Micromechanical Modeling of Stress Evolution Induced During Cure in a Particle-Filled Electronic Packaging Polymer

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Abstract—**Particle-filled polymers are widely used in electronic industries. From microscale view, cure-induced residual stress can be generated not only by the external constraints but also by the constraint effect among the particles. In this paper, a three-dimensional micromechanical finite element method (FEM) model has been setup for a silica particle filled epoxy. In the micromechanical model, the epoxy matrix is modeled with a previously developed cure-dependent viscoelastic constitutive model, whereas the silica particles are modeled as elastic with high stiffness. Cure shrinkage is applied to the matrix as an initial strain for each time increment. The cure-dependent viscoelastic properties were obtained from shear and tension-compression dynamical mechanical analysis measurements. Cure shrinkage and reaction kinetics were characterized with online density measurement and differential scanning calorimeter measurements, respectively. In order to simulate a partly constrained object, the micromechanical model is coupled with a macromodel FEM analysis. The displacements from the macromodel are used as boundary conditions for the micromodel. The effect of external constraints on the generation of the micro stresses is studied by using the boundary conditions related to different external constrained states.**

Index Terms—**Cure-induced stress, curing process, micromechanical modeling, particle-filled composite, viscoelasticity.**

I. INTRODUCTION

THERMOSETTING polymers are widely used in electronic industries, for instance, as adhesives, underfills, and encapsulants. They are usually highly filled with particles to decrease the coefficient of thermal expansion (CTE) and increase the modulus to meet the specific requirements for electronic packaging. As one of the major packaging processes, the curing process will introduce residual stress if the polymerization shrinkage is (partly) constrained. For a highly filled thermosetting polymer, from microscale view, residual stress may be generated during the curing process not only by the external constraints but also by the constraint effect among the particles. If the stress is high enough to exceed the cohesive strength of the matrix or the adhesion strength of the particle and the matrix, (micro) damage

in the matrix or debonding in the particle/matrix interface will be initiated. Therefore, in order to investigate the root cause of the failure issues in packaging polymers, it is necessary to get insight of stress buildup and evolution in curing polymers at microscale. Many researchers have used micromechanical models to study the mechanical behavior of metal matrix composites (MMC) and the generation of residual stresses in MMC [1]–[3]. This methodology has been applied to polymer-based composites [4]–[6]. Recently, Chen *et al.* [6] used a three-dimensional (3-D) micromechanical model to investigate the evolution of residual stresses in a [0/90] glass fiber/epoxy cross-ply laminate. However, to the best of the authors' knowledge, so far no finite element modeling (FEM) microscale results on the cure-induced stress of a particle-filled polymer have been presented.

There are a few difficulties concerning the microscale simulation of cure-induced stresses in a particle-filled polymer. First of all, it is a challenging task to establish proper models to describe the material property development in the matrix resin during the curing process. The curing process is not only a physical process, but also a chemical process. During the curing process, the thermosetting polymer transforms from a liquid state into a viscoelastic solid, accompanied with crosslinking shrinkage. So far, no well developed model for curing process is available. Second, it is very difficult to characterize experimentally the material parameters by current technology because of ongoing change of the properties during cure. Third, proper boundary conditions should be incorporated in the simulation to model the effect of the external constrained state on the microscale stresses.

In this paper, as a first attempt to model the microscale cure-induced stresses in the particle-filled polymer, first, a previously developed cure-dependent viscoelastic constitutive model [7], [8] is discussed and extended to take into account the temperature effect. A model based on scaling analysis is adopted to describe the evolution of the equilibrium modulus. In Section III, the experimental characterization of the viscoelastic behavior, the cure shrinkage, and the reaction kinetics is presented in detail. Then, in Section IV, a (3-D) micromechanical FEM model has been set up for a silica particle-filled epoxy. The viscoelastic model for the resin matrix is implemented in a FE code. A macromodel FEM analysis is carried out to simulate the stress and strain evolution in a disk shaped specimen of the filled epoxy, which could be considered to be in a nearly 3-D constrained state at the middle part. The displacements from the macromodel are used as the boundary conditions for the micromodel. The effect of external constraints on the generation of

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the micro stresses is studied by using the boundary conditions related to different external constrained states.

