Enhancement of Antifillet Cracking Performance for No-Flow Underfill by Toughening Method

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ABSTRACT: Fillet cracking of no-flow underfill in a flip-chip device during a reliability test such as thermal shock or thermal cycling has been a serious reliability problem. The effect of toughening agents and modification of epoxy on fillet cracking of no-flow underfill was investigated. The best epoxy formulation and the appropriate loading level of toughening agent regarding the antifillet cracking performance were found. In the case where the epoxy was modified with polysiloxanes, the second-phase particle with a submicron particle size was formed and the size of the particle depended on the kind of toughening agent. The morphology was observed by a scanning electron microscopy and confirmed by a dynamic mechanical analyzer measurement. The physical properties such as the fracture toughness, flexural modulus, coefficient of thermal expansion, and adhesion were measured, and the liquid–liquid thermal shock (LLTS) test under −55 to 125°C was performed with different formulations. One of the formulations toughened by amine/epoxy-terminated polysiloxane, which has higher die shear strength, lower modulus, and higher toughness, passed 1000 cycles of the LLTS test. In order to obtain a high reliable no-flow underfill, the physical properties of the no-flow underfill should be well controlled and balanced. Finally, a correlation between physical properties of the no-flow underfill and the fillet cracking capability for those approaches was discussed. © 2003 Wiley Periodicals, Inc. J Appl Polym Sci 88: 2439–2449, 2003

INTRODUCTION

The demand for smaller, thinner, lighter-weight, higher performance, and cost-effective electronic products has driven the advanced electronic packaging to a new level. Flip-chip technology, as the one of the solutions to those requirements, has been developed and applied for more than three decades.[1–3] However, current flip-chip technology process requires flux dispensing, soldering reflow, flux cleaning, underfill dispensing, capillary flow, and off-line underfill curing process, respectively, as shown in Figure 1. To reduce the process steps and costs of the conventional flip-chip technology, a promising underfill technology for the next generation flip-chip application, the so-called no-flow underfill technology, has been invented and developed as illustrated in Figure 2.[3] The first-generation of no-flow underfill formulation was developed in our group.[3] However, the major technical obstacle in applying the no-flow underfill technology is the resulting limited package reliability performance,[3,4]

especially, fillet cracking of the no-flow underfill during a reliability test such as thermal shock or thermal cycling has been a serious reliability problem. The fillet cracking starts from a specific region of the underfill where there is delamination between the integrated circuit (IC) chip and underfill or between the underfill and the substrate. When the stress concentrated on the delaminated region is higher than the toughness of underfill, the crack on the underfill starts and propagates, leading to the underfill fillet cracking. Therefore, the fracture toughness of underfill is one of the crucial factors that affect fillet cracking. Figure 3 shows the typical fillet cracking of underfill in flip-chip device.

Considerable effort has been dedicated to improve the toughness of cured epoxy.[5–10] Many kinds of toughening methods and chemical agents have been suggested and tested to render the epoxy more flexible, while such methods should not degrade the epoxy performance.[11] However, toughness of epoxy depends on various parameters, such as the molecular weight of either epoxy or toughening agent, interfacial adhesion force between epoxy and toughening agent, and whether the second phase of toughening agent is formed or not. In addition, it will depend on whether the toughening agent could be helpful at a specific environmental condition.[12] As such, it is difficult to choose the appropriate toughening agent for a specific polymer matrix. The
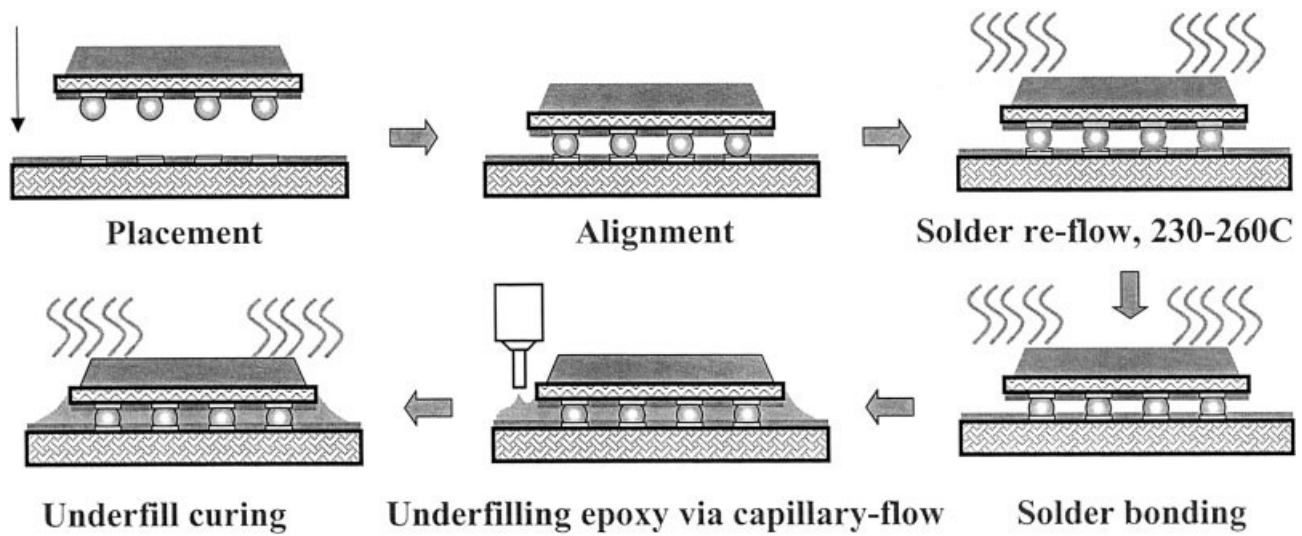
Figure 1 Schematic illustration of a conventional underfilling process for flip-chip bonding.

effect of a number of toughening agents on the toughness of epoxy has been reported.[5–10] Especially, thermoplastic polymer, rubber modifier, inorganic particles, etc., have been suggested as toughening agents to increase the epoxy toughness. Incorporation of inorganic particles such as silica seems to be attractive since it would provide a low coefficient of thermal expansion, which is extremely important for multicomponent configuration of electronics assembly to reduce thermal stress, as well as high modulus and toughness. However, incorporating an inorganic filler such as silica into the no-flow formulation causes interconnection problem.[13] During the chip bonding process, the silica particles remaining on the substrate pad could impede the contact between a solder ball and the substrate pad surface, resulting in no interconnects. Therefore, incorporating silica fillers to the no-flow underfill formulation is usually difficult. Among the various toughening agents, polysiloxane could give

considerably high thermal stability since its glass transition temperature is much lower as compared to other toughening agents.[14,15] Using polysiloxane is also attractive since it has low moisture uptake, which is desirable for microelectronic application.

