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ACA Curing Process Optimization Based on Curing Degree Considering Shear Strength of Joints

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ABSTRACT This paper presents an optimized curing process for the shear strength achievement and retention for anisotropic conductive adhesive (ACA) flip-chip interconnections. The curing kinetics of ACA is first presented and then a curing degree (α) relationship between curing temperature T and curing time t is established. The influence of different curing degree states on the interfacial shear strength is then reported, including the effect of degree of curing on shear strength both before and after hygro-thermal aging.

INDEX TERMS Curing degree, ACA, parameters optimization, shear strength.

I. INTRODUCTION

Anisotropic conductive adhesive (ACA) has numerous advantages—it is environmentally friendly and lead-free, has a lower packaging temperature, and results in reduced thickness. It is used in many electronic packaging interconnect products, such as flip-chip bonding of radio frequency identification (RFID) inlays, chip-on-glass (COG) packaging for liquid crystal displays (LCDs), and chip-in-flex packages for wearable electronics [1]–[4]. However, its interfacial shear strength reliability is still one of the most critical issues of ACA joints, which greatly limits wider application [5]–[8].

Generally, the ACA bonding process has three different parameters: bonding time, bonding temperature, and bonding pressure. These bonding parameters not only influence the contact resistance of ACA joints but also greatly determine their adhesive strength. Among them, bonding time and bonding temperature have a significant influence on the bonding shear strength, whereas bonding pressure influences the electrical resistance.

Many studies have investigated the conductive properties and electrical reliability of ACA joints, and have reported that the degree of deformation of the conductive particles is determined by the amount of bonding pressure applied during the bonding process [9]–[12].

Some studies on ACA bonding have found that the interfacial shear strength reliability of ACA joints depends to a large extent on the bonding parameters, including curing time and temperature [13]–[16]. The shear strength of the ACA joint depends on the curing degree of the ACA matrix, which is related to two of the bonding parameters—bonding time and bonding temperature. The bonding pressure seems to have no direct relationship to the shear strength of the ACA joint.

Many studies have focused on the contact resistance reliability of ACA joints, but few studies have looked at the shear strength of ACA joints. This study focuses on the shear strength of ACA joints with the other two bonding parameters, bonding time and bonding temperature. The adhesive shear strength is considered a key criterion to determine the optimum bonding parameters for reliability of the interconnection from a systematic viewpoint of ACA curing reaction mechanism.

Generally, the curing reaction of ACA is characterized by the curing degree of the epoxy resin. For an ACA curing process, the measured heat flow dQ/dt is proportional to the curing reaction rate $d\alpha/dt$. This assumption is valid if no other enthalpic events occur except for chemical reactions such as evaporation, enthalpy relaxation, or significant changes

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in heat capacity conversion. According to the assumption mentioned above, the instantaneous change of the conversion rate is usually defined as [17]:

$$\frac{d\alpha}{dt} = \frac{dQ/dt}{\Delta Q} \tag{1}$$

where ΔQ is the total exothermic heat of the reacting groups, expressed as heat per mol (KJ·mol^{-1}) or per mass of materials (J · g^{-1}). Usually, the curing kinetics equations of thermosetting materials can be expressed as

$$\frac{d\alpha}{dt} = k(T)f(\alpha) \tag{2}$$

where $f(\alpha)$ is the curing kinetic model and k is the temperature-dependent rate constant given by the Arrhenius equation:

$$k = A \cdot \exp(-\frac{E}{R \cdot T}) \tag{3}$$

where E is the activation energy, R is the gas constant (R = 8.314 J/(mol · K), T is the absolute temperature, and A is the frequency factor. The aim of the kinetic analysis of DSC (differential scanning calorimeter) data is to find the kinetic model that gives the best description of the studied process and allows the behavior of the system to be predicted under selected thermal conditions.

It is useful to define the two following functions [18]–[20]:

$$y(\alpha) = \left(\frac{d\alpha}{dt}\right)\exp(x) \tag{4}$$

$$z(\alpha) = \pi(x)\left(\frac{d\alpha}{dt}\right)\frac{T}{\beta} \tag{5}$$

where $x = E/RT$, and $\pi(x)$ can be expressed as

$$\pi(x) = \frac{x^3 + 18x^2 + 88x + 96}{x^4 + 20x^3 + 120x^2 + 240x + 120} \tag{6}$$

Both the $y(\alpha)$ and $z(\alpha)$ functions can be obtained from experimental DSC data and can easily be used for the kinetic model determination.

