# Volume Shrinkage Characterization of Underfill Materials

Shang-Shiuan Deng, Chia-Yi Ho, Huei-Huang Lee, Sheng-Jye Hwang, and Durn-Yuan Huang

***Abstract*—In general, different coefficients of thermal expansion (CTE) values of constitutive materials are considered as the main cause of warpage for flip chip packages. Accordingly, during the past years, the cure-induced volume shrinkage in integrated circuit packages has been neglected in warpage simulation. However, in recent years, there are more and more evidences showing that it is not sufficient to predict the amount of warpage if considering only the CTE values of different component materials in a package especially after molding. There have been some reports about cure induced shrinkage for EMC materials. However, there is no cure induced volume shrinkage report about underfill materials. In this study, the behavioral evolution of volume shrinkage of underfill material during curing process was described by a pressure-volume-temperature-cure (P–V–T–C) equation. The P–V–T–C equation of the underfill material was developed by employing the thermal analyzer, dilatometer and statistical technique. Three successive procedures were conducted for the measurement of the P–V–T–C relation. First, the reaction kinetics and isothermal conversion of the underfill were determined by employing DSC and statistical technique. Second, isobaric and isothermal volume shrinkage of underfill was monitored with a pressure-temperature-controlled dilatometer. Third, the P–V–T–C equation was established by combining the data of volume shrinkage and conversion.**

***Index Terms*—Cure-induced shrinkage, flip chip package, pressure-volume-temperature-cure (P-V-T-C) equation, underfill, warpage.**

## I. Introduction

UNDERFILL material is one kind of thermosetting polymers. The underfill is epoxy resin based and usually filled with $SiO_2$, curing agent and other additives. Table I lists the basic underfill physical properties in this study. For a Flip Chip package, underfill material acts as a structural material to reduce the stress level between the die and the substrate. It was injected to the gap between the die and the substrate by capillary action. The cure reaction is caused by heating. The die and the substrate are tightly bonded by underfill material after curing. However, chemical-induced volume shrinkage is a by-product of the cure reaction, and affects the residual stress and the amount of warpage in a package.

Manuscript received July 26, 2009; revised May 26, 2010; accepted May 26, 2010. Date of publication December 23, 2010; date of current version March 23, 2011. This work was recommended for publication by Associate Editor D. Lu upon evaluation of the reviewers comments.

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Digital Object Identifier 10.1109/TCPMT.2010.2100950

TABLE I
BASIC PHYSICAL PROPERTIES OF THE UNDERFILL MATERIAL

| | |
|---|---|
| Specific Weight | 1.6 |
| Viscosity (at 25°C) | 60 Pa•s |
| $T_g$ | 137°C |
| CTE (Below $T_g$) | 32 ppm/°C |
| CTE (Above $T_g$) | 120 ppm/°C |

In most studies previously, the amount of cure-induced volume shrinkage was ignored from calculation of warpage and residual stresses [1]–[3]. In recent years, it was known that considering CTE difference between constitutive materials in a package is not accurate enough to predict warpage and residual stresses for most of the modern integrated circuit (IC) packages. This case is especially serious for packages after molding and before post mold cure.

Kelly *et al.* [4], [5] predicted warpage in IC plastic encapsulated by considering both thermal-induced shrinkage and cure-induced shrinkage of the encapsulated compound. Nishimura and Nakagawa designed an apparatus to measure the volume shrinkage of liquid compound under isothermal conditions [6]. They also proposed a model concerning temperature-volume-cure relation for liquid encapsulants. Snow and Armistead measured the volume shrinkage of polymer during cure reaction by dilatometer [7]. Chang and Hwang had developed a test procedure [8] by using a double-plunger-type dilatometer to monitor the evolvement of non-isothermal volume change and proposed a pressure-volume-temperature-cure (P-V-T-C) relation [9]. Later, they developed a single plunger type dilatometer. Isobaric and isothermal volume variation of EMC was monitored with a single-plunger-type pressure-temperature-controlled dilatometer. The pressure-volume-temperature-cure equation of EMC was thus established [10]. Hong and Hwang [11] used this P-V-T-C relation to establish the stress-strain formulation that predicts warpage after mold-filling process. By using finite element method and mold flow analysis which showed that the quantity of warpage was governed by thermal-induced shrinkage and cure-induced shrinkage. Teng and Hwang considered both thermal-induced shrinkage and cure-induced shrinkage to predict warpage of IC products by FEM and also considered paddle shift problem during filling process [12], [15]–[17]. They found that the amount of warpage induced by CTE mismatch was of the same dimension as that induced by cure induced shrinkage after encapsulation process.