In this study, first, the base epoxy resin formulation that has good antifillet cracking performance was investigated. Second, polysiloxanes with different reactive functional groups were employed to enhance the toughness of base epoxy formulation that was selected by first screening. In order to investigate the effect of toughening agents on the reliability enhancement of the no-flow underfill under harsh condition, the base epoxy resin was modified with polysiloxanes, and their thermal and physical properties of these modified formulations were measured and analyzed. Correlation of fillet cracking of no-flow underfills and their physical properties were discussed and presented through the measurement of morphological, mechanical, and thermal properties.
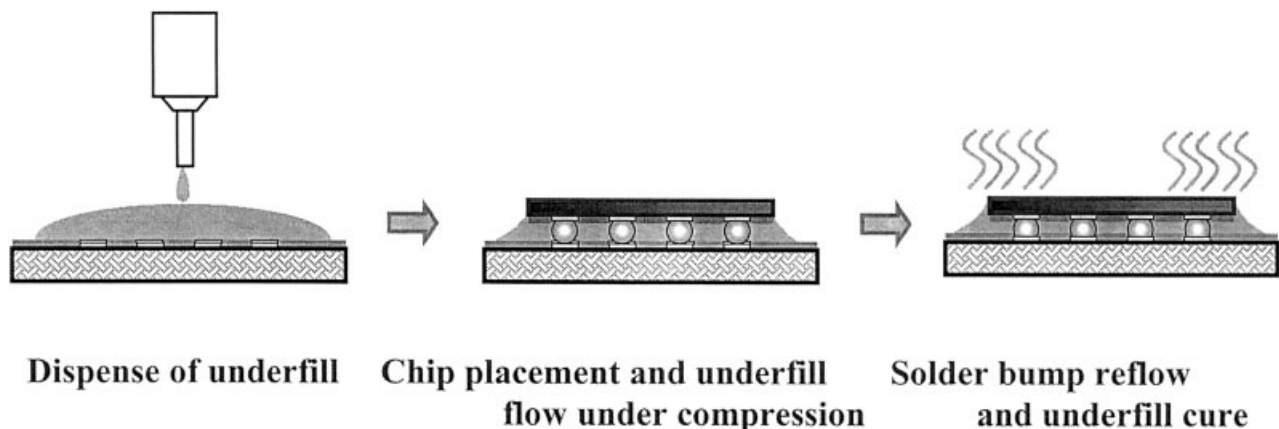
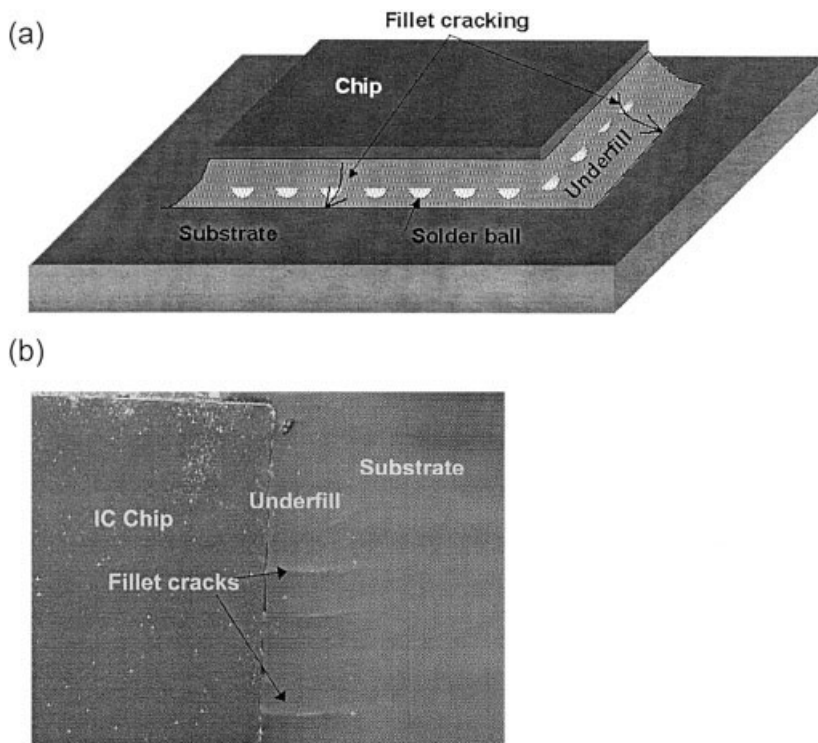**Figure 2** Schematic illustration of a no-flow underfilling process.

Figure 3 Typical fillet cracking of underfill in a flip-chip device: (a) side view and (b) top view.

EXPERIMENTAL

Materials

Bisphenol-A, bisphenol-F, cycloaliphatic type, and their mixture were used as base epoxy resin. Methyl hexahydrophthalic anhydride (MHHPA), cobalt (II) acetylacetonate (Aldrich Chem. Co.), 3-glycidoxypro-plytrimethoxysilane (A187J), and glycerol were also used as the curing agent, catalyst, adhesion promoter, and fluxing agent, respectively. Polysiloxanes with different functional groups were employed as toughening agents. Functional end group and epoxy/amine equivalent weight values of polysiloxane toughening agents used in this study are shown in Table 1.