II. MATERIAL MODELS FOR MATRIX RESIN DURING CURE

A. Viscoelastic Model

A cure-dependent model was proposed to describe the evolution of the viscoelastic behavior of thermosetting polymers during the curing process [7], [8]. It was applied for study on the curing-induced stress of the underfill resin in a flip chip package and its effect on the reliability issues [9], [10]. Though our previous effort has led to qualitatively reasonable results being verified by experiments, it is limited to a relatively low curing rate, because of the limitation of dynamical mechanical analysis (DMA) method for continuing cure. In this paper, the model is extended by incorporating a rubbery model to describe the equilibrium moduli and by considering the temperature effect. In a stress relaxation form, the constitutive relation can be written as a cure-dependent hereditary integral form

$$\sigma_i = \int_0^t C_{ij}\left\{\alpha, T, (t-\xi)\right\}$$
$$\cdot \left\{\left(\frac{\partial \varepsilon_j}{\partial \xi}\right)_\xi - \left(\frac{\partial \varepsilon_j^*}{\partial \xi}\right)_\xi\right\} d\xi, \quad (i,j=1,\ldots,6) \quad (1)$$

where

σ_i	stresses;
C_{ij}	stress relaxation functions;
t	current time;
ξ	curing time or loading time;
T	the curing temperature or loading temperature;
α	degree of conversion;
ε_j	strains;
ε_j^*	initial strains, including thermal and curing shrinkage strain, etc.

It should be mentioned that C_{ij}, the relaxation moduli, are supposed to be cure and temperature dependent. The key issue is how to establish the relaxation functions.

If the material is assumed to be isotropic, the relaxation moduli C_{ij} can be decomposed into two independent parts, i.e., the shear relaxation modulus G and the bulk relaxation modulus K

$$C_{ij}\left\{\alpha, T, (t-\xi)\right\} = K\left\{\alpha, T, (t-\xi)\right\} \cdot V_{ij}$$
$$+ G\left\{\alpha, T, (t-\xi)\right\} \cdot D_{ij} \quad (2)$$

where $[V_{ij}]$ and $[D_{ij}]$ are the volumetric and deviatoric (constant) coefficient matrices, with

$$[V_{ij}] = \begin{vmatrix} 1 & 1 & 1 & 0 & 0 & 0 \\ 1 & 1 & 1 & 0 & 0 & 0 \\ 1 & 1 & 1 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 \end{vmatrix} \quad (3)$$

$$[D_{ij}] = \begin{vmatrix} \frac{4}{3} & -\frac{2}{3} & -\frac{2}{3} & 0 & 0 & 0 \\ -\frac{2}{3} & \frac{4}{3} & -\frac{2}{3} & 0 & 0 & 0 \\ -\frac{2}{3} & -\frac{2}{3} & \frac{4}{3} & 0 & 0 & 0 \\ 0 & 0 & 0 & 2 & 0 & 0 \\ 0 & 0 & 0 & 0 & 2 & 0 \\ 0 & 0 & 0 & 0 & 0 & 2 \end{vmatrix}. \quad (4)$$

The generalized Maxwell model with a limited number of elements (Prony series) is used for approximating the shear and bulk relaxation moduli functions, because it is convenient and effective for numerical implementation and it is flexible to expand as needed. The bulk and shear moduli have the following form:

$$K\{\alpha, T, (t-\xi)\} = K^\infty(\alpha, T)$$
$$+ \sum_{n=1}^N K^n(\alpha, T) \cdot e^{-(t-\xi)/\tau_n} \quad (5)$$

and

$$G\{\alpha, T, (t-\xi)\} = G^\infty(\alpha, T)$$
$$+ \sum_{n=1}^N G^n(\alpha, T) \cdot e^{-(t-\xi)/\tau_n} \quad (6)$$

where $G^n(\alpha, T)$ and $K^n(\alpha, T)$ are the nth stiffness coefficients, and τ_n is the nth relation time. $G^\infty(\alpha, T)$ and $K^\infty(\alpha, T)$ are the shear and bulk moduli for infinitely large relaxation times, which actually represent the evolution of the "rubbery" shear and bulk moduli with the degree of conversion. Due to the ongoing cure, direct measurement of $G^\infty(\alpha, T)$ and $K^\infty(\alpha, T)$ with high accuracy by using current technology is very hard. Based on scaling analysis, Adolf *et al.* [11] presented a model to describe the evolution of the equilibrium shear modulus

$$G^\infty(\alpha) = G_f^\infty \left[\frac{\alpha^2 - \alpha_{gel}^2}{1 - \alpha_{gel}^2}\right]^{8/3} \quad (7)$$

where $\alpha(\xi)$ and α_{gel} are the extent of reaction at curing time ξ and at the gel point, respectively. G_f^∞ is the rubbery shear modulus at fully cured state ($\alpha = 1.0$). We assumed that the equilibrium bulk modulus has a similar dependence on the degree of conversion

$$K^\infty(\alpha) = K_l + \left(K_f^\infty - K_l\right) \left[\frac{\alpha^2 - \alpha_{gel}^2}{1 - \alpha_{gel}^2}\right]^{8/3} \quad (8)$$

where K_l and K_f^∞ are the bulk moduli in the liquid state and at the fully cured state, respectively.