Whichever kinetic model is adopted, the activation energy E and frequency factor A for the curing process must be calculated. There are two different methods to estimate them according to the DSC test method adopted. For the isothermal DSC test, they can be estimated from the linear logarithm plot of the Arrhenius equation based on the isothermal curing test data. For the dynamic DSC test, the estimation of the activation energy can be achieved through the well-known Kissinger equation [21]–[25]:

$$\frac{d[\ln(\beta/T_p^2)]}{d[1/T_p]} = -\frac{E}{R} \tag{7}$$

where β is the heating rate and T_p is the peak temperature of the reaction curve. The equation indicates that there is a linear relationship between $\ln(\beta/T_p{}^2)$ and $1/T_p$. If the least squares linear fitting is met, then E can be estimated from the slope and intercept of the linear plot fitted.

However, little work has been done to reveal the correlations of adhesion strength of ACA joints with the curing degree of the ACA matrix. In this paper, the effects of different curing degrees on the mechanical properties of a typical ACA are studied, and an optimum curing degree is suggested to achieve highly reliable ACA joints.

Two main parameters of ACA curing are the curing temperature (T) and the curing time (t). The curing degree (α) plays an important role in determining the shear strength of the ACA joints. The curing degree depends on the bonding temperature and time. As the bonding temperature and time increase, the curing degree also increases. A certain curing degree is required to provide enough mechanical strength for the ACA joints. Generally, the curing process of the ACA joints is achieved by controlling some key curing parameters, such as curing time and temperature, instead of controlling the curing degree directly. Therefore, the curing process needs to correlate the curing degree to those key parameters through curing kinetics modelling, by which the optimum curing parameters can be chosen for a given curing degree. Firstly, we should reveal the relationship between the curing degree and curing parameters, and then study the relationship between the curing degree and the shear strength. Finally, considering the shear strength both before and after aging, the optimum curing degree as well as the recommended range can be suggested. The methodology used in this study is graphically illustrated in Figure 1.

As shown in Figure 1, the ACA curing kinetics is experimentally characterized, and the equation of α (curing degree) with curing parameters T (curing temperature) and t (curing time) can be obtained as $\alpha = f(T, t)$. The shear strength data of ACA joints can be obtained from a shear strength test both before and after hygro-thermal aging tests, where the relationship of S (shear strength) and α (curing degree) can be graphically expressed. Then, considering the shear strength both before and after aging, the optimum curing degree as well as the recommended range can be suggested.

The rest of this paper is organized as follows. Section II presents the experimental procedures. Section III presents the results and discussion. Finally, some useful conclusions are drawn in Section IV.

II. EXPERIMENTS

The ACA joint sample was a radio frequency identification (RFID) inlay composed of three components: a silicon (Si) chip, the ACA layer, and a flexible substrate (Al/PET), as shown in Figure 2. The dimensions of the Si chip were 0.59 mm length and 0.59 mm width, with four 60-μm-long rectangular bumps. The ACA used in this study was DELO MONOPOX AC265 thermosetting conductive adhesive supplied by DELO, Inc. It was a composite consisting of micro-sized (about 3 μm) spherical metallic conductive particles that were uniformly mixed in the adhesive matrix, and the adhesive matrix contained thermo-set epoxy resin and other additives. The substrate used in this study was 50 μm thick with a 20 μm Al pad on it.

