process by considering only the metal volume that constitutes the sessile drop. The use of flux cored filler material contextualizes this example.

The evaluation of systems with higher contact angles, specially higher than 90° requires special attention for the A parameter measurement due to the top-view projection indicates R value instead, as can be noticed in Fig. (2). In addition, for such larger angles, the drop tends to assumes ellipsoidal shapes (Erbil, 1997). However, as previously stated, these considerations are beyond the application of proposed method, which is applicable to commonly used brazing systems, where the contact angles tend to be very low.

4. CONCLUSION

The comparison of the projected area method evaluation and the cross-sectional results for the contact angle for the copper-stainless steel system brazed under a pure hydrogen atmosphere showed that fundamental geometry can be used as a first approach to evaluate a sessile drop experiment, demanding only the measurement of filler metal mass and sessile drop projection.

Experimental concordance higher than 83% was achieved when comparing standard and proposed methodologies results for each sample and the mean value of contact angle for evaluated system was identical within the 95% confidence interval. Obtained results indicate that the proposed method can be successfully applied as a complementary analysis for the sessile drop test in both qualitative and quantitative manner.

5. REFERENCES

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