A generalized Vogel equation [12] is used to describe the temperature effect on the relaxation times. Assuming that all relaxation times be shifted in the same way, the shift factor is expressed as

$$\log a_T = \log \frac{\tau_n(T)}{\tau_n(T_{ref})} = \frac{C}{T - T_\infty} - \frac{C}{T_{ref} - T_\infty} \quad (9)$$

where C is a constant.

B. Cure Kinetics Models

In order to model the evolution of the cure-dependent viscoelastic properties, we need to know the cure kinetics. The differential scanning calorimeter (DSC) has been widely used to obtain the curing process parameters, such as the degree and rate of chemical conversion, glass transition temperature (T_g). The degree of conversion is commonly determined from the residual heat of reaction (ΔH_R) of the partially cured resin measured by DSC, as

$$\alpha = 1 - \frac{\Delta H_R}{\Delta H_T} \tag{10}$$

where ΔH_T is the total heat of reaction associated with the fully cured resin.

When the conversion reaches a high level, a quantitative measurement of the residual heat is very difficult due to the small exotherm. In that case, measuring T_g is a more sensitive way to determine the degree of conversion. It is well accepted that for many thermosetting polymers there is a unique relationship between T_g and the degree of conversion. The modified empirical DeBenedetto equation is used to describe such a relationship, as

$$\frac{T_g - T_{g0}}{T_{g\infty} - T_{g0}} = \frac{\lambda\alpha}{1 - (1 - \lambda)\alpha} \tag{11}$$

where T_{g0} is the glass transition temperature of the material without cure, $T_{g\infty}$ is the glass transition temperature at fully cure state $(\alpha = 1.0)$, and λ is a material constant.

An autocatalytic model, proposed by Kamal and Sourour [13], is used to model the cure kinetics

$$\frac{d\alpha}{dt} = k_T \alpha^m (1 - \alpha)^n \tag{12}$$

where m and n represent the reaction orders, k_T is the reaction rate constant following an Arrhenius temperature dependence:

$$k_T = A \exp\left(-\frac{Q}{RT}\right) \tag{13}$$

where A is a constant, Q the activation energy, R the gas constant, and T the absolute temperature.

To consider the diffusion effect, a diffusion factor, which is based on free-volume theory, is given as [14]

$$f(\alpha) = \frac{1}{1 + \exp[C(\alpha - \alpha_c)]} \tag{14}$$

in which C is a material constant and α_c is the critical conversion depending on the curing temperature. The effective reaction rate at any conversion is considered to be equal to the chemical reaction rate [see (12)] multiplied by $f(\alpha)$.

III. EXPERIMENTAL METHODOLOGY AND RESULTS

A. Materials and Sample Preparation

The epoxy resin used as matrix material in this study was novolac epoxy (EPN 1180) from Vantico, Ltd., with an equivalent weight of epoxy groups equal to 175–182 g/eq. Bisphenol-A was used as hardener, which has an equivalent epoxy weight of
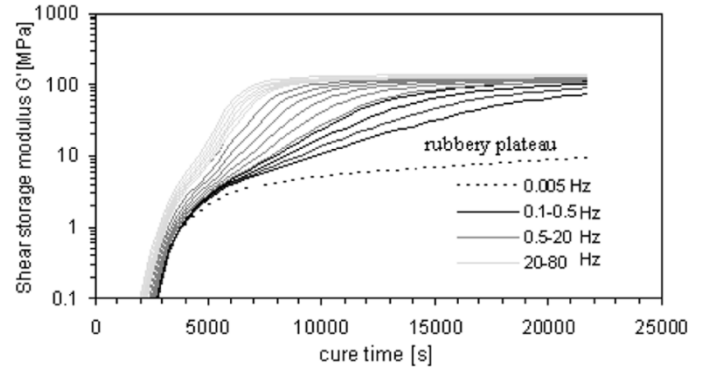

Fig. 1. Shear storage modulus measured from DMA under 120 °C isothermal curing.
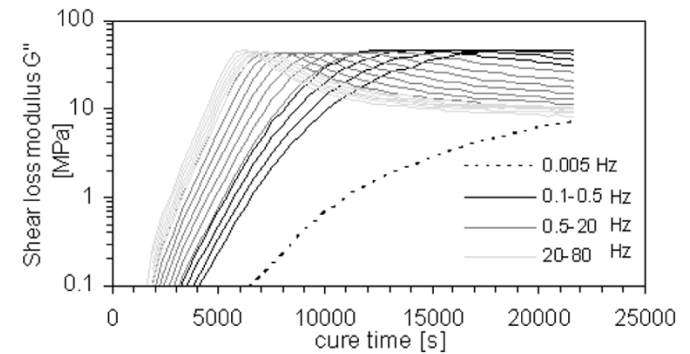

Fig. 2. Shear loss modulus measured from DMA under 120 °C isothermal curing.