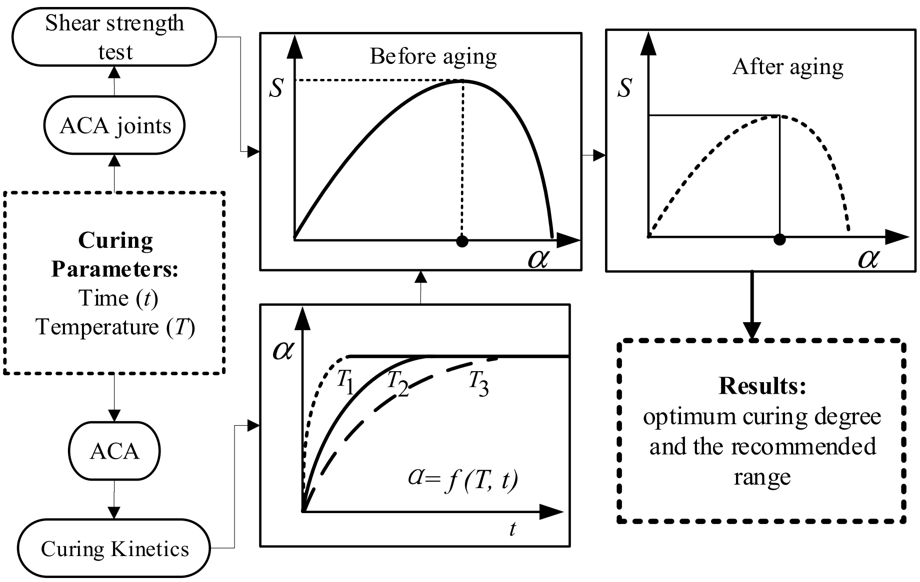


FIGURE 1. Methodology of this research.

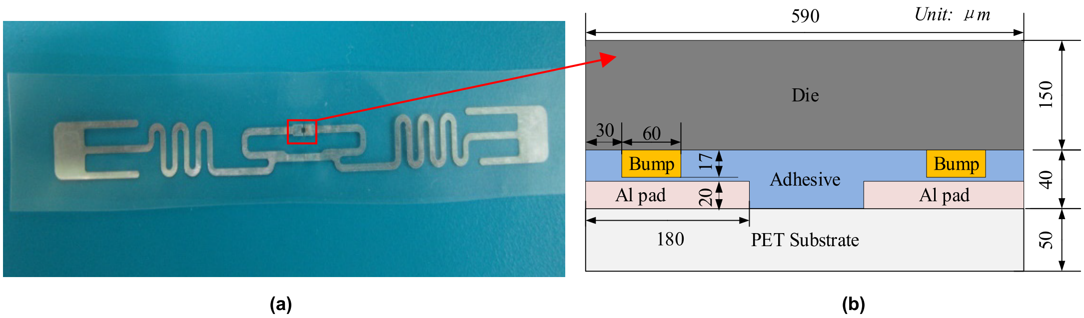


FIGURE 2. ACA joint sample: (a) photograph; and (b) schematic.

To study the curing kinetics of epoxy resin, several different methods have been proposed over the past decades. Among them, DSC analysis is most frequently used to analyze the curing kinetics of ACA [26], [27]. In this work, the curing kinetics of ACA was tested using a Diamond DSC tester, as shown in Figure 3(a). Firstly, the samples were prepared. The sample size taken for this study was 5–10 mg with an accuracy of 1% using an analytical balance. Then, the sample was put into the DSC equipment. The DSC tests were conducted with different ramp rates (β), and the rates of heat generation as a function of the temperature and time were recorded correspondingly.

The ACA joint samples were flip-chip bonded using a FINEPLACER_Lambda bonding instrument, as shown in Figure 3(b). For each group of bonding parameters, 20 samples were bonded. Among them, 10 samples were prepared for the shear strength test before aging and 10 samples were prepared for the aging test and shear strength test after aging.

The shear strength test principle was based on the instructions for a CONDOR 70-3 multifunctional bond tester and the solder ball shear test standard [28], [29], as shown in Figures 4(a) and (b). A rigid clamping device was used to fix one side of the substrate, and a vacuum plate was used to absorb the bottom of the sample. The shearing speed was 100 μm/s, and the height of the shear blade above the substrate was 50 μm. The blade was pushed horizontally from one side. The maximum shear force for each joint, which finally separated the chip from the substrate, was recorded, as shown in Figure 4(c).