However, there is still no data describing the volume shrinkage behavior of underfill materials. The underfill material is liquid before curing, thus is different from EMC pellets. In

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TABLE II
REACTION PARAMETERS OF THE KINETIC MODEL FOR THE UNDERFILL MATERIAL

| Parameter | Value |
|---|---|
| $m$ | 1.24 |
| $n$ | 1.66 |
| $A_1$ (1/sec) | $8.74\text{x}10^4$ |
| $A_2$ (1/sec) | $7.35\text{x}10^5$ |
| $E_1$ (K) | $7.30\text{x}10^3$ |
| $E_2$ (K) | $7.24\text{x}10^3$ |

this paper, a procedure was developed to measure the volume shrinkage behavior of the underfill material and a P-V-T-C relation was established for future computation need.

## II. PROCEDURES

The approach presented here contains four procedures and described as follows.

1) The cure reaction was measured by DSC thermal analyzer and the reaction kinetics of an underfill material was determined by employing a statistical technique. The conversion data in several isothermal states could be determined.
2) To observe the effect of cure-induced shrinkage of underfill material, the volume shrinkage was measured experimentally in an isothermal and isobaric state by a P-V-T-C Tester [9]. The viscosity of underfill material before curing is low. Thus, preheating of the underfill material was executed before pressure was applied during measurement in order to prevent resin overflow in the mold.
3) The relationship between pressure, volume shrinkage, temperature and conversion was built by combining data of conversion and volume shrinkage. The relation was formulated by statistical technique.
4) Since the underfill material was preheated, the conversion value was not zero at the beginning of the test. By combining the offsetting formulation and original equation, the P-V-T-C equation of the underfill material was established. And the volume shrinkage under no-pressure state could also be predicted by the P-V-T-C equation.

During the progressing of cure reaction, the reaction rate ($\dot{C}$) is defined as the heat flow divided by the overall heat of reaction [8]. The dynamic testing method is adopted with a Perkin-Elmer DSC-7 instrument under a dry nitrogen atmosphere and the instrument is kept well calibrated. The kinetic model used here is the $n$th-order autocatalytic model of Kamal [13], [14] which investigate the reaction kinetics of the given underfill. The detailed mathematical model is expressed as follows:

$$\dot{C} = \frac{dC}{dt} = (k_1 + k_2 C^m)(1 - C)^n \tag{1}$$

$$k_1 = A_1 \exp\left(\frac{-E_1}{T}\right) \tag{2}$$

$$k_2 = A_2 \exp\left(\frac{-E_2}{T}\right) \tag{3}$$

where $C$ was the conversion (degree of cure). $k_1$ and $k_2$ were the reaction rate constants related to the Arrhenius temperature

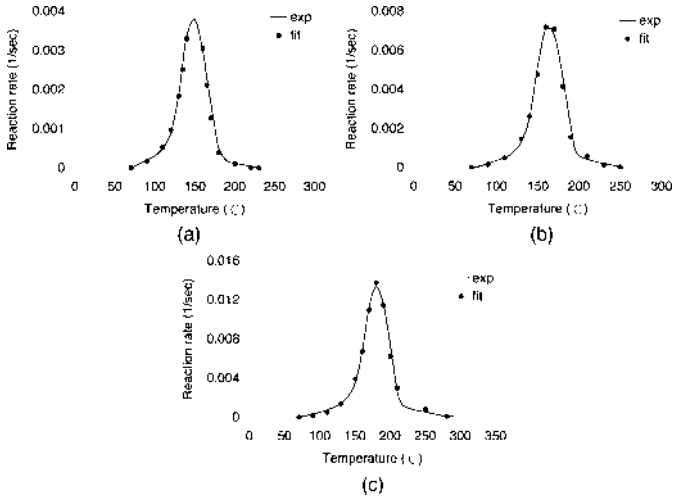


Fig. 1. Reaction rate versus temperature for the underfill material at (a) 10°C/min, (b) 20 °C/min, (c) 40 °C/min.

dependency with two different factors of reaction frequency $A_1$ and $A_2$, respectively. $E_1$ and $E_2$ were constants of activation energy. $m$ and $n$ were the pre-exponential factors.