46 g/eq, and triphenylphosphine (TPP) as a catalyst. The novolac epoxy and the hardener were mixed in a stoichiometric ratio of 1:1. The catalyst ratio is 0.5 g TPP per 100 g epoxy. Fused silica spheres (FB-940), from Denka Co., were used as filler. The filler has a median diameter of 15 μm, with a density of 2.20 g/cm^3.

Epoxy resin and hardener were well mixed at 150 °C and then cooled down to 85 °C, and the catalyst was added and fully stirred. Afterwards, the mixture was degassed at 75 °C for about 20 min. Then, the mixture was immediately cooled down and stored in a refrigerator at −15 °C. Later it was used for test samples or as matrix resin of the particle-filled composite.
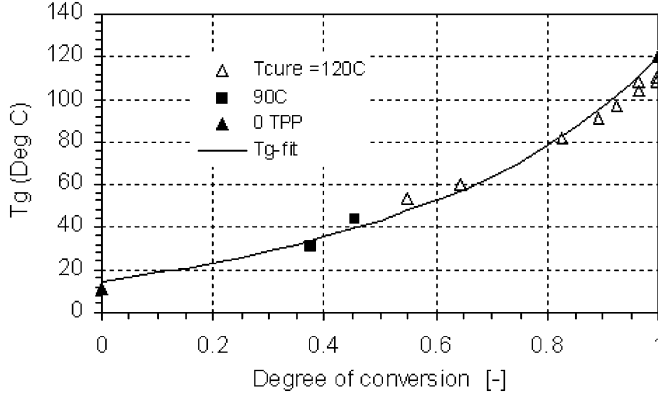
To make a particle-filled composite, a certain amount of filler was added to the matrix resin, and they were well mixed in a special mixer (Kenwood, K-beater). 40%, 50%, and 65 wt% filler were used for this study.

B. DMA Measurements

DMA was used to determine the cure-dependent parameters, i.e., the cure-dependent stiffness coefficients K^n and G^n in (3) and (4). Sandwich shear DMA tests and axial tension-compression DMA tests were performed by using a Metravib Viscoanalyzer VA4000, under isothermal curing conditions. Four groups of frequencies were used, such as: 0.005 Hz, 0.1–0.5 Hz, 0.5–20 Hz, and 20–80 Hz. In the last three groups, several logarithmically spaced frequencies were applied. Figs. 1 and 2 present the shear storage and loss moduli measured from DMA under 120 °C isothermal curing.

TABLE I
RELAXATION TIMES USED IN (5) AND (6)

τ_n	Relaxation time (s)	τ_n	Relaxation time (s)
τ_1	0.00316	τ_7	3.16
τ_2	0.0100	τ_8	10
τ_3	0.0316	τ_9	32
τ_4	0.100	τ_{10}	100
τ_5	0.316	τ_{11}	316
τ_6	1.00	τ_{12}	1000

Fig. 3. Relationship between T_g and the degree of conversion as measured from DSC. The fit of the DeBenedetto equation [see (11)] is shown in the solid line.

Thirteen Prony terms were used, in which the relaxation times τ_n were chosen such that they are logarithmically equally spaced, as shown in Table I. The relaxation time τ_{13} was set to be infinite, thus creating a time independent contribution. The measured storage modulus G' and loss modulus G'' were fitted to (15) and (16) to obtain the stiffness coefficients G^n at each cure state

$$G'\{\alpha, T, \omega\} = G^{\infty}(\alpha, T)$$
$$+ \sum_{n=1}^{N} G^n(\alpha, T)\frac{\omega^2(\tau_n a_T)^2}{1+\omega^2(\tau_n a_T)^2} \quad (15)$$

$$G''\{\alpha, T, \omega\} = \sum_{n=1}^{N} G^n(\alpha, T)\frac{\omega\tau_n a_T}{1+\omega^2(\tau_n a_T)^2}. \quad (16)$$

In the previous equations, ω is the radian frequency.