Measurement of materials properties

Curing profile, coefficient of thermal expansion (CTA) glass transition temperature (T_g), storage and loss mod-

TABLE I
Functional End Group and Equivalent Weight Values of Polysiloxanes Used in this Study

Siloxane	Reactive group	Equivalent weight to epoxy or amine
X01	Amine	1800
X02	Epoxy	9000
X03	Epoxy	3000
X04	Epoxy	4000
X05	Amine	600

uli (G' and G'', respectively), and adhesion strength were measured using a differential scanning calorimeter (DSC) (TA Instruments model 2970, New Castle, DE), thermomechanical analyzer (TMA) (TA Instruments model 2940), dynamic mechanical analyzer (DMA) (TA Instruments model 2980), and die shear tester (Royce Instrument system 552 100K, Napa, CA), respectively. For the die shear test, the configuration of the test vehicle was made so that adhesion between SiN passivation of a silicon die and the underfill should be measured. The configuration of die shear test is illustrated in Figure 4. Fracture toughness was determined by measuring the stress intensity factor (K_{Ic}) and strain release energy (G_{IC}). The measurement of stress intensity factor was conducted using single edge notch bending (SENB) (ASTM-E24) specimens on a Universal Test Machine (MicroTester 5548 Instron Co.) as shown in Figure 5. K_{Ic} is given by

$$K_I = \frac{SP_c}{BW^{3/2}} \, f\!\left(\frac{a}{W}\right) \qquad (1)$$

where

$$f\!\left(\frac{a}{W}\right) = \frac{3\left(\frac{a}{W}\right)^{1/2}\left[1.99 - \frac{a}{W}\left(1 - \frac{a}{W}\right)\right.}{\left. \times \left(2.15 - 3.93\frac{a}{W} + 2.7\left(\frac{a}{W}\right)^2\right)\right]}{2\left(1 + 2\frac{a}{W}\right)\left(1 - \frac{a}{W}\right)^{3/2}} \qquad (2)$$
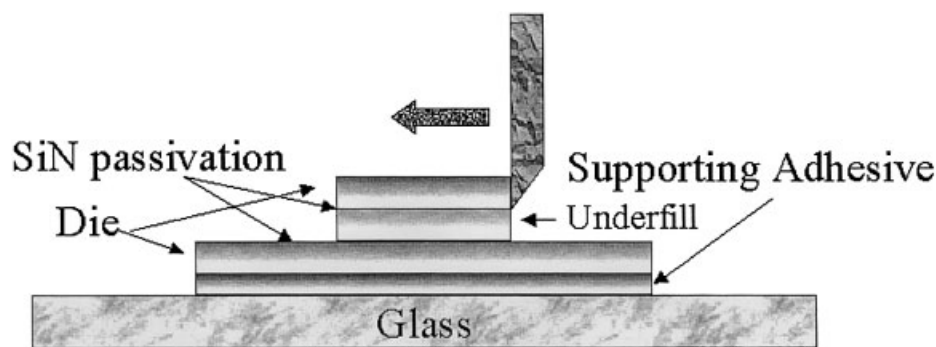
Figure 4 Schematic diagram of a die shear test.

B, W, S, P_c, and a are the width, thickness, span of the specimen, load at break, and total length of initial crack (machined V-notch + sharp notch). Establishing valid fracture toughness ($K_{IC} = K_I$),[16] the experimental validation procedures are followed to determine if they would meet all requirements such as stress condition and they are consistent with specimen size. Strain release rate (G_{IC}) is also given by

$$G_{IC} = K_{IC}^2/E \qquad (3)$$

where E is Young's modulus. The morphology of the formulations was observed by using a scanning electron microscopy (SEM) (Hitachi model S-800).

Reliability test

Silicon bare die with the size of 10 × 10 mm and the FR-4 board were used for the liquid–liquid thermal shock (LLTS) test. The surface of the silicon die was passivated with SiN. The FR-4 board was coated with the solder mask (from Taiyo Co.) with a thickness of 61.5 μm. In order to render a typical standoff between the flip-chip and the substrate as that of flip-chip assembly, the glass beads of 70 μm were added to the formulations by less than 0.5 wt % as a spacer. The LLTS test was carried out from −55 to 125°C by thermal cycling, with dwelling time of 5 min at each stage.
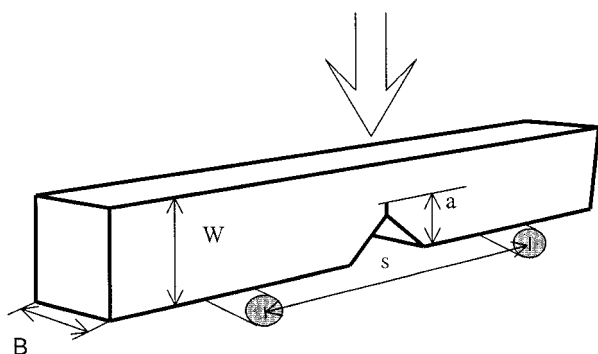

Figure 5 Schematic diagram of a single edge notch beam (SENB) specimen.

RESULTS AND DISCUSSION

Selection of base epoxy formulation and amount of modifier

In order to optimize the base epoxy formulation for no-flow underfill in terms of fillet cracking, three different epoxy combinations were used for our first evaluation. A mixture of bisphenol-A and bisphenol-F epoxy is designated as M1, that of bisphenol A and cycloaliphatic as M2, and cycloaliphatic itself as M3. The composition of the mixture of two epoxies was 50:50 by weight (M1 and M2). Table II shows the physical properties and LLTS results of three different epoxy formulations. The M3 formulation has the appropriate curing profile and fluxing capability for no-flow application, which has been developed in our group.[2,3] It has higher T_g and coefficient of thermal expansion (CTE) value compared with other formulations. However, the drawback of this formulation is low package reliability, as shown in the reliability data of Table II. For M3, fillet cracking was found just after 100 cycles. While M1 shows a bit better reliability than M3, M2 shows the best performance for LLTS test.

