An Improved Methodology for Determining Temperature Dependent Moduli of Underfill Encapsulants

Yang Rao, S. H. Shi, and C. P. Wong, *Fellow, IEEE*

***Abstract*—Finite element analyses (FEAs) have been widely used to preventively predict the reliability issues of flip-chip (FC) packages. The validity of the simulation results strongly depends on the inputs of the involved material properties. For FC packages Young's modulus-temperature relationship is a critical material property in predicting of the package reliability during −55 °C to 125 °C thermal cycling. Traditional tensile tests can obtain the modulus at selected temperatures, but it is tedious, expensive, and unable to accurately predict the Young's modulus-temperature relationship within a wide temperature range. Thus, this paper is targeted to provide a simple but relatively accurate methodology to obtain the Young's modulus-temperature relationship. In this paper, three commercial silica filled underfill materials were studied. A simple specimen (based on ASTM D638M) preparation method was established using a Teflon mold. A dynamic-mechanical analyzer (DMA) was used to obtain the stress-strain relationship under controlled force mode, storage and loss modulus under multi-frequency mode, and stress relaxation under stress relaxation mode. A simple viscoelastic model was used and an empirical methodology for obtaining Young's modulus-temperature relationship was established.**

***Index Terms*—Loss modulus, modulus-temperature relationship, storage modulus, stress-strain relationship, underfill encapsulant, viscoelastic model, Young's modulus.**

I. Introduction

THE rapid development of integrated circuit (IC) fabrication technology and the accelerated growth of the market for faster, smaller, yet less expensive products continue to challenge IC packaging technology [1], [2]. As an ultimate solution for this trend, flip-chip technology on organic substrate has been invented, developed, and practiced for more than ten years [3], [4]. Current FC technology needs underfill material to extend the fatigue life of C4 (controlled collapse chip connection) interconnects. Evaluation of the underfill encapsulants has become an indispensable step in implementing the FC technology since most of the commercially available encapsulants are not specifically designed for a certain flip-chip application. Generally speaking, the sequential underfill evaluation steps include the material property characterization, selection of underfill materials with the aid of computer simulation, and in-situ reliability testing using assembled functional flip-chip packages as test vehicles. Always, there are numerous new underfills emerging in the market place every year and in-situ reliability testing is time- and cost-intensive. FEA computer simulation has become one of the crucial screening processes for the selection of new underfills. But it needs reliable characterized material property data as inputs. The characterization of underfill materials has become one of the major factors in determining a new FC product development cycle and cost. One of the challenges in the development of a flip-chip package is the package cracking, especially the die cracking. Stress-strain relationship and Young's modulus-temperature relationship of underfill encapsulants are two of the main factors for the package cracking [5], [6]. Therefore, the development of a simple and reliable approach to obtain these two critical material properties of the underfills is of practical importance. This work thus explored a simple methodology to obtain the stress-strain relationship and Young's modulus- temperature relationship by only using DMA instrument. The established methodology was mainly based on the storage modulus-temperature relationship of the materials. Because currently widely used underfill materials all have the similar storage modulus-temperature relationship; the established methodology should be applicable to all these underfill materials within the temperature range up to glass transition temperature (Tg).

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The authors are with the School of Materials Science and Engineering and Packaging Research Center, Georgia Institute of Technology, Atlanta, GA 30332-0245 USA (e-mail: gt5406c@prism.gatech.edu; cp.wong@mse.gatech.edu).

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II. Theoretical Background

There are three categories of materials: elastic, viscous and viscoelastic materials. Underfill materials are typical viscoelastic polymeric materials. Fig. 1 shows the stress responses under constant strain loading of pure elastic and viscoelastic materials, respectively [7]. Generally, the stress response for viscoelastic polymeric material is a function of time while that for elastic material is a constant, i.e. time-independent.