The axial tension-compression DMA was done and the data was treated in a similar way to obtain K^n.

C. DSC Analysis

The DSC measurements were performed in a DSC2920 Differential Scanning Calorimeter of TA Instruments. Both isothermal and dynamic heating conditions were used. For dynamic cure, the samples were heated at six different rates as 0.5, 1, 2, 5, 10, and 20 °C/min. For isothermal cure, the samples were cured under a prescribed temperature for different time, cooled quickly to −40 °C, and scanned at a heating rate of 10 °C/min to 300 °C.

Fig. 3 shows the relationship between T_g and conversion from DSC. The T_g of the uncured sample $(\alpha = 0)$ is determined by measuring the T_g of the mixed resin without adding catalyst.
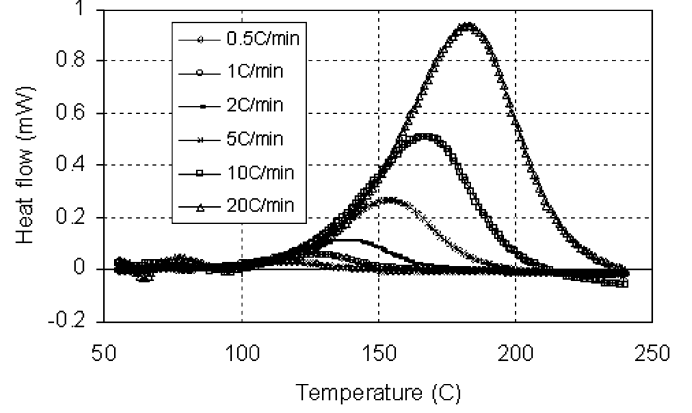

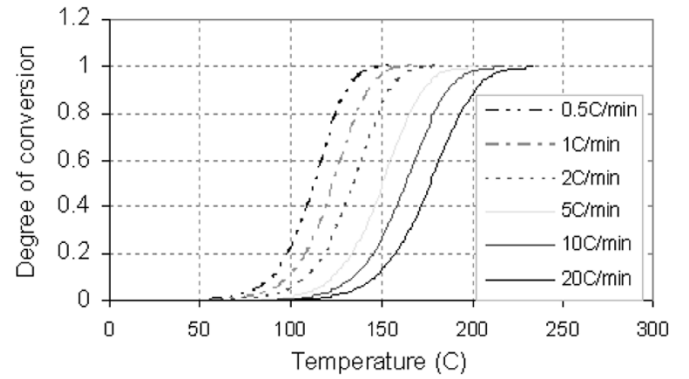Fig. 4. Dynamic DSC thermograms of the matrix epoxy from scanning with the different heating rates.

Fig. 5. Degree of conversion versus temperature.

TABLE II
KINETICS PARAMETERS OF EPOXY MATRIX

A_2 [1/s]	Q_2 [J/mol]	m	n
1.4E+07	77109	0.156	1.157

The other measured data is acquired from isothermal curing and subsequent heat scanning. The continuous line is obtained by fitting to the DeBenedetto equation [see (11)], with parameters as $T_{g0} = 12.5\,°C$, $T_{g\infty} = 120\,°C$, and $\lambda = 0.377$.

Fig. 4 shows the DSC thermograms for dynamic cure at different heating rates. The degree of conversion with temperature can be calculated from the dynamic cure and is shown in Fig. 5. The data is fitted to (12) and the kinetics parameters are listed in Table II. Fig. 6 presents the degree of conversion versus the curing time for 120 °C isothermal cure. A reasonably good agreement can be seen. It should be mentioned that the diffusion effect has not been included yet $(f(\alpha) = 1)$.