The six parameters $A_1, A_2, E_1, E_2, m$, and $n$ were determined with statistical regression analysis or a commercial statistical software package such as SPSS.

However, no explicit expression for the isothermal condition of conversion exists because of the non-integral characteristic of the (1). The conversion versus time in an isothermal state cannot be obtained directly from (1). Nevertheless, it can be approximated using the following equation [10]:

$$\begin{aligned} &C_{\mathrm{i}}(t)_{T=\mathrm{const.}} \\ &\quad = C_{\mathrm{i}-1} + \Delta t \times \dot{C}_{\mathrm{i}-1} \\ &\quad = C_{\mathrm{i}-1} + \Delta t \left(k_1 + k_2 C_{\mathrm{i}-1}^m\right)(1 - C_{\mathrm{i}-1})^n. \end{aligned} \tag{4}$$

The initial conversion $C_0$ is zero at the starting time $t_0$, and the conversion at any instant $t_{\mathrm{i}}$ is expressed by $C_{\mathrm{i}}$, which accumulates during former conversion $(C_{i-1})$ and $(\Delta t \cdot C_{\mathrm{i}-1})$ at a time increment in a short period of time. Moreover, (4) is very convenient for computation using Microsoft Excel or any kind of computer languages.

Table II listed all parameters of Kamal's model for the underfill material. Fig. 1 showed the comparison between Kamal's model and the experimental data at different raise temperature rate. Figs. 2 and 3 showed the plots of conversion and reaction rate for the underfill material in three isothermal states: 140 °C, 150 °C, and 160 °C.

In this study, isothermal and isobaric volume shrinkage of the underfill material was measured by a pressure-temperature-controlled dilatometer, which was based on the concept of single-plunger-type [9]. The schematic of the dilatometer was shown in Figs. 4 and 5. The isothermal and isobaric volume shrinkage was defined as

$$VS_{\mathrm{i}}(P,t)_{T=\mathrm{const.}} = \frac{V_0 - V_{\mathrm{i}}}{V_0} = \frac{h_0 - h_{\mathrm{i}}}{h_0} \tag{5}$$

where $V_0$ was the volume of a sample at the starting time (the time when cavity pressure reached a preset pressure level). $V_{\mathrm{i}}$ was the volume of a sample at any instant. The volume change,

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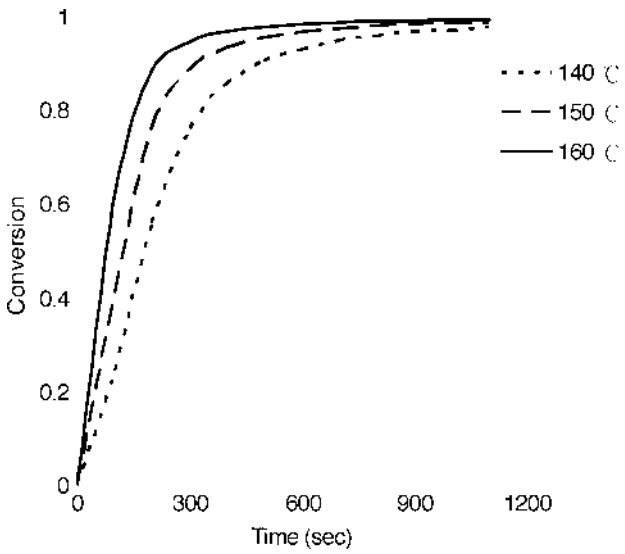


Fig. 2. Conversion versus time for the underfill material.

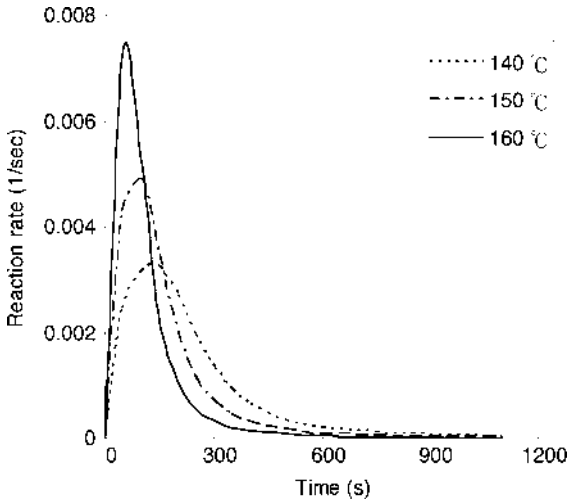


Fig. 3. Reaction rate versus time for the underfill material.