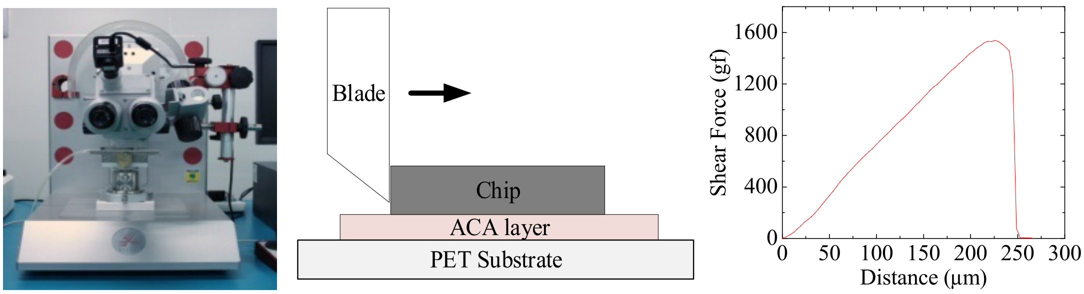
The ACA samples were aged in 85 °C/85% RH conditions for 168 h in a temperature-humidity chamber ZYGDW/SJ-100L, as shown in Figure 3(c). After the aging test, the samples were measured using the CONDOR 70-3 multifunctional bond tester.

III. RESULTS AND DISCUSSION

The detailed curing kinetics equations for the ACA are provided in the Introduction. The curing kinetics equation can be expressed as $\mathrm{d}\alpha/\mathrm{d}t = k(T) \cdot f(\alpha)$. Herein, $f(\alpha)$ is the

(a) (b) (c)

FIGURE 3. Test instruments: (a) Diamond DSC; (b) FINEPLACER_Lambda; and (c) Temperature-humidity chamber ZYGDW/SJ-100L.



(a) (b) (c)

FIGURE 4. Test instruments and procedures: (a) CONDOR 70-3 multifunctional bond tester; (b) measurement principle; and (c) plot of maximum shear force.

TABLE 1. Dynamic DSC data of the ACA tested.

β (K·min^{-1})	T_{onset} (°C)	T_p (°C)	ΔH (J/g)
5	108.3	115.03	316.2
10	118.53	125.90	316.5
15	125.72	132.44	317.1
20	131.86	136.62	318.5

curing kinetic model, and $k(T)$ is the temperature-dependent rate constant given by the Arrhenius equation as $k = A \cdot \exp(-E/(R \cdot T))$. The aim of the curing kinetic analysis is to determine the $k(T)$ and the kinetic model $f(\alpha)$ through DSC test.

DSC tests were performed from 50 °C to 200 °C with four different ramp rates (β), namely, 20 °C/min, 15 °C/min, 10 °C/min, and 5 °C/min. During testing, the rates of heat generation as a function of the temperature and time were recorded correspondingly. The plots of the dynamic DSC scans are shown in Figure 5, and the resultant data are listed in Table 1.

Figure 5 shows that the larger the heating rate is, the sharper the curve. That means the curing process of the ACA is quickened with the increment of the heating rate. As shown in Table 1, T_{onset} is the onset cure temperature, T_p is the peak cure temperature, and ΔH is the exothermic heat.

The coefficients estimation of the aforementioned ACA curing kinetics model is achieved through a group of dynamic DSC experimental results. The activation energy

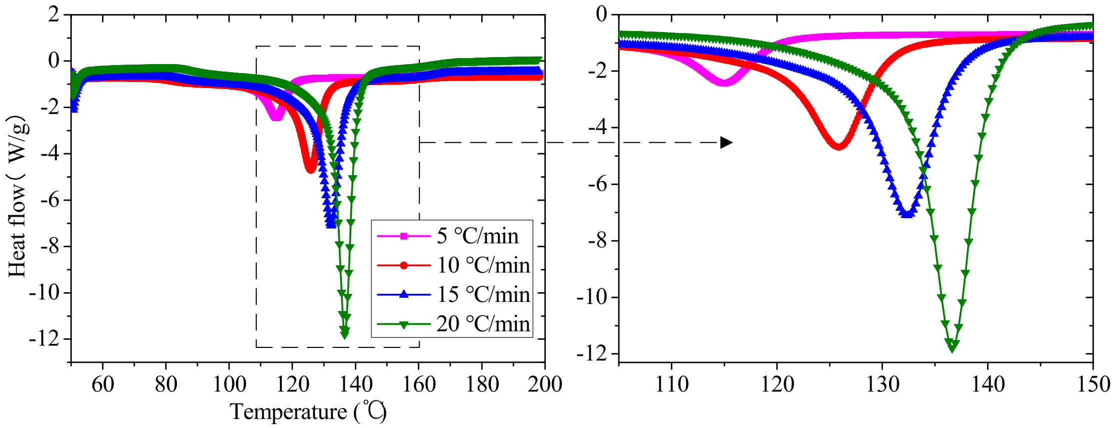


FIGURE 5. Dynamic DSC plot of ACA for different heating rates.