One important property used to describe the viscoelastic polymeric materials is relaxation modulus $E(t)$. The relaxation modulus $E(t)$ is defined as the ratio of stress and strain [7], i.e.,

$$E(t) = \frac{\sigma(t)}{\varepsilon_0}. \tag{1}$$

If an underfill is assumed to be linear viscoelastic, the linear superposition law can be applied to the underfill materials. The superposition law states that the entire history of the stress can be reconstructed by adding the stress histories that correspond

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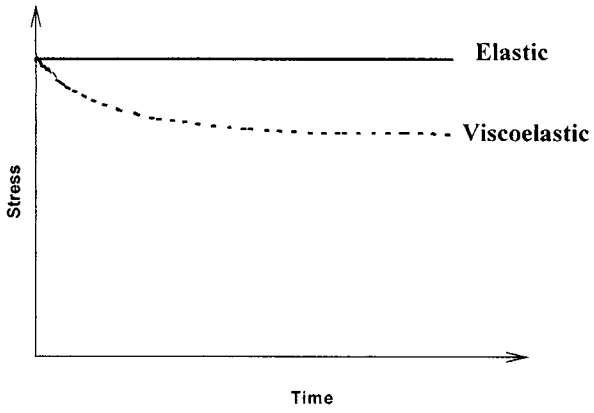


Fig. 1. Stress response of elastic and viscoelastic material under constant strain.

to individual bits of a strain history, i.e., the stress response is the convolution integrals of strain history, as shown in (2) [7].

$$\sigma(t) = \int_{-\infty}^{t} E(t-\tau)\frac{d\varepsilon}{d\tau}\,d\tau \tag{2}$$

where $E(t-\tau)$ is the relaxation modulus at time τ. When strain is chosen to be in the form of $\varepsilon(t) = \varepsilon_0 \sin(\omega t)$, where ω is the circular frequency in radians per second. The static stress can be obtained from the (2). The ratio of stress over strain is defined as complex modulus $E^*(\omega)$. From (2), it can be found that $E^*(\omega)$ is the Fourier transform of relaxation modulus $E(t)$ [7]:

$$E^*(\omega) = i\omega \cdot \int_0^{\infty} E(t)e^{-iwt}\,dt. \tag{3}$$

Therefore, as long as either relaxation modulus or complex modulus is known, other mechanical properties can be calculated through (3). Storage modulus and loss modulus is defined as the real and imaginary part of complex modulus, respectively.

Another consideration is that the mechanical properties of underfill materials are highly temperature dependent. Theoretically, if Young's modulus or complex dynamic modulus of a glassy polymeric material is known at one temperature, these moduli at any temperature can be estimated using time-temperature superposition shift factor, which can be calculated from the Williams–Landel–Ferry (WLF) equation with the measured glass transition temperature (Tg) value [8]. However, the accuracy of such kind of estimation is a real concern since some basic assumptions may be invalid when the time-temperature superposition principle is applied to the complicated silica filled cross-linked thermosetting underfill materials. Therefore, an alternative solution is needed for real-life application. Our idea is to establish a relationship between Young's modulus and complex dynamic modulus within the temperature range up to Tg based on a modified Maxwell model [see (4)]. Since the complex modulus in a wide temperature range can be conveniently obtained by simply running a specimen on a DMA instrument, the Young's modulus in the same temperature range can be easily obtained by just converting the complex modulus from the established relationship.

III. Simulation Model

Maxwell model gives a reasonable prediction of relaxation behavior of polymers in molten state [7]

$$E(t) = ke^{-nt}. \tag{4}$$

Combining (4) and (3), we can get complex modulus as [7]

$$E^*(\omega) = \frac{k\omega^2}{n^2+\omega^2} + i\frac{kn\omega}{n^2+\omega^2}. \tag{5}$$

From (5), we can calculate strength of relaxation k and characteristic time n from the storage modulus and loss modulus at different temperatures. If Maxwell model is valid for underfill material, we can use parameter k to represent the Young's modulus of underfill material. However, Maxwell model is too simple to satisfactorily describe the physical feature of an underfill material. Thus, for underfill materials, k can not be simply considered as the Young's modulus. On another hand, k represents elastic property of material because it is the initial relaxation modulus. It is reasonable to introduce a fitting factor $f(T)$ that correlate Young's modulus E and k. In another word, we are trying to find a function of temperature for underfill materials that can connect k and Young's modulus

$$E = f(T) \cdot k \tag{6}$$

$f(T)$ was chosen in the form of

$$f(T) = f_0 + f_1 \cdot (1 - e^{f_2 \cdot T}) \tag{7}$$

where f_0, f_1 and f_2 are constants. Now, if f_0, f_1, and f_2 can be identified, the Young's modulus at any temperature can be quickly calculated using (6). The reason that we chose such a fitting factor lies in the fact that (7) can universally describe the storage modulus-temperature relationship with very good accuracy. The three constants f_0, f_1, and f_2, are then derived from Young's modulus at three different temperatures using fitting theory. From above assumption, we think this method can be used to predict temperature dependent Young's Moduli of other linear viscoelastic materials, no only underfill materials. This point needs to be proved through more research.