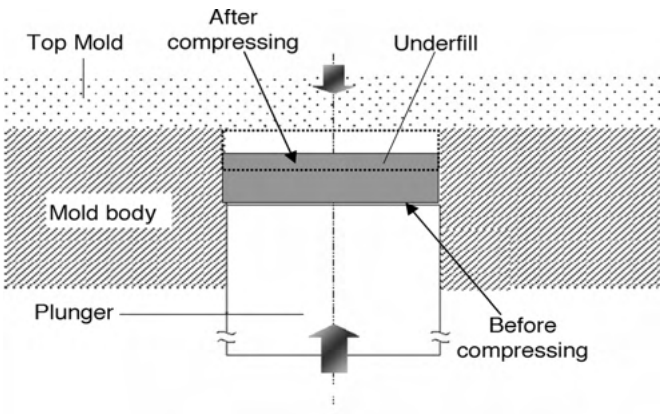


Fig. 4. Local structure of the single-plunger-type dilatometer.

$V_0 - V_i$, was calculated by measuring the decrease in the height of a sample $(h_0 - h_i)$ in the mold cavity.

In the study, experiment measurements of volume shrinkage under three different temperatures: 140 °C, 150 °C, and 160 °C

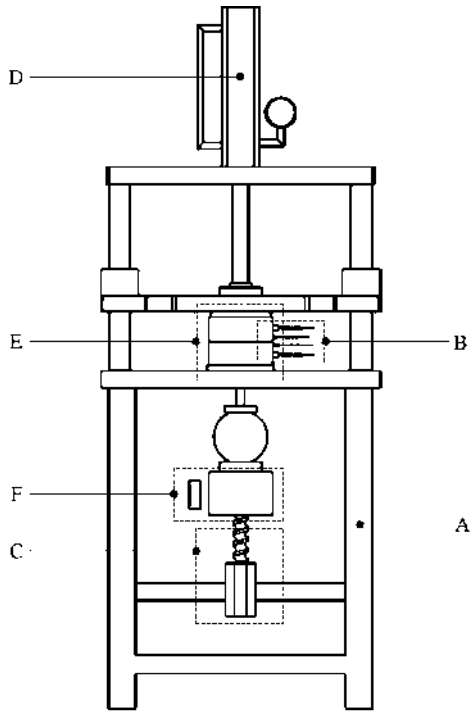


Fig. 5. Schematic of the P-V-T-C Tester. A: Frame. B: Heater controller. C: Loading actuator. D: Pneumatic-hydraulic actuator for clamping system. E: Mold. F: Data acquisition.

and three different pressures: 0.56, 0.98, and 1.40 kgf/cm$^2$ were performed for the underfill material. In order to avoid overflow, the underfill material needed to be preheated for a while before experiment due to its good fluidity (low viscosity) under room temperature before curing. The preheating period was determined by DSC experiment data, so the preheating period was different for different experiment temperature. From DSC experiment, it was found that when conversion of underfill material is larger than 0.135, leakage in the dilatometer would not happen. Thus, the underfill material was preheated to conversion of 0.135 and the preheating period for different temperature was thus determined. The test results under three pressure and three temperature levels were shown in Fig. 6.

The relationship of pressure, volume shrinkage, temperature and conversion could be built by combining data of kinetics model and volume shrinkage experiment. Fig. 7 was the results of combining data. In Fig. 7, the preheating period of the underfill material was not considered for all the experiments. The beginning point was not from the origin. Further offset of the curve needed to be done in order to have a complete model. The relationship of the volume shrinkage and conversion was assumed to be linear at the beginning of the measurement. Linear extrapolation was used to find the volume change during the preheating period by connecting the experiment data points when conversion was 0.135 and 0.4. The intersectional point of the vertical coordinate line and the line connecting the experiment data points of conversion 0.135 and 0.4 was assumed to be the negative value of the initial volume change during the preheating period. Then the offset was executed by adding the initial volume change during the preheating period to the experiment data in Fig. 7.