TABLE 2. Fitting results.

β (°C/min)		5	10	15	20	Results
$1/T_p$ (K^{-1})		2.58e-3	2.51e-3	2.47e-3	2.44e-3	
Kissinger	$\ln(\beta/T^2_P)$	-10.31	-9.67	-9.30	-9.04	E = 77.736 kJ/mol R^2 = 0.9991

E is estimated through the well-known Kissinger equation [18], [21]–[24], as shown in Equation (7). The DSC test data listed in Table 1 were used to model the relationship of $\ln(\beta/T^2{}_P)$ and $1/T_p$, and it was found that they follow a linear relationship, as shown in Figure 6. The fitting results can be obtained as shown in Table 2.

From Table 2, it can be seen that the fitting result of R-squared values (R^2) is approximate to 1, which means that the model fitting quality is good. The resultant activation energy E is 77.743 kJ·mol^{-1}.

The DSC data shown in Figure 5 are converted to $y(\alpha)$ and $z(\alpha)$ functions using Equations (4) and (5), as shown in the Introduction. These functions normalized within (0, 1) interval are plotted in Figure 7 for different heating rates. Figure 7 shows the curves of normalized $y(\alpha)$ and $z(\alpha)$ versus curing degree. The maximum α_M of the $y(\alpha)$ function is 0 and the maximum α_p^∞ of the $z(\alpha)$ function is 0.634.

From Figure 7, the curing kinetic model is determined based on the methods described in reference 19 and the most suitable $f(\alpha)$ model corresponds to the J-M-A ($n < 1$) model, as shown in Equation (8).

$$\frac{d\alpha}{dt} = k(T)n(1-\alpha)[-\ln(1-\alpha)]^{1-\frac{1}{n}} \tag{8}$$

Once the kinetic model has been determined, the kinetic parameters, such as A and n, can be easily calculated by linear fitting. The fitting curve is shown in Figure 8, and the results are shown in Table 3.

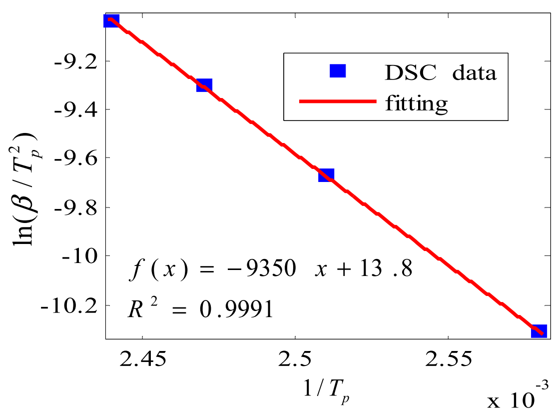


FIGURE 6. Curve of $\ln(\beta/T^2{}_p)$ versus 1/Tp for the tested ACA.

Substituting A and n into Equation (8), the expression for curing degree, curing temperature, and curing time can be obtained, as shown in Equation (9). For a certain curing degree, the relationship of curing time and curing temperature can be obtained by Equation (9).

$$\alpha = 1 - \exp(-[2.63 \times 10^8 \exp(-\frac{77736}{RT})t]^{0.788}) \tag{9}$$