The above three formulations were modified with an epoxy-terminated polysiloxane varying the content of the polysiloxane in order to study the optimized content of the toughening agent. Figure 6 shows SEM photographs of the fracture surface of the M2 formulation modified with polysiloxane, where the polysiloxane particles with a diameter of around 0.5 μm are

TABLE II
The Physical Properties and LLTS Results for Three Different Epoxy Formulations

Property	M1	M2	M3
Curing onset temp. (°C)	131	95	92
Curing peak temp. (°C)	173/217	190	178
$\alpha 1$ (ppm, CTE below T_g)	76	71	79
$\alpha 2$ (ppm, CTE above T_g)	196	196	193
T_g (°C)	113	153	174
The number of cycles for first fillet cracking observed	<300	<600	<100
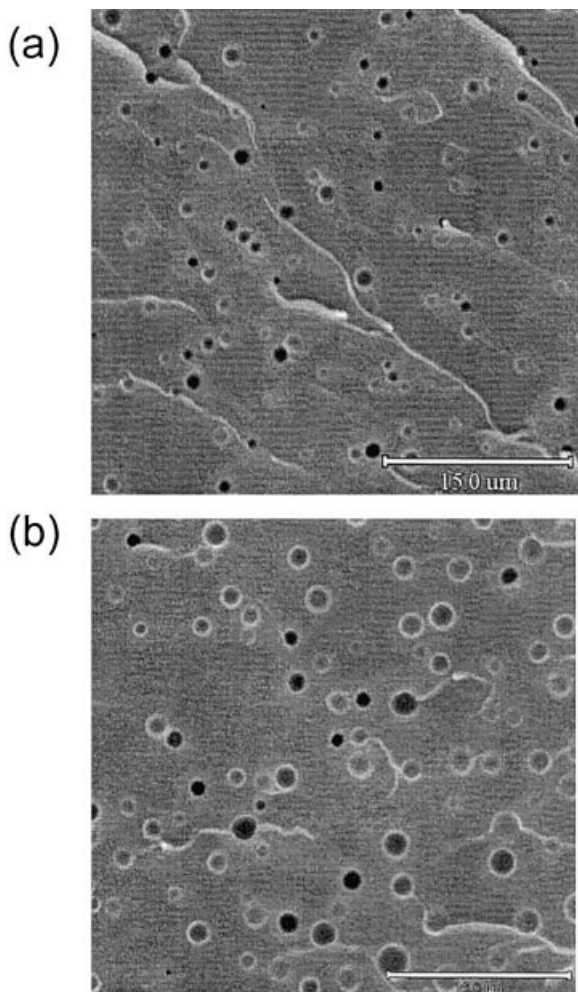
Figure 6 SEM photos of fracture surface of M2 system: (a) 5 phr and (b) 10 phr.

Figure 7 DMA results of unmodified and siloxane-modified epoxy formulations.

well dispersed in the epoxy matrix. The results for other formulations are not presented here since they have similar particle size dispersion. It has been known that the second phase such as a rubbery particle in the thermosetting polymer matrix forms through the competition between nucleation/growth process and curing of the polymer matrix. However, if the interfacial energy between rubbery phase and ma-
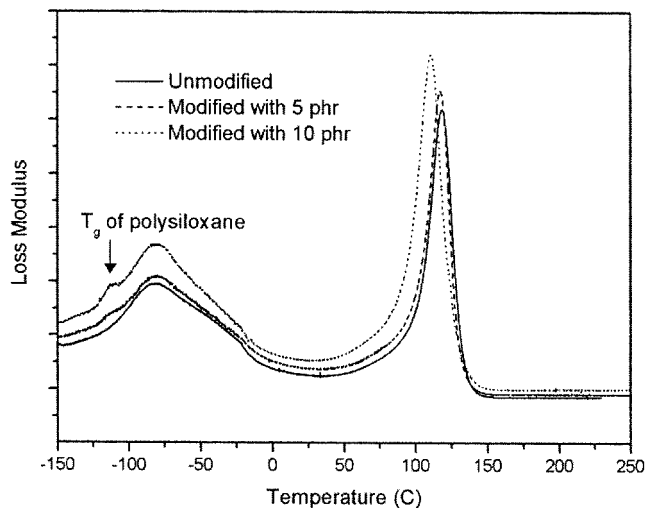
trix material is too high, the two phases would not be miscible, as such, it results in totally separated phases.[8] Such formulations could not give rise to uniform dispersion. On the other hand, if the miscibility between matrix material and toughening agent is too good, a homogeneous one phase will be obtained. Therefore, appropriate miscibility between two components would be important for the second-phase particle formation. From morphological observation, the polysiloxane used in this study is thought to have appropriate miscibility with our epoxy systems. It can also be seen that the particle size increases with the increase of content of polysiloxane. The particle size of the rubbery phase in epoxy can be affected by various parameters such as curing temperature, the content of rubbery phase, and interfacial energy between rubber and epoxy.[8] In some cases, the diameter of rubbery phase would be proportional to the content of the rubber. Figure 7 shows the results of the DMA for unmodified and polysiloxane-modified samples. For the modified formulation, the small peak at around −120°C indicates the glass transition of the second phase of polysiloxane formed in the epoxy matrix. The

TABLE III
The Physical Properties and LLTS Results for the Siloxane-Modified Epoxy Formulations

Base epoxy formulation	M1		M2		M3	
Siloxane loading (phr)[a]	5	10	5	10	5	10
Curing onset temp. (°C)	121	125	99	94	86	88
Curing peak temp. (°C)	174/222	172/217	189	195	178	179
$\alpha 1$ (ppm)	75	84	73	81	79	82
$\alpha 2$ (ppm)	197	203	198	198	187	198
T_g (°C)	115	98	152	148	181	165
The number of cycles for first fillet cracking observed	<300	<100	<700	<100	<100	<100

[a] Phr: part per hundred resin.