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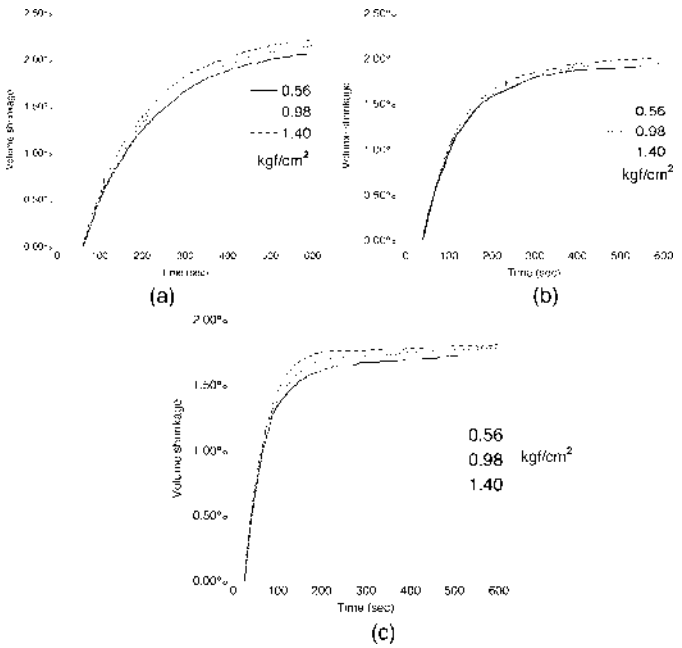


Fig. 6. Volume shrinkage versus time for the underfill material at three pressure levels under (a) 140 °C, (b) 150 °C, (c) 160 °C.

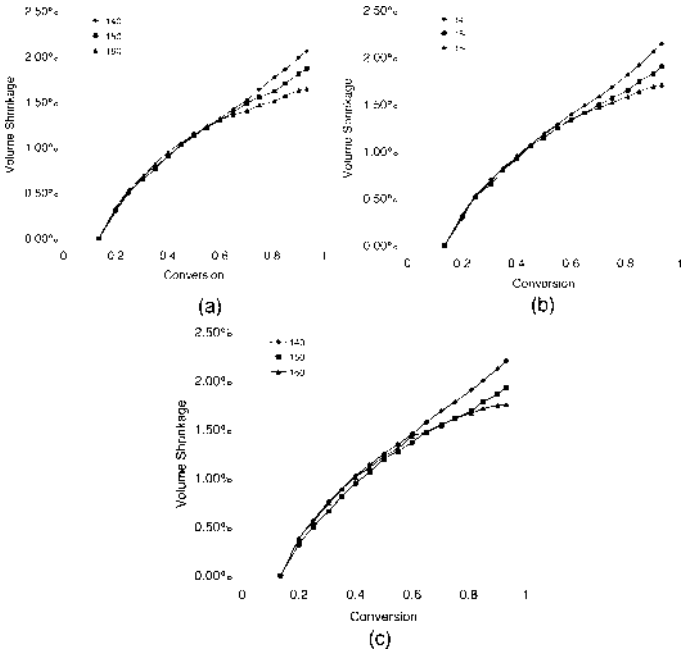


Fig. 7. Volume shrinkage versus conversion for the underfill material at three temperature levels under (a) 0.56 kgf/cm$^2$, (b) 0.98 kgf/cm$^2$, (c) 1.40 kgf/cm$^2$.

## III. P-V-T-C Equation

In most studies, the cure and resin compressibility induced shrinkage effect was ignored or the cure induced shrinkage ration was assumed to be a constant. From Hwang's research [8], it was found that the cure/compressibility-induced volume shrinkage of EMC was affected by pressure, temperature and degree of cure. So equation describing the cure induced and resin compressibility volume shrinkage as a function of pressure, temperature, and degree of cure.