D. Curing Shrinkage Measurement

The chemical shrinkage of the polymer during the curing process was measured with an online density measuring setup based on the Archimedes principle. The uncured sample was well weighted and hold in a teflon cup. Then the cup and the sample were put into silicon oil, which has been kept at the desired temperature. The evolution of the sample's density was obtained by measuring the weight change. Fig. 7 shows the density variation for a 6-h cure at 120 °C. Fig. 8 presents the density
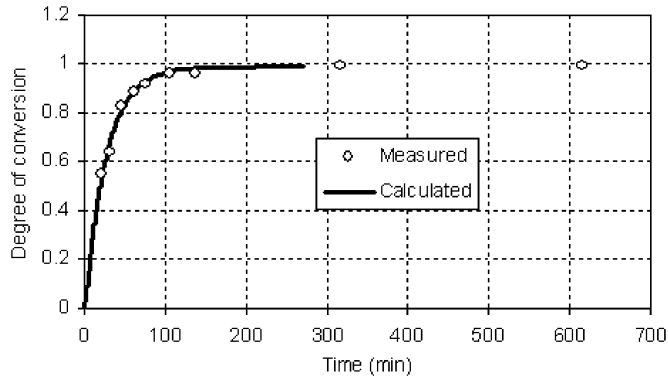
Fig. 6.   Degree of conversion versus time at 120 °C curing. The solid line is calculated from (12).
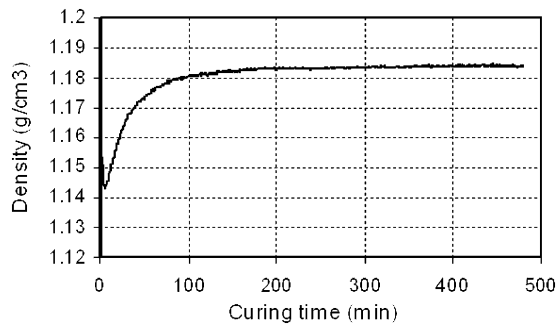

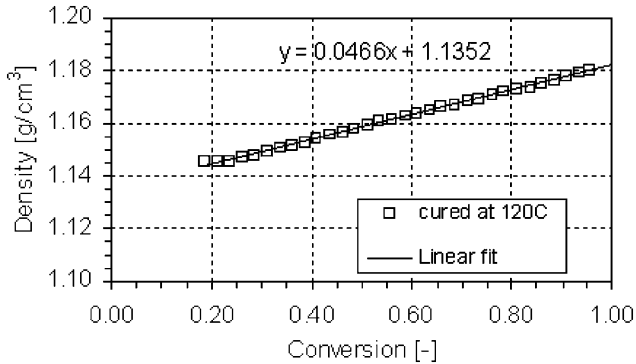Fig. 7.   Density evolution during isothermal cure at 120 °C.

Fig. 8.   Density evolution versus conversion during cure at 120 °C.

change with the conversion. It turns out to be linear and with a total volume shrinkage of 4.1%.

IV. MICROMECHANICAL MODELING

A. Implementation of the Material Model Into FE Code

The cure-dependent viscoelastic model described in Section II has been implemented in a commercial finite element code MSC Marc. In Marc, a general hereditary integral form of viscoelastic constitutive relation is already built in the program [15]. For our case, we have designed user subroutines to incorporate the cure-dependent properties into the standard program. The main functions and features of these subroutines are as follows.
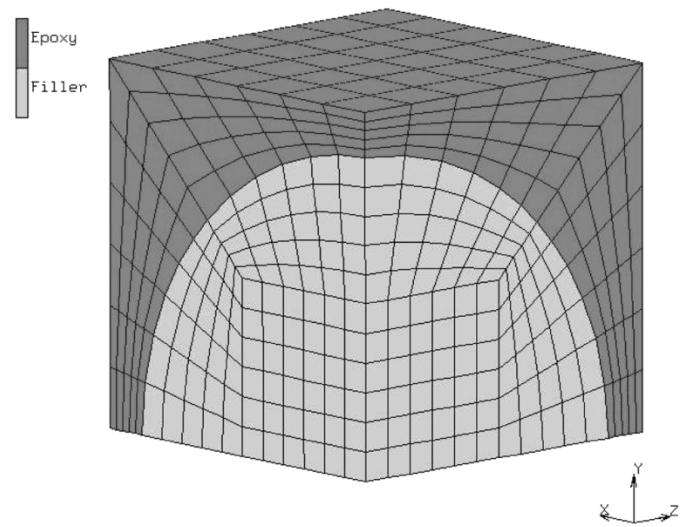

Fig. 9.   FE mesh of the micromechanical model.

1) The cure-dependent equilibrium moduli and relaxation modulus functions are calculated, and the stiffness matrices are reassembled.
2) Thermal strain and the cure shrinkage strain are introduced as initial strain at the beginning of each increment.
3) The temperature effect is realized by a time shift factor.

B. Geometrical Model and Boundary Conditions

A (3-D) FE model is built for the filled polymer. In a particle-filled composite, the actual distribution of the particle in the matrix is random. For simplicity reasons, micromechanical models usually assume a periodic arrangement of fillers, from which a representative volume element (RVE) or unit cell can be established. There are many publications which have discussed the establishment of RVE for particle-filled composites (for example [16] and [17]). In [18], an extensive investigation on how to apply correct boundary conditions for RVE was presented. In this study, we assume the spherical silica particles to be distributed in a cubic array. Due to the symmetry, only one-eighth of the cubic structure is needed for analysis. Three filler weight percentages were used to make the composite, i.e., 40%, 50%, and 65%, which correspond to filler volume fractions approximately as 27%, 35%, and 50%, respectively.