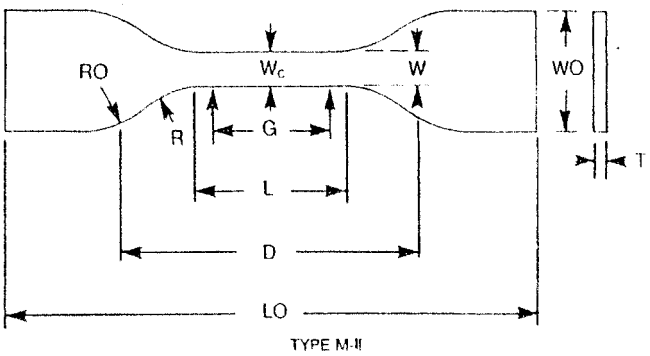
IV. Experiments

A. Materials

Three commercial underfill materials A, B, and C were supplied by Dexter Hysol Co. of California. These materials were stored in a freezer at $-40\,^{\circ}$C. Before these materials were used to prepare the specimens, they were taken out of the freezer and warmed at room temperature for 1 h.

B. Specimens Preparation

Different specimens were prepared to fit different tests. For dynamic tests, the specimen preparation procedures are as follows: About 4 g of warmed underfill material was placed into an aluminum pan (37.5 mm diameter), then the pan was transferred to an 80 ^{o}C preheated connective oven, and then the material was cured using the curing recipe given by Dexter. After curing, the material was removed from the oven and cooled down to



TYPE M-II

W-Width of narrow section	2.5 mm	G-Gage length	7.5 mm
L-Length of narrow section	10 mm	D-Distance between grips	25 mm
WO-Width of overall, min.	10 mm	R-Radius of fillet	15 mm
LO-Length overall, min.	50 mm	RO-Outer radius (Type III)	---

Fig. 2. Shape and dimensions of the prepared specimens in this study.

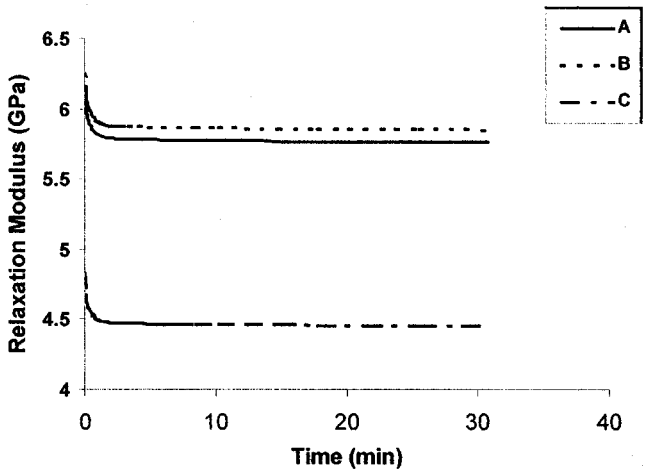


Fig. 3. Relaxation moduli of underfill material A, B, and C at 30 ^{o}C.

room temperature. The aluminum pan was then peeled off and the cured sample was polished into a disk with two parallel surfaces. Finally, a diamond saw was used to cut the cured sample into strips with dimensions of about $32 \times 11 \times 3$ mm.

For tensile tests and stress-relaxation tests, the specimens were prepared by dispensing the warmed underfill material into the cavity of a Teflon mold that had been pre-heated to $\sim 70^{o}$C for better underfill flow. The Teflon mold was then transferred to a convective oven and the underfill material was cured following the curing condition given by Dexter Company. The specimens, which comply with ASTM D638M standard, were then removed from the Teflon mold. The shape and dimensions of the prepared specimens are shown in Fig. 2. The thickness of the specimens is ~ 0.5 mm.