TABLE IV
Labels of the Base Epoxy- and Polysiloxane-Modified Formulations

Formulation label	Toughening agent
S00	No siloxane
S01	X01
S02	X02
S03	X03
S04	X04
S05	X05

T_g of polysiloxane is much lower than that of epoxy-toughening agents such as carboxylic/amine/epoxy-terminated butadienes (CTBN, ATBN, ETBN), which have been commonly used for toughening epoxy resin. Therefore, high performance at low temperature for polysiloxane-modified formulations would be expected.

The physical property and LLTS result for these modified formulations are shown in Table III. The polysiloxane does not significantly affect the curing profile. The 10 phr loading of polysiloxane seems to be too high to obtain the desirable performance and property. For the 10 phr loading, the formulation decreased its T_g substantially and increased CTE considerably. When the particle of toughening agent forms in the matrix material, the T_g of the entire system should not be changed since the system contains two different components such as the second phase parti-

cles and polymer matrix. However, all portions of toughening agent cannot be particles. Somehow, a part of the toughening agent could be dissolved into the matrix material. That is the reason why the T_g of the modified formulation decreased. It is thought that the amount of toughening agent that was dissolved into epoxy resin for the 10 phr loading sample, not phase-separated, was larger than for the 5 phr loading. The low reliability of the formulation modified with 10 phr is believed to be due to the rather higher CTE than that of the 5 phr loading samples. From the above results, the M2 formulation and polysiloxane of 5 phr were selected as the base epoxy formulation and the appropriate content of polysiloxane, respectively.

Incorporation of modifiers into epoxy formulations

To study the effect of toughening agent on the fillet cracking, polysiloxanes with different reactive groups such as the epoxide and amino group were incorporated into the M2 formulation. Polysiloxanes in this study and their corresponding formulations are shown in Table IV.

The X02, X03, and X04 are miscible in the epoxy resin used in this study since they have the epoxy functional group, which has a proper miscibility. The suitable miscibility results in enabling the simple mixing of the modifiers with epoxy resin just by stirring at room temperature. The modifiers were added to the
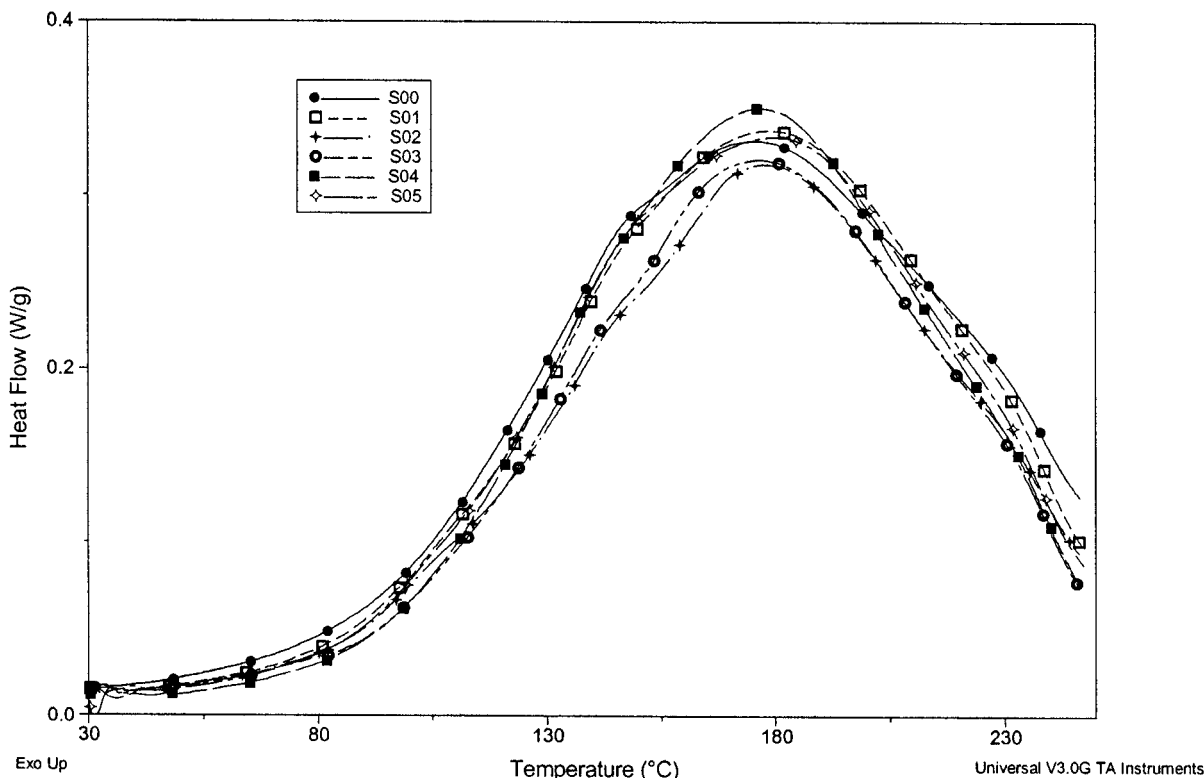