Fig. 9 shows the FE mesh of the unit cell. Perfect bonding is assumed between the interface of the filler and matrix. The silica filler is assumed to be elastic, with Young's modulus 778 GPa and Poisson ratio 0.18. The property of the matrix resin is described by the above cure-dependent viscoelastic model.

As for the boundary conditions, symmetric conditions are applied on the boundary surfaces $X = 0$, $Y = 0$, and $Z = 0$, i.e.,

$$u(0, Y, Z) = 0 \qquad (17)$$
$$v(X, 0, Z) = 0 \qquad (18)$$
$$w(X, Y, 0) = 0. \qquad (19)$$

On the surface $X = l_0$, $Y = l_0$, and $Z = l_0$ of the FE model, boundary conditions are applied by the option "Tying" in MARC, such that each of these plane surfaces remains plane during deformation, but is free to displace in its own plane.
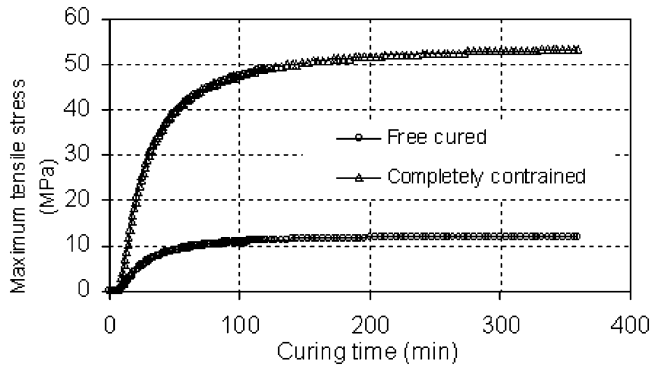
Fig. 10. Evolution of the maximum tensile stresses during cure (filler percentage: 50 wt%).
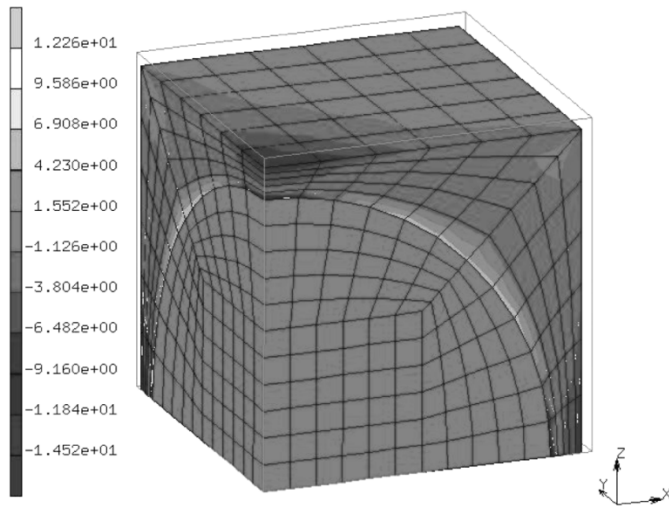

Fig. 11. Contour plot of stress distribution at the end of curing in free cured state (filler percentage: 50 wt%).

(a)

(b)

Fig. 12. Contour plot of stress at the end of curing: (a) free cure and (b) completely constrained cure. The filler percentage is 50 wt%.

In order to study the effect of external constraint on the stress buildup during the curing process, three methods are used to apply prescribed displacements in the directions x, y, and z on the corresponding surfaces $X = l_0$, $Y = l_0$, and $Z = l_0$, respectively. They are as follows.

1) Totally free. This boundary condition corresponds to the situation of being cured freely, without any or with negligible geometrical constraint on the material.
2) Completely constrained in these directions. Such a condition represents the situation of being cured under fully constrained state. This is an extreme situation.
3) The displacements calculated from macromodels are applied as their respective boundary conditions. In the macromodels, a disk shaped specimen and a QFP package with the 65% filled epoxy as encapsulant are shown as demonstrators.

C. Results and Discussion

Fig. 10 shows the evolution of the maximum tensile stress in the matrix during the whole curing process for free cure and fully constrained cure (filler percentage 50 wt%). At the end of cure, the maximum tensile stresses are 12.26 and 53.28 MPa for the free and constrained cure conditions, respectively. Note
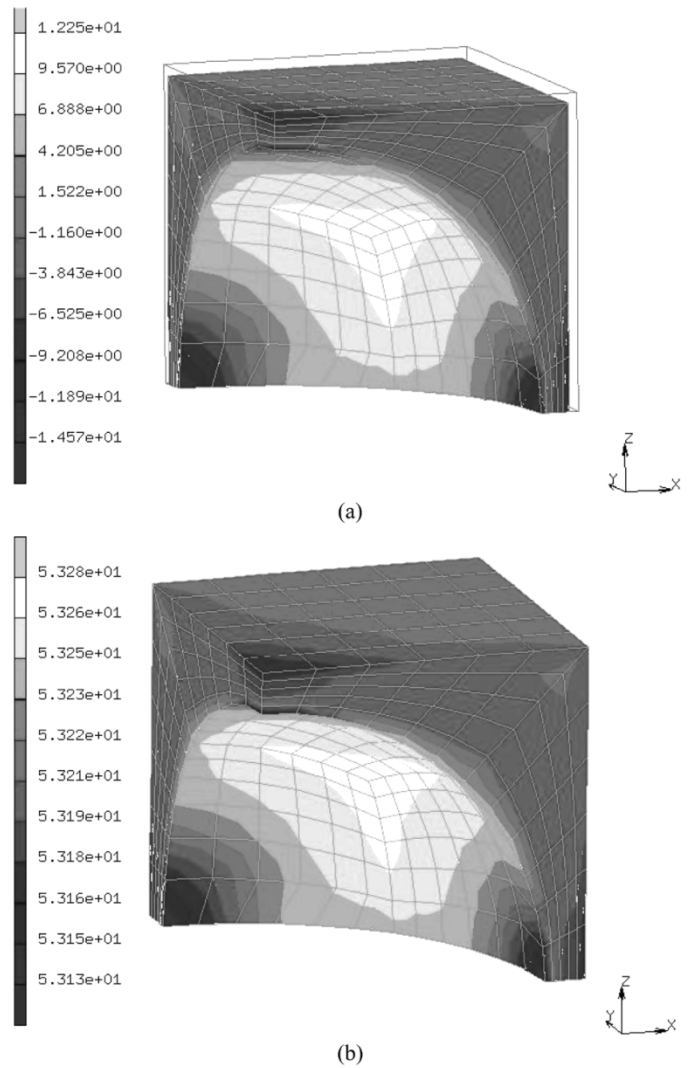
that for free cure a macromodel should give zero stress in the material. It can also be seen that the stress level strongly depends on the constraint situation by external materials.

Fig. 11 shows the stress distribution in the whole unit cell at the end of cure at a free cured state. Fig. 12(a) and (b) present the stress component σ_{xx} at the end of curing in the freely cured, and completely constrained cured states. It can be seen that micromechanical modeling can give a detailed description of the microscopic stresses and strains induced during the curing process. Further study on the effect of the filler volume fraction and external constraint state from specified electronic packages is under way.

V. CONCLUSION

As a first attempt to investigate the evolution of the curing process induced stresses in a silica particle-filled polymer, a (3-D) micromechanical FEM model has been built. In the micromechanical model, the epoxy matrix is modeled with a developed cure-dependent viscoelastic constitutive model. A combinational approach of DMA and DSC is used to characterize the cure-dependent parameters.

The experimental results show that the combination of DMA and DSC is an effective way for the characterization of the cure-dependent parameters. The simulation results indicate that the constraint situation used for the boundary condition has a strong influence on the cure-induced stress level. From the micromechanical modeling for the free cure condition, the cure-induced maximum tensile stress is about 12 Mpa, whereas a macromodel should give zero stress in the material. Micromechanical modeling can give a detailed description of the microscopic stresses and strains induced during the curing process. Further research on the effect of the filler volume fraction is needed.

ACKNOWLEDGMENT

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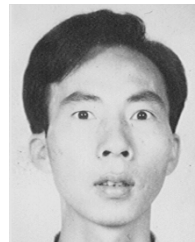
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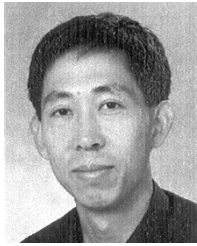
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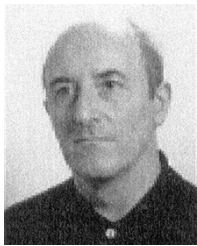
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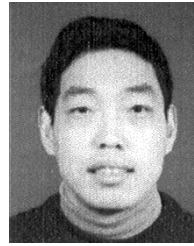
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