C. Mechanical Testing

Tensile tests, dynamic tests, and stress relaxation tests were performed on a Dynamic Mechanical Analyzer (DMA model 2980 of *TA Instruments*) instrument, which is equipped with an environmental chamber to precisely control temperature changes (-145^{o}C–600 ^{o}C, 0–50 ^{o}C/min). Fig. 4 shows the film tension clamp of DMA. Using the film tension clamp, we can conduct tensile and stress relaxation tests. Dynamic tests were performed under single cantilever mode using 1 Hz sinusoidal strain loading (strain amplitude $= 0.2\%$), which gives the storage modulus and loss modulus of the material in the temperature range from -60^{o}C to 250 ^{o}C. Tensile tests were performed under DMA *control force mode* that gives the stress-strain relationship at a particular loading rate and temperature. Stress relaxation tests were performed under *stress relaxation mode* that gives the stress response versus time under a constant strain as shown in Fig. 3. Prior to the testing, all specimens were annealed at the temperature near their Tg's for 1 h to release residual stresses introduced during the specimen preparation. Dog-bone specimens have the advantage of reducing grip effect that may cause specimen early failure due to the stress concentration. In the tensile testing, only a few specimens failed at the location close to the grips. The failure of most specimens occurred at locations far away from the grips, indicating that it was reasonable to use this type of specimen. DMA can record the deformation between two grips and calculation strain. The actual length between two grips is in the range of 10–15 mm. The loading rate of tensile tests is 0.1 N/min. The tensile tests were performed at seven different temperatures: -30 ^{o}C, 30 ^{o}C, 40 ^{o}C, 60 ^{o}C, 70 ^{o}C, 90 ^{o}C, 120 ^{o}C.

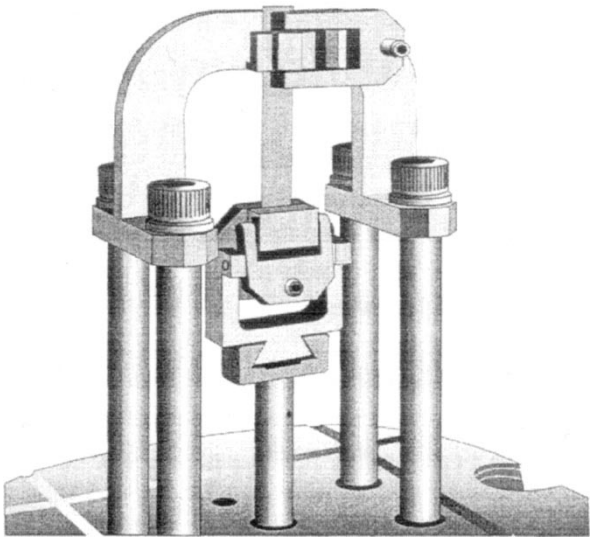


Fig. 4. Film tension clamp of DMA.

V. Results and Discussion

Figs. 5 and 6 show the storage moduli and loss moduli of the three underfill material A, B, and C. From the figures, we can know that the glass transition temperature (Tg) of these three materials are around 150 ^{o}C.

As shown in (5), the parameter k for the three underfill materials can be calculated using tested storage moduli and loss moduli. Fig. 7 shows the calculated k for these materials in the temperature range between -30 ^{o}C to 120 ^{o}C. At 30 ^{o}C,

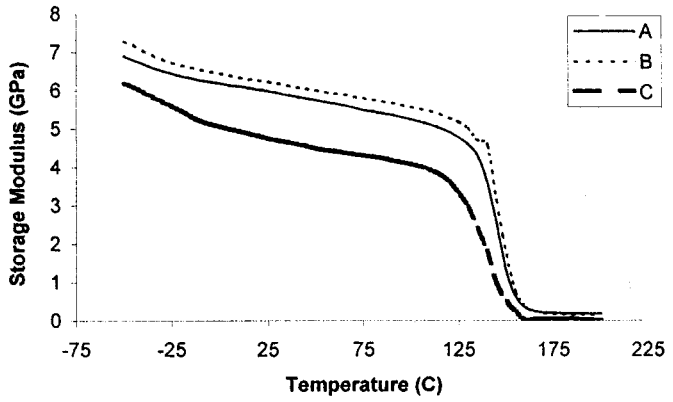


Fig. 5. Storage moduli of three underfill materials A, B, and C.