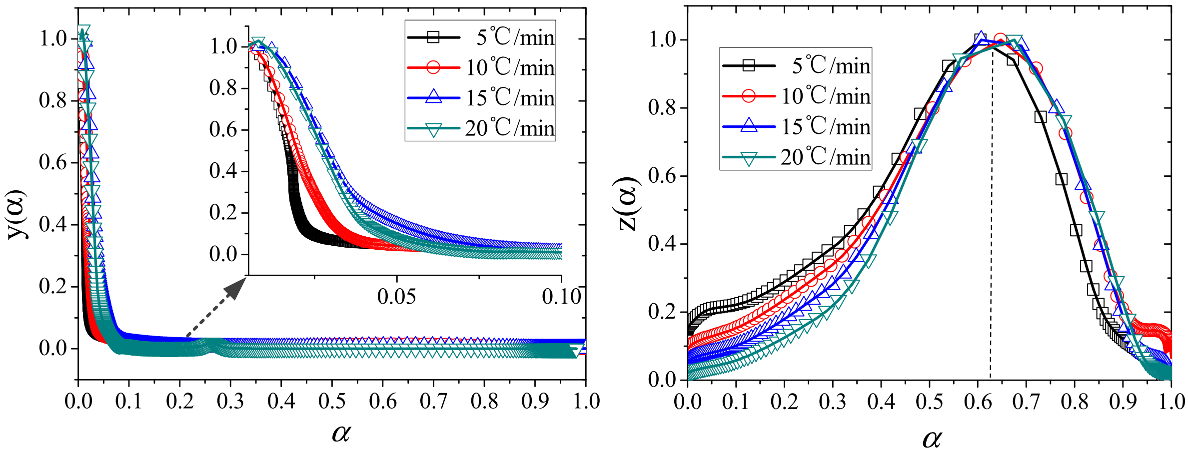


FIGURE 7. Normalized y(a) function and z(a) function.

TABLE 3. Fitting results.

β (°C /min)	n	A
5	0.794	2.56E8
10	0.799	2.71E8
15	0.785	2.64E8
20	0.774	2.59E8
Average	0.788	2.63E8

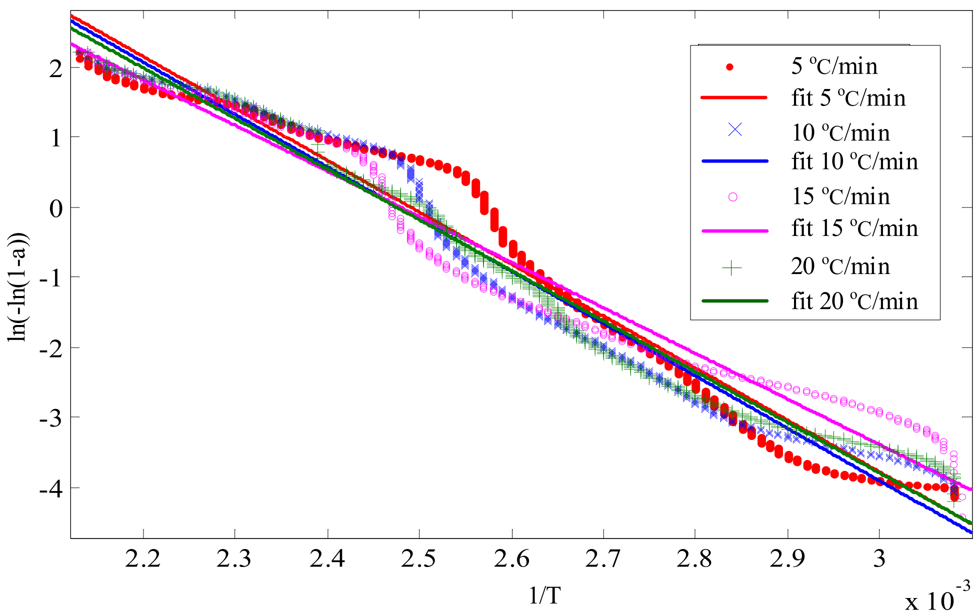


FIGURE 8. Fitting curve.

The results of shear strength under different curing degrees are shown in Table 4. The relationship curve of shear strength and curing degree before aging is plotted in Figure 9.

Figure 9 shows that shear strength increases at first with the curing degree, whereas it decreases quickly after 85%. Between 0% to 30% curing degrees, shear strength is low,

TABLE 4. Results of shear strength under different curing degrees (before aging).

Degree	Shear Strength (kgf)	Degree	Shear Strength (kgf)
100%	1.156	50%	0.521
95%	1.456	45%	0.459
90%	1.515	40%	0.362
85%	1.521	35%	0.216
80%	1.324	30%	0.102
75%	1.172	25%	0.044
70%	1.063	20%	0.032
65%	0.854	15%	0.024
60%	0.751	10%	0.013
55%	0.634	0	0