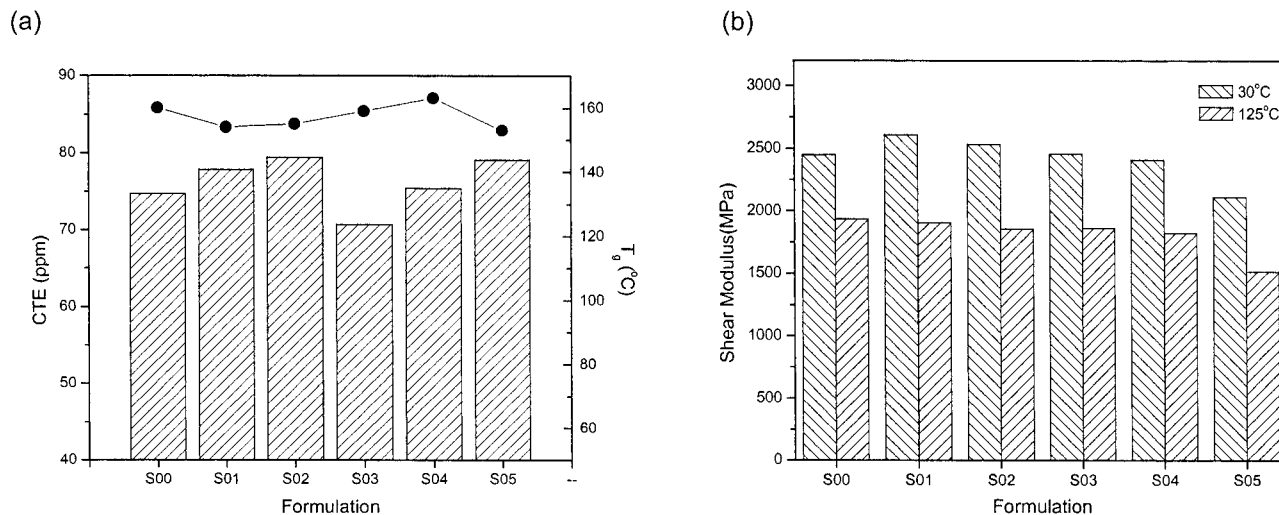**Figure 8** Curing profiles of polysiloxane-modified M2 formulations.

(a)

(b)

Figure 9 CTE (from TMA), T_g and shear modulus (from DMA) of polysiloxane-modified epoxy: (a) CTE and (b) shear modulus.

epoxy resin that had already been mixed with hardener, catalyst, flux agent, and coupling agent, and stirred rigorously for 10 min at room temperature. Polysiloxane X01 and X05 could not be mixed well with the epoxy resin used in this study only by stirring since these polysiloxanes have an amine functional group and are less compatible with epoxy resin. When X01 or X05 was mixed with epoxy resin by the same method as X02, X03, and X04, macrophase separation took place as if it were the mixture of water and oil. It has been reported that the uniform dispersion of amine-terminated siloxanes in epoxy resin would not be easily obtained, but epoxy-terminated polysiloxane could be a compatibilizer that helps amino-polysiloxane form a copolymer in epoxy resin, and reduce the interfacial tension between siloxane and epoxy.[17,18] X02 was used as a compatibilizer in order to mix amino-polysiloxanes with epoxy resin. The ratio of X02 and amino-siloxane in this study was 1:1 by weight. Yet, the mixture of X02 and amino siloxane could not be miscible unless at elevated temperature and with high shear blending. X02 and amino siloxane were mixed at 120°C and added to the epoxy resin, and then it could be mixed uniformly in the epoxy resin. The content of siloxane was fixed at 5 phr, resulting from a previous experiment. Figure 8 shows the curing profiles for bisphenol-A/cycloaliphatic epoxy with five different modifiers. As shown in the figure, those siloxane modifiers do not significantly affect the curing profile. All formulations have similar onset points and peak temperatures. The modifiers used did not influence the curing profile characteristics of the formulations.

CTE and T_g

Figure 9 shows CTE, glass transition temperature (T_g), and shear modulus of those formulations. Siloxane-

modified formulations show similar or slightly higher CTE than the unmodified one since the rubber phase of siloxane has a higher CTE than rigid epoxy phase. Glass transition temperature values from TMA measurement are also comparable for all formulations.

Adhesion strength

Adhesion is also a critical parameter for high performance underfill since fillet crack easily occurs and propagates once delamination between the underfill and a silicon die takes place.[19,20] Good adhesion could keep the interface between the chip and the underfill from delaminating. Figure 10 shows die shear strength of siloxane-modified formulations. All formulations, except S05, show rather lower adhesion strength than the unmodified one. X1, X2, X3, and X4 are reactive
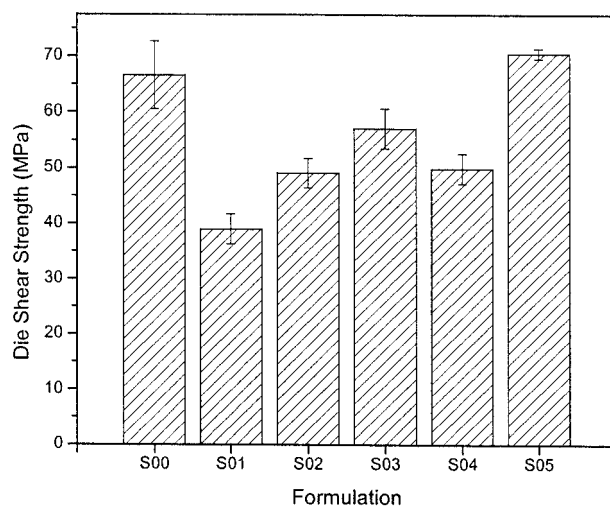