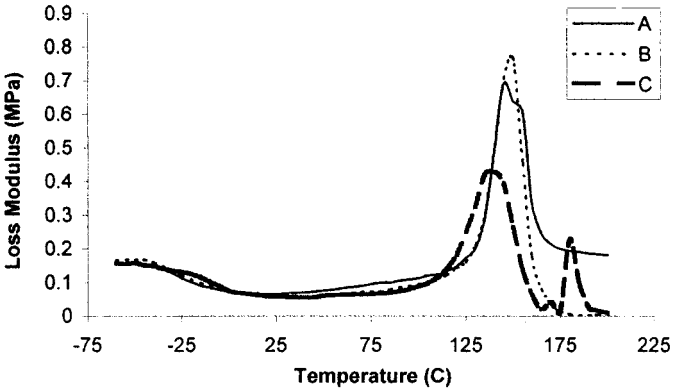


Fig. 6. Loss moduli of three underfill materials A, B, and C.

k is equal to 5.96 GPa, 6.2 GPa, and 4.72 GPa for the underfill materials A, B, and C, respectively. The stress relaxation result (Fig. 2) shows that the initial relaxation moduli of underfill materials A, B, and C are 6.11 GPa, 6.35 GPa, and 4.83 GPa, respectively. The comparison between the calculated k and measured initial relaxation modulus finds that the two values comply with each other very well for all the three underfill materials. This finding gives a strong support to the verification of our simulation model [see (4)]. In order to obtain the function $f(T)$ that correlates k and Young's modulus, static tensile tests were conducted at three different temperatures: -30^{o}C, 60 ^{o}C and 120 ^{o}C. Figs. 8–10 show the stress-strain curves at three different temperatures for underfill materials A, B, and C, respectively. The Young's moduli are obtained as the average slope of stress-strain curves in low strain range (strain $< 0.1\%$). DMA can only conduct force control tensile testing and loading rate is 0.1 N/min. Table I lists the Young's moduli at different temperatures for the three underfill materials. From the measured modulus and calculated k values of each material, the parameter f_0, f_1 and f_2 for this material can then be calculated using (6) and (7). The calculated f_0, f_1 and f_2 of each material were tabulated in Table II and the $F(T)$ function was shown in Fig. 11. Thus, f_0, f_1 and f_2 can be used to estimate Young's modulus at any temperatures by multiplying $f(T)$ and k values using (6) and (7). This actually is the ultimate purpose of this work.

Figs. 12–14 compare the simulated Young's moduli and tested Young's modulus of underfill material A, B, and C (at temperature 30 ^{o}C, 40 ^{o}C, 70 ^{o}C and 90 ^{o}C), respectively. From

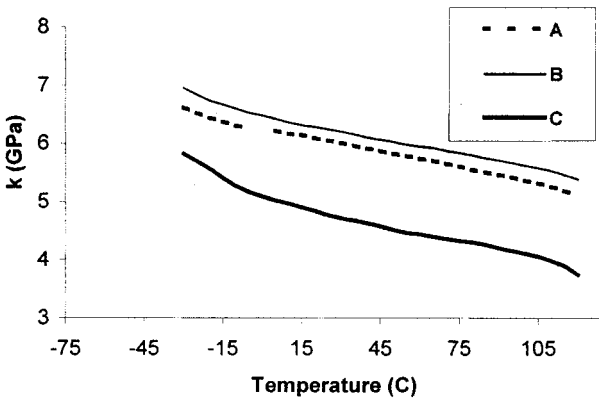


Fig. 7. Calculated parameter k of the three underfill materials A, B, and C.

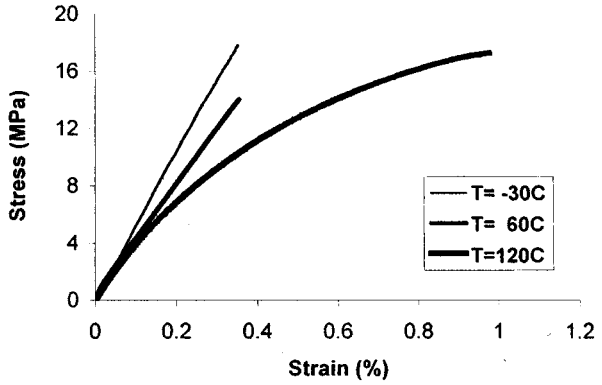


Fig. 8. Stress-strain curves of underfill material A at -30 ^{o}C, 60 ^{o}C, and 120 ^{o}C.