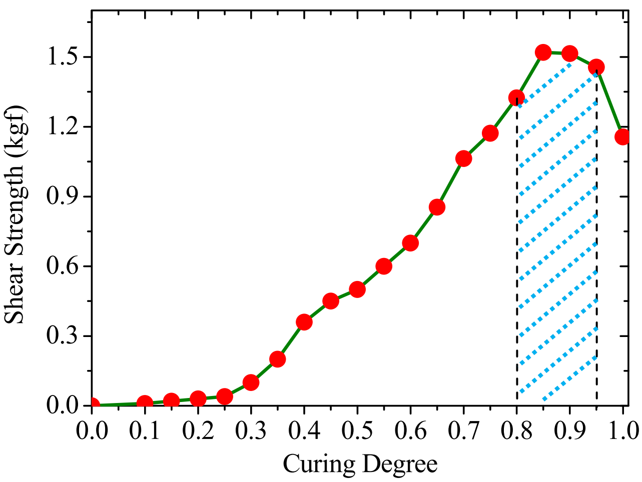


FIGURE 9. Plot of shear strength versus curing degree.

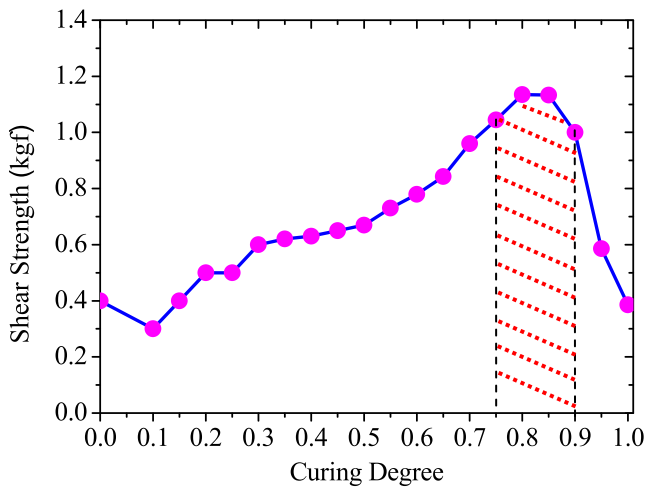


FIGURE 10. Plot of shear strength versus curing degree.

which implies that the cross-linking of the ACA interface is not strong. Between 30% to 85%, shear strength increases quickly with the curing degree. However, shear strength starts to decrease after 85%, which means that too high a curing degree will decrease the shear strength of the ACA joints. Generally, as for the polymer adhesive itself, the strength of the polymer adhesive increases with the degree of curing. When the curing degree is 100%, the strength could be the largest. However, in this paper, the experimental results show that the shear strength of the ACA joints is not largest at 100% curing degree. There are several reasons for this phenomenon. On the one hand, ACA is a composite consisting of micro-sized spherical metallic conductive particles that are uniformly mixed in the adhesive matrix, and the adhesive matrix contains thermo-set epoxy resin and other additives. Therefore, the ACA is different from the polymer. On the other hand, this study focuses on the shear strength of ACA joints, and the shear strength is the maximum shear force for ACA joints, which finally separated the chip from the substrate. Therefore, the shear strength of the ACA joints is different from the strength of the polymer itself. The results in Figure 9 show that the optimum curing degree to achieve highly reliable joints is 85%, and the recommended range is from 80% to 95%.

In order to further study the relationship of shear strength and curing degree under hygro-thermal conditions, 85 °/85% RH aging test was used for the strength degradation of ACA joints. The results are shown in Table 5, and the relationship curve of shear strength and curing degree after aging is plotted in Figure 10.

TABLE 5. Results of shear strength under different curing degrees (after aging 168 h).

Degree	Shear Strength (kgf)	Degree	Shear Strength (kgf)
100%	0.386	50%	0.671
95%	0.586	45%	0.656
90%	1.002	40%	0.638
85%	1.133	35%	0.623
80%	1.135	30%	0.602
75%	1.045	25%	0.516
70%	0.961	20%	0.501
65%	0.843	15%	0.424
60%	0.782	10%	0.332
55%	0.734	0	0.425

TABLE 6. Some typical value-pairs of curing temperature and time to reach certain curing degrees in the recommended optimum range*.

	Curing Degrees								
	80%			85%			90%		
T (ºC)	160	170	180	160	170	180	160	170	180
t (s)	16.5	10.1	6.4	20.3	12.5	7.8	26	16	10

*T is the curing temperature, t is the curing time, and s is seconds.