TABLE III
Data-Fitted Coefficients of the P-V-T-C Equation for the Underfill Material

| Coefficients | Value |
|---|---|
| $a_2$ | $1.223\times10^{-5}$ |
| $a_1$ | $-2.227\times10^{-3}$ |
| $a_0$ | $1.156\times10^{-1}$ |
| $b_2$ | $4.231\times10^{-4}$ |
| $b_1$ | $-1.363\times10^{-1}$ |
| $b_0$ | $1.112\times10^{1}$ |
| $c_2$ | $-7.291\times10^{-6}$ |
| $c_1$ | $6.074\times10^{-4}$ |
| $c_0$ | $3.514\times10^{-2}$ |

By combining the data of volume shrinkage and conversion of the underfill material, the relationship of P-V-T-C could be formulated. By observing the results of volume shrinkage, the equation was proposed to be in the form as

$$\mathrm{VS}(P,T,C) = F_1(P,T)\cdot C^{F_2(P,T)} + F_3(P,T) \quad (6)$$

$$F_1 = a_2 f^2(P,T) + a_1 f(P,T) + a_0 \quad (7)$$

$$F_2 = b_2 f^2(P,T) + b_1 f(P,T) + b_0 \quad (8)$$

$$F_3 = c_2 f^2(P,T) + c_1 f(P,T) + c_0 \quad (9)$$

$$f = T - 4\cdot P \quad (10)$$

where $F_1, F_2, F_3$, and $f$ were functions of pressure (kgf/cm$^2$) and temperature (°C). In this study, experiments of volume shrinkage measurement under three different temperatures: 140 °C, 150 °C, and 160 °C and three different pressures: 0.56, 0.98, and 1.40 kgf/cm$^2$ were performed for the underfill material. These nine data-fitted coefficients: $a_2, a_1, a_0, b_2, b_1, b_0, c_2, c_1$, and $c_0$ for the underfill material were listed in Table III. Fig. 8 showed the comparison of volume shrinkage between experiment data and model under three isothermal and three isobaric states for the underfill material.

The P-V-T-C equation used these mathematic forms without apparent physical significance. The P-V-T-C equation shown here was only a result of data fitting.

In this study, confirm experiments were also executed to avoid overfit and ensure the P-V-T-C equation could use under different conditions. The confirm experiments of volume shrinkage measurement were executed under two different temperatures: 145 °C and 155 °C and three different pressures: 0.56, 0.98, and 1.40 kgf/cm$^2$. The confirmed test results under three pressure and two temperature levels were shown in Figs. 9 and 10. It was found the P-V-T-C equation fit the confirm experiment data well under different conditions.

To offset the preheating time in the model, a new function $F_4(P,T)$ which contained pressure and temperature terms was

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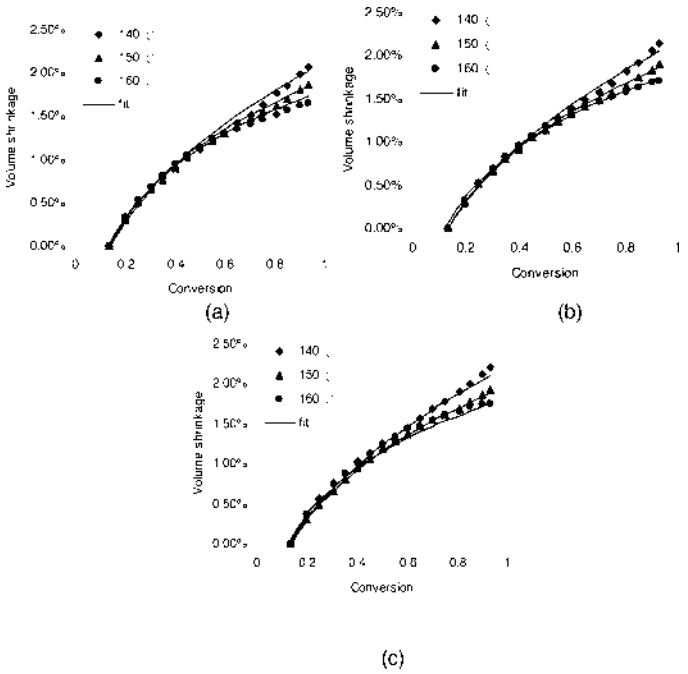


Fig. 8. Comparison of volume shrinkage between experiment data and model at three temperature levels under (a) 0.56 kgf/cm$^2$, (b) 0.98 kgf/cm$^2$, (c) 1.40 kgf/cm$^2$.