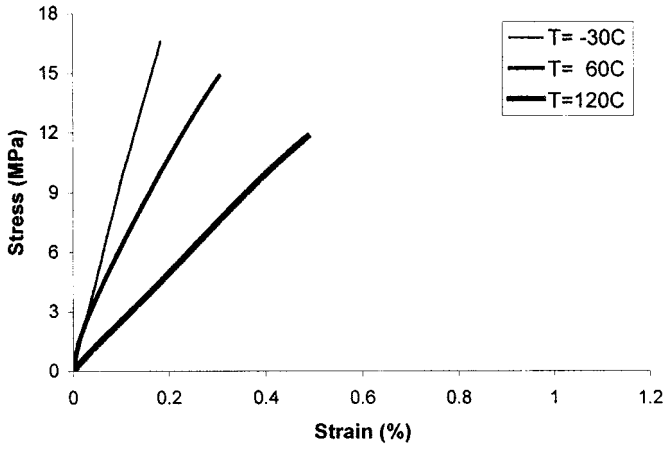


Fig. 9. Stress-strain curves of underfill material B at -30 ^{o}C, 60 ^{o}C, and 120 ^{o}C.

those curves, we can see the tested Young's moduli comply with simulation curves very well. Table III presents the error R of simulated values compared to tested data using (8)

$$R = \frac{E_s - E_t}{E_t} \times 100\% \tag{8}$$

where: E_s is the simulation value of Young's modulus, while E_t is the tested value.

Table III shows that the errors of simulated Young's moduli of all three underfill materials are less than 10%, which satisfies the requirement of the industry. Thus, we find a practical approach

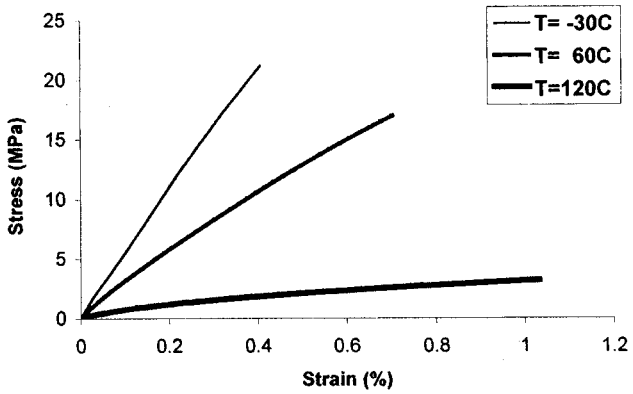


Fig. 10. Stress-strain curves of underfill material C at $-30\ ^{o}$C, 60 ^{o}C, and 120 ^{o}C.

TABLE I
YOUNG'S MODULI OF THREE UNDERFILL MATERIALS OBTAINED BY TENSILE TESTS

	E_A(GPa)	E_B(GPa)	E_C(GPa)
T= -30 ^{0}C	5.049	8.48	5.215
T= 30 ^{0}C	4.917	7.644	3.698
T= 40 ^{0}C	4.356	6.702	3.625
T= 60 ^{0}C	3.963	5.588	3.137
T= 70 ^{0}C	3.724	5.172	2.941
T= 90 ^{0}C	2.639	4.115	2.547
T=120^{0}C	1.431	2.459	1.671

TABLE II
PARAMETERS F_0, F_1 AND F_2 OF THE THREE UNDERFILL MATERIALS

	f_0	f_1	f_2
A	0.8455	0.0982	0.01604
B	1.2186	0.2434	0.01214
C	0.8414	0.1704	0.00963

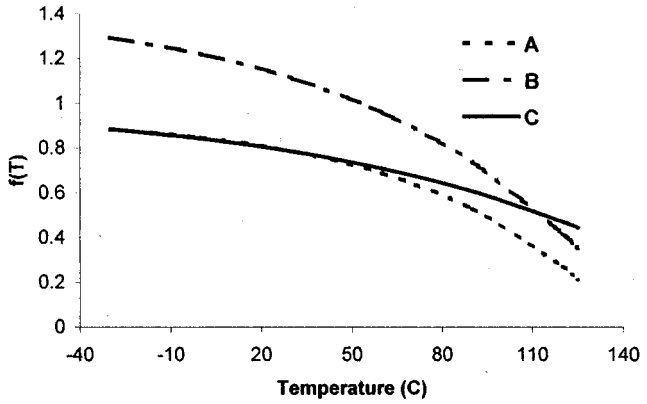


Fig. 11. Fitting function $f(T)$ of underfill material A, B, and C.

that can estimate Young's modulus of underfill materials in certain temperature range (below Tg). The entire methodology includes the following steps.