Figure 10 Die shear strength of polysiloxane-modified formulations.
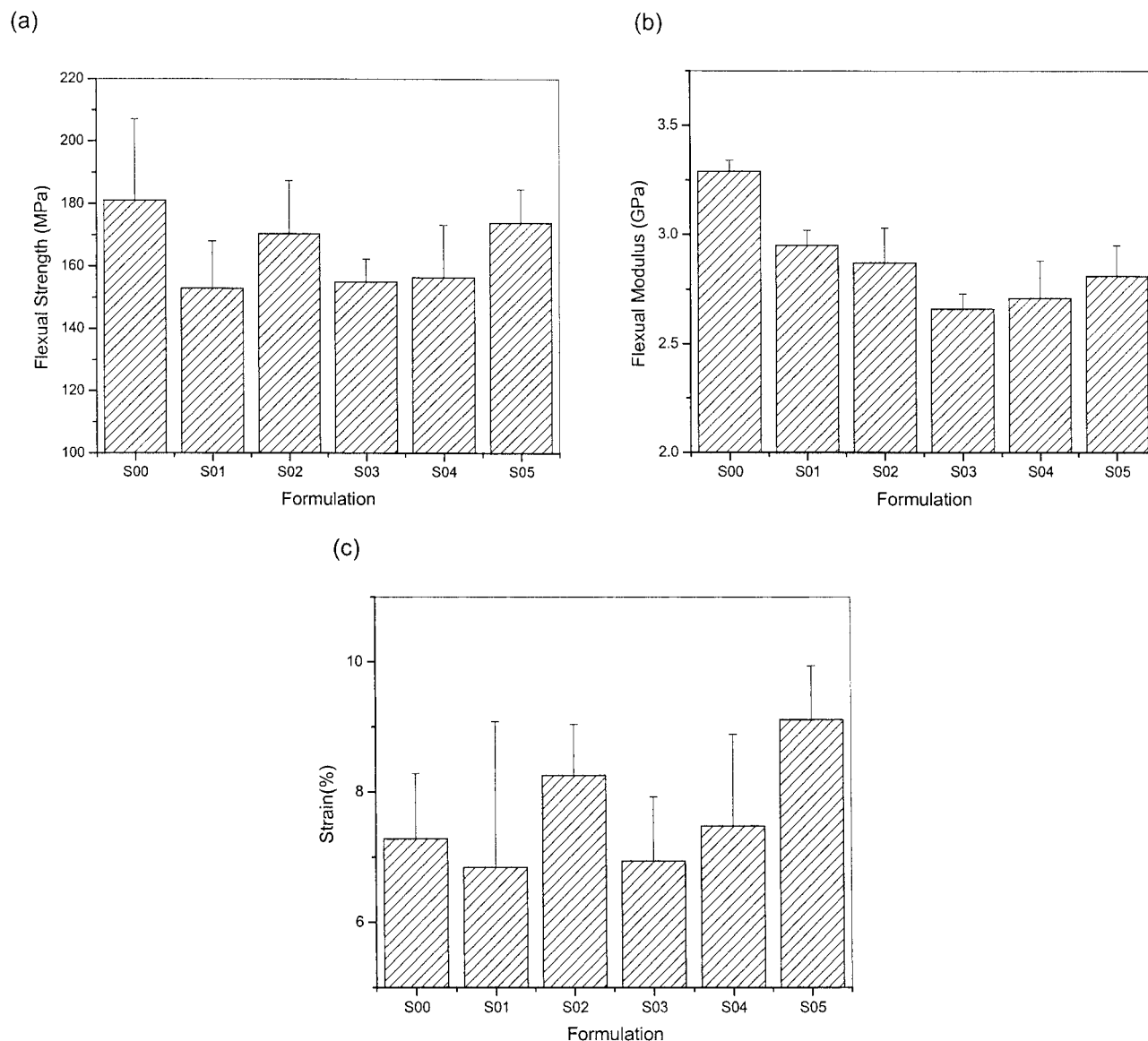
(a)

(b)

(c)

Figure 11 Flexual strength, modulus, and strain at break of modified epoxies: (a) flexual strength, (b) modulus, and (c) strain at break.

siloxane modifiers; nevertheless, it was found that they degrade the adhesion strength of the control formulation. Only S05 formulation shows slightly higher adhesion strength than the unmodified one.

Bending properties

Flexual modulus, fracture strength, and strain at break for S00–S05 are shown in Figure 11. The addition of a toughening agent to an epoxy system reduces the modulus due to the flexibility of the second-phase in the epoxy matrix. Since the internal or thermal stress of epoxy is proportional to elastic modulus, lowering the elastic modulus has been known to be preferable to reduce the internal stress. All polysiloxane modifiers used in this study were

effective in considerably reducing modulus. Fracture strength values for modified system are lower than that of the unmodified one, since the siloxane rubber has lower fracture strength than epoxy. Elongation at break could be another parameter to determine the flexibility of the materials. The S05 formulation shows the highest elongation at break.