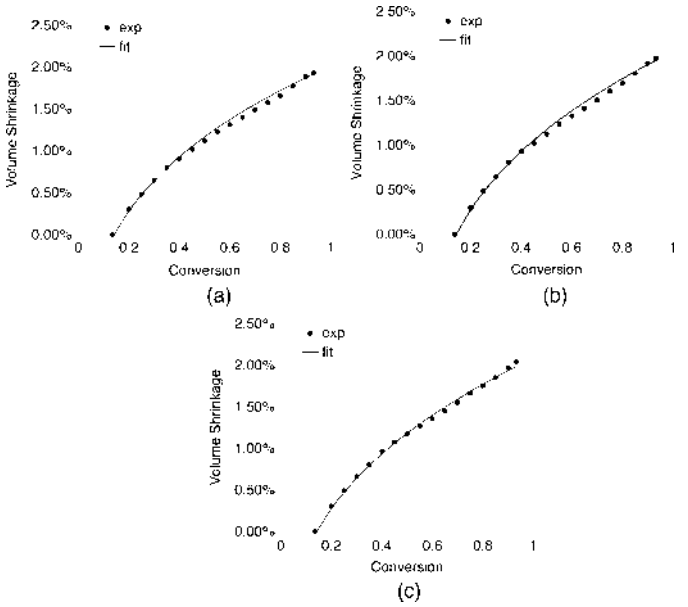


Fig. 9. Volume shrinkage versus conversion for the underfill material at 145 °C under (a) 0.56 kgf/cm$^2$ (b) 0.98 kgf/cm$^2$ (c) 1.40 kgf/cm$^2$.

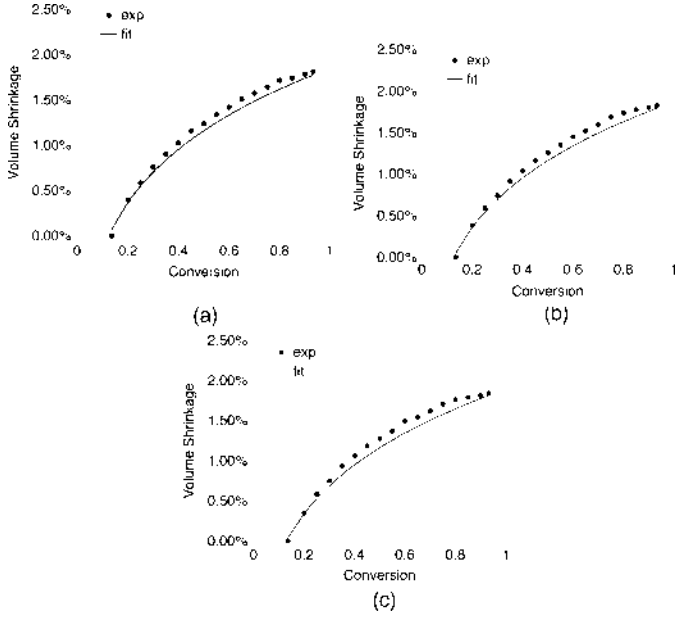


Fig. 10. Volume shrinkage versus conversion for the underfill material at 155°C under. (a) 0.56 kgf/cm$^2$ (b) 0.98 kgf/cm$^2$ (c) 1.40 kgf/cm$^2$.

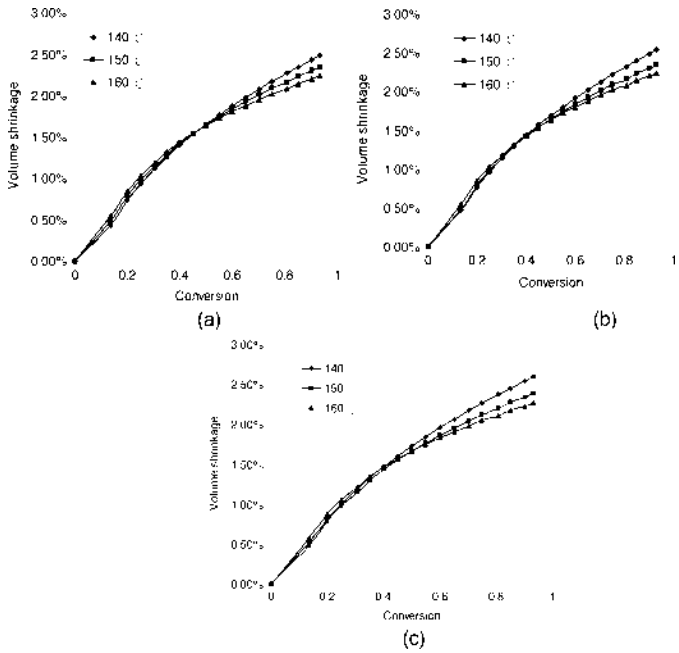


Fig. 11. Prediction of total volume shrinkage at three temperature levels under. (a) 0.56 kgf/cm$^2$. (b) 0.98 kgf/cm$^2$. (c) 1.40 kgf/cm$^2$.

proposed. $F_4(P,T)$ was found by extrapolation so the total amount of cure induced volume shrinkage could be found. The formulation $F_4(P,T)$ was proposed to be in the form as (11) and (12).