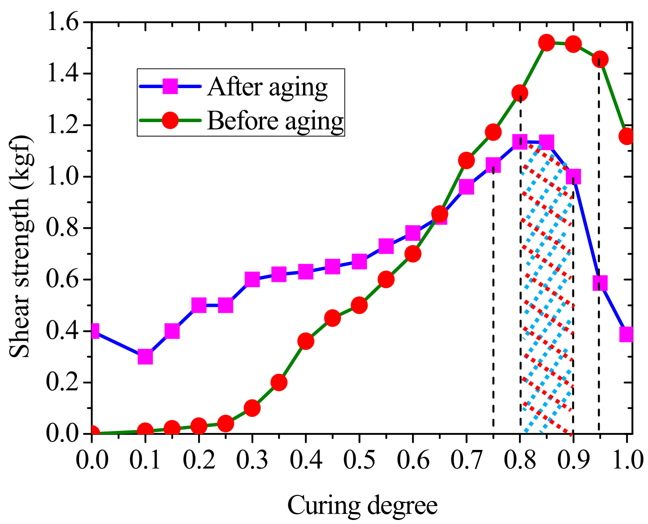


FIGURE 11. Plot of shear strength versus curing degree both before and after aging conditions.

According to Figure 10, it can be found that the shear strength from 0% to 65% increases after 168 h aging compared to the results in Table 4. This result implies that the adhesive comes secondary curing among 0% to 65% curing degree, so the cross-linking of the ACA interface is stronger than before and the shear strength has increased. However, the shear strength decreases sharply between 90% to 100% curing degrees, which means that too a high curing degree will easily lead to shear strength degradation under hygro-thermal aging conditions. Figure 10 shows that the optimum curing degree to achieve highly reliable joints is from 80% to 85% and the optimum curing degree range is from 75% to 90%.

Then, considering the shear strength of ACA joints both before and after the hygro-thermal aging test, the optimum curing degree value as well as the recommended range can be obtained. The relationship curves of shear strength and curing degree both before and after aging are shown in Figure 11.

As shown in Figure 11, for the 80%∼90% curing degree, the shear strength remains at a relatively high level both before and after aging conditions, and the optimum curing degree to achieve highly reliable joints is 85%. Therefore, the recommended optimum curing degree range to achieve highly reliable joints is from 80% to 90%. Herein, using Equation (9), some typical value-pairs for the curing time and temperature are chosen for the optimum curing degree range, i.e., 80%∼90%, and listed in Table 6.

It should be stated that the AC265 datasheet also gives some recommended range of curing parameters for users or customers, e.g., at temperatures between +150 °C and +210 °C at the adhesive in 6 to 19 s using a thermode [30]. The datasheet, however, does not provide a method to select the optimum parameters. Users, therefore, still do not know how to choose the appropriate parameters from the datasheet. Compared with the AC265 datasheet, this study provides a methodology to find the appropriate parameters and gives more detailed information on how to optimize curing parameters in order to improve the reliability of the ACA joints. The developed method can also be used to optimize the curing parameters for other ACAs.

IV. SUMMARY AND CONCLUSION

This paper identified the curing temperature T and curing time t to optimize the ACA curing process using a curing degree metric, α. The curing kinetics of the ACA was investigated by experiments, and the curing kinetics equation for curing degree α, curing temperature T, and curing time t was established for various ACAs. The measure of success was based on the shear strength of ACA joints both before and after hygro-thermal aging tests.

The results show that the optimum value for the curing degree is 85% and the recommended range is from 80% to 90% for acceptable ACA joints. The recommended curing parameters to reach the desirable curing degree can then be calculated by the curing kinetics equation of the ACA. For example, when the curing degree is 85%, one recommended pair of values for curing temperature and curing time is 170 °C and 12.5 s, respectively. This provides support to optimize the curing process in order to improve the reliability of various ACA-based packaging applications, such as the chip-on-glass (COG) packaging for LCDs and flip-chip bonding of electronics chips.

For other ACAs, this process can be used to determine the curing degree and to optimize the bonding parameters. It should be noted that, for different expected use conditions or different adhesive materials, the optimum curing degree and recommended range may not be the same as in this paper.

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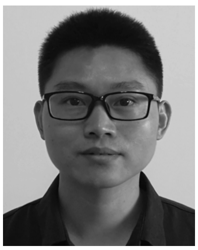

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