Microstructure observation

The fracture surface of siloxane-modified epoxy system is shown in Figure 12. The S01 formulation consists of X01 and X02 as a compatibilizer. X01 was well dispersed and the second-phase particle was so clearly formed with the help of the compatibilizer. However, it can be seen that the size of particles are
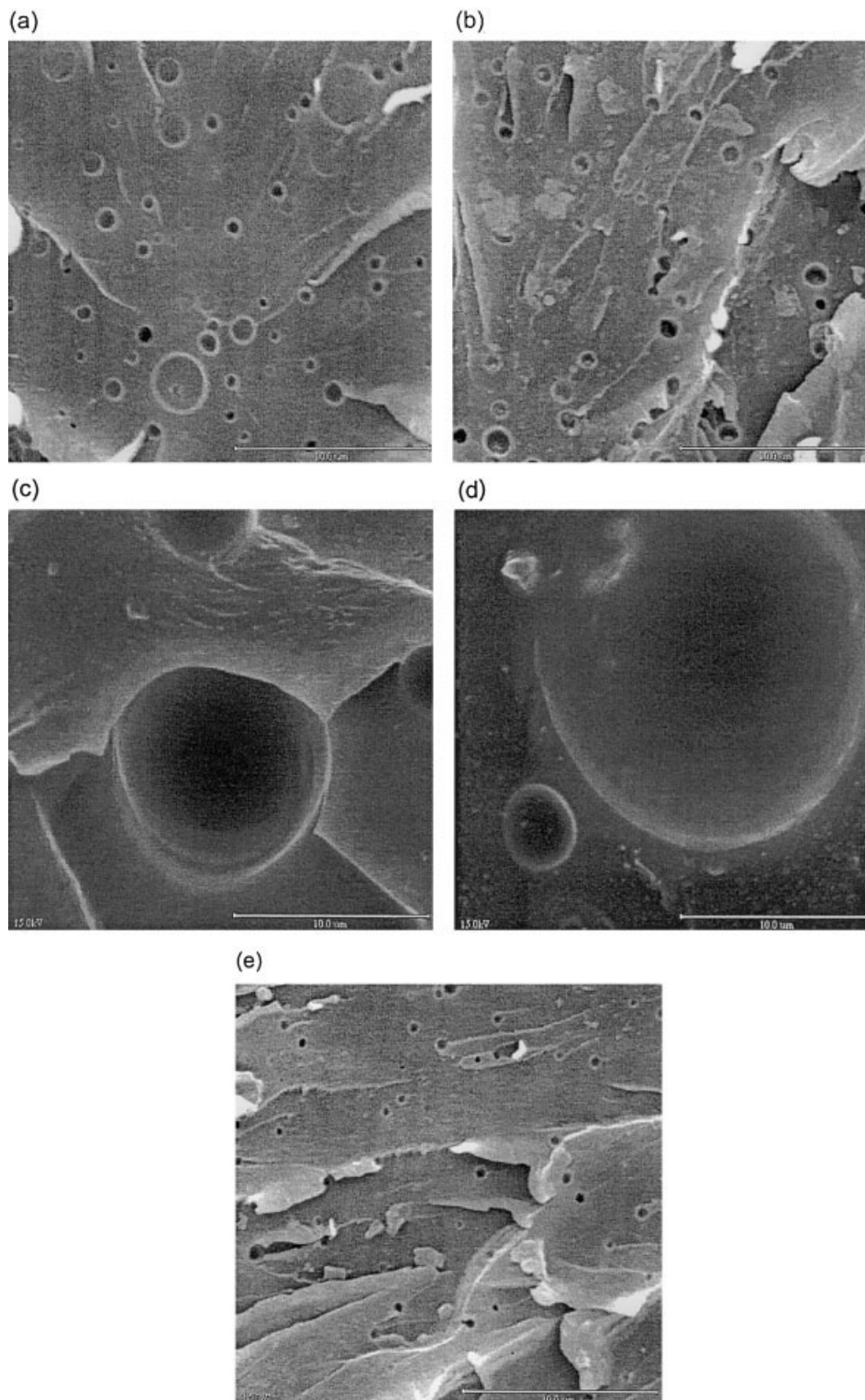
Figure 12 SEM photographs of polysiloxane-modified epoxies (×3000, bar size is 10 μm): (a) S01, (b) S02, (c) S03, (d) S04, and (e) S05.

not so uniform. The compatibility between X01 and the compatibilizer was good to improve the miscibility of X01 to epoxy formulation. The average diameter of the particles in the S02 formulation is about 0.5 μm; the size is reasonable to improve the

toughness of the system. For the S03 or S04 formulation, the particles of the diameter over 10 μm are seen, and the size seems to be too large to effectively improve the toughness. It is known that for improving the toughness of cured epoxy resins by the dispersed silicone elas-
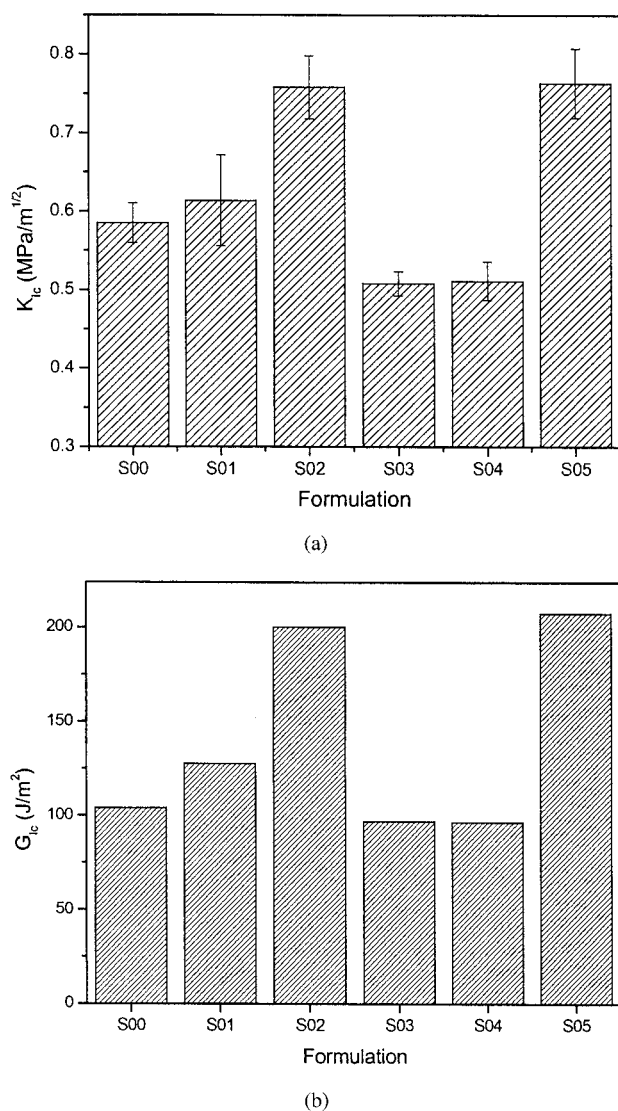
(a)

(b)

Figure 13 Stress intensity factor (K_{Ic}) and strain release rate (G_{Ic}) of polysiloxane modified epoxy: (a) K_{Ic} and (b) G_{Ic}.

tomer, the average diameter of the dispersed silicone phase should be decreased to the submicron size order.[21] The S05 formulation shows the finest siloxane phase with the diameter of 0.1–0.2 μm.

Fracture toughness

Figures 13(a) and (b) show the fracture toughness of stress intensity factor (K_{Ic}) and strain energy release rate (G_{Ic}) for siloxane-modified formulations. It can be seen that the S03 and S04 formulations have a rather lower K_{Ic} than the unmodified one. The