The modified P-V-T-C equation was then defined as (13). Fig. 11 showed the offset results of P-V-T-C relation of total volume shrinkage versus conversion for the underfill material. Furthermore, the underfill material is injected into the gap between the chip and the substrate by capillary action during the encapsulation process. The underfill material is supposed to maintain basically at zero gauge pressure state during the process. Therefore, the zero gauge pressure state was predicted by P-V-T-C equation in this study as shown in Fig. 12

$$\mathrm{F}_4 = d_1 x + d_0 \tag{11}$$

$$x = P \cdot T + T \tag{12}$$

where $d_1 = 3.183 \times 10^{-6}, d_0 = 3.903 \times 10^{-3}$

$$\mathrm{VS} = F_1(P,T) \cdot C^{F_2(P,T)} F_3(P,T) + F_4(P,T). \tag{13}$$

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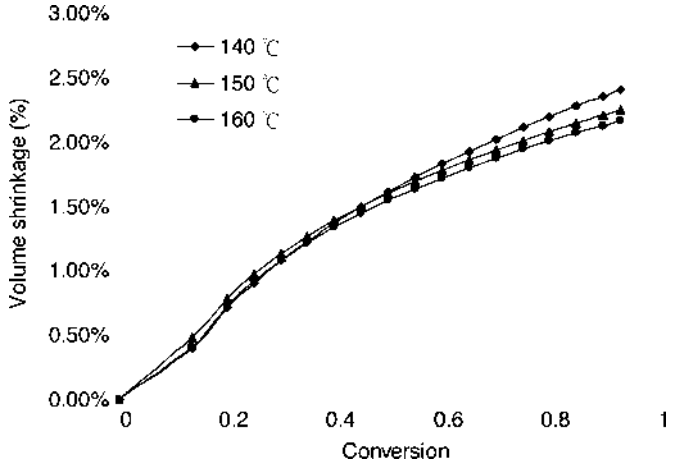


Fig. 12. Prediction of volume shrinkage for the underfill material under three temperature levels and zero gauge pressure state by the P-V-T-C equation.

## IV. Discussion and Conclution

From the test results, the total amount of cure induced volume shrinkage of underfill material was strongly affected by both process pressure and process temperature.

The higher was the process pressure, the larger amount was the volume shrinkage. There has been physical evidence for this. When the process temperature was higher, the cure reaction rate would be higher and the available relaxation period for the molecular chains would be shorter. The cross linked structure of the polymer of higher process temperature case would not be as organized as that of the lower process temperature case. Thus, the higher was the process temperature, the smaller amount was the volume shrinkage. In comparison with pressure, temperature affected more of the total amount of cure induced volume shrinkage. The amount of volume shrinkage for the underfill material during cure process could be as high as 2.5%.

The P-V-T-C relation of the underfill material was successfully measured via a single plunger type dilatometer. The dilatometer was designed to measure volume shrinkage of epoxy molding compound under isothermal and isobaric states. Thus, without preheating the liquid underfill material first, there would be overflow in the tool. To avoid overflow in the tool, preheating was executed. Volume shrinkage during preheating time was not known. An extrapolation technique was proposed to compensate the volume shrinkage during the preheating time. The total amount of cure induced volume shrinkage during encapsulation could be found.

With the measurement result, the P-V-T-C mathematic model was proposed and this model could predict the total amount of cure induced volume shrinkage well.

With this model, the total amount predicted volume shrinkage under zero gauge pressure state could also be predicted. This P-V-T-C relation could be applied by engineers to predict the total amount of cure induced volume shrinkage. Combining the cure induced volume shrinkage data and volume shrinkage due to CTE mismatch, the amount of warpage of flip chip package after the underfill material was cured could be found.

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