1) Use DMA to measure storage modulus and loss modulus of a material in desired temperature range.

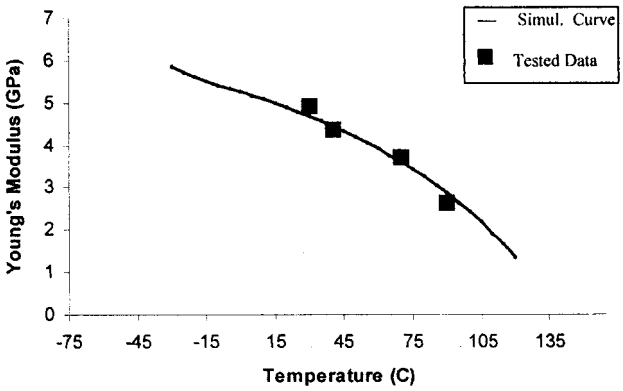


Fig. 12. Simulated Young's modulus curve and experimental Young's modulus of underfill material A.

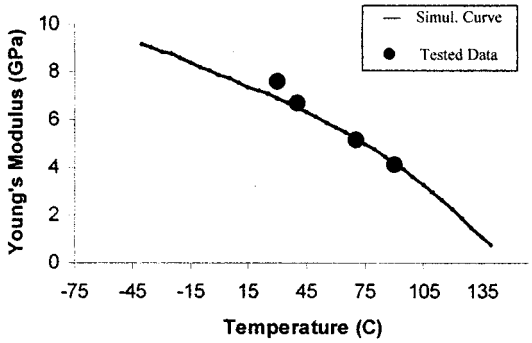


Fig. 13. Simulated Young's modulus curve and experimental Young's modulus of underfill material B.

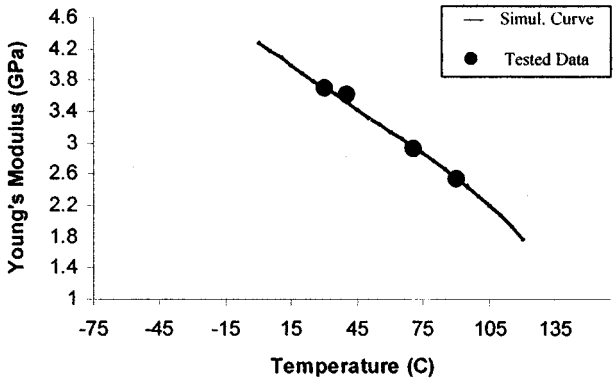


Fig. 14. Simulated Young's modulus curve and experimental Young's modulus of underfill material C.

TABLE III
ERROR OF YOUNG'S MODULUS SIMULATION OF UNDERFILL MATERIAL A, B, AND C

	R_A(%)	R_B(%)	R_C(%)
T= 30 ^{0}C	-4.92	-9.8	0.01
T= 40 ^{0}C	1.9	-2.94	2.89
T= 70 ^{0}C	-3.39	1.33	0.39
T= 90 ^{0}C	8.37	2.18	0.01

2) Use (7) to calculate parameter k.
3) Conduct static tensile testing at three different temperatures, which can properly cover the interested temperature range.
4) Use the data we get in 2) and 3) to obtain the parameter of fitting function.
5) Use (6) to calculate simulation value of Young's modulus.

VI. Conclusion

The mechanical properties of underfill materials are always the key concern of FC technology. Obviously, we need to study the detailed relationship between the molecular structure and the properties if we want to predict materials properties properly. The work shown here is just a valuable attempt to predict materials' static mechanical properties through dynamic properties. It does provide a relative simple and valuable approach that forecasts Young's modulus within the interested temperature range with error less than 10%.

References

[1] R. Marrs, "Trends in IC packaging," *Electron. Packag. Prod.*, pp. 24–30, Aug. 1996.
[2] R. R. Tummala, E. J. Rymaszewski, and A. Klopfenstein, *Microelectronics Packaging Handbook, Part II*, 2nd ed. New York: Chapman & Hall, 1997.
[3] F. Nakano, T. Soga, and S. Amagi, "Resin-insertion effect on thermal cycle resistivity of flip-chip mounted LSI devices," in *Proc. Int. Soc. Hybrid Microelectron. Conf.'87*, 1987, p. 536.
[4] D. Suryanarayana, R. Hsiao, T. P. Gall, and J. M. McCreary, "Flip-chip solder bump fatigue life enhanced by polymer encapsulation," in *Proc. 40th IEEE Electron. Comp. Technol. Conf.'90*, 1990, p. 338.
[5] V. Sarihan, Y. Guo, T. Lee, and S. Teng, "Impacting electronic package design by validated simulations," in *Proc. 48th IEEE Electron. Comp. Technol. Conf.'98*, 1998, p. 330.
[6] S. E. Lindsey, J. Aday, B. Blood, Y. Guo, B. Hemann, and J. Kellar, "JACS-Pak™ flip-chip chip scale package development and characterization," in *Proc. 48th IEEE Electron. Comp. Technol. Conf.'98*, 1998, p. 511.
[7] S. Matsuoka, *Relaxation Phenomena in Polymers*. New York: Hanser, 1992.
[8] I. M. Ward, *Mechanical Properties of Solid Polymers*, 2nd ed. New York: Wiley, 1983.

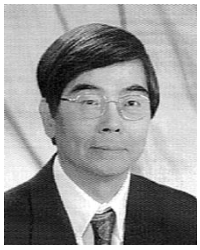
Yang Rao received the B.S. and M.S. degrees in solid mechanics from the Mechanics Department, Peking University, Beijing, China, in 1992 and 1995, respectively, the M.S. degree in material science from the School of Materials Science and Engineering, Georgia Institute of Technology, Atlanta, in 1998, where he is currently pursuing the Ph.D. degree.

His research interests include modeling of embedded capacitor, development of high dielectric constant polymer-ceramic composite, and modeling of effective properties of composite materials.

S. H. Shi (M'94) received the B.S. degree from Tsinghua University, Beijing, China, in 1991, the M.S. degree from Fudan University, Shanghai, China, in 1994, and is pursuing the Ph.D. degree in the School of Materials Science and Engineering, Georgia Institute of Technology (Georgia Tech), Atlanta.

He worked at Motorola, Inc., Atlanta, GA, on chip scale packaging during the summer time of 1997 and 1998.

Mr. Shi shared the best paper award of the 47th ECTC meeting, received Motorola Scholarship for excellent contribution in 1997, was elected a National Science Foundation Fellow of the Packaging Research Center in Georgia Tech in 1997, and is a member of IMAPS and SPE.



C. P. Wong (SM'87–F'92) received the B.S. degree in chemistry from Purdue University, West Lafayette, IN, and the Ph.D. degree in organic/inorganic chemistry from Pennsylvania State University, University Park.

He spent 19 years with AT&T Bell Labs and was elected a Bell Labs Fellow in 1992. He is a Professor with the School of Materials Science and Engineering and a Research Director with the Packaging Research Center, Georgia Institute of Technology, Atlanta. He holds over 45 U.S. patents, numerous international patents, and over 240 technical papers in the related area. His research interests lie in the fields of polymerica materials, reaction mechanism, IC encapsulation, hermetic equivalent plastic packaging, electronic packaging processes, interfacial adhesions, PWB, SMT assembly, and component reliability.

Dr. Wong received the AT&T Bell Laboratories Distinguished Technical Staff Award in 1987, the AT&T Bell Labs Fellow Award in 1992, the IEEE Components Packaging and Manufacturing Technology (CPMT) Society Outstanding and Best Paper Awards in 1990, 1991, 1994, 1996, and 1998, the IEEE Technical Activities Board Distinguished Award in 1994, the 1995 IEEE CPMT Society's Outstanding Sustained Technical Contribution Award, the 1999 Georgia Tech's Outstanding Faculty of the Year Award, the Georgia Tech Sigma Xi Faculty Best Research Paper Award in 2000, and the University Press (London, U.K.) Award of Excellence in 2000. He is a member of the National Academy of Engineering, a Fellow of AT&T Bell Labs, and was the General Chairman of the 41st Electronic Components and Technology Conference in 1991. He served as Technical Vice President (1990 and 1991) and President (1992 and 1993) of the IEEE CPMT Society. He currently chairs the IEEE/IMAPS International Symposium on Advanced Packaging Materials in 1999 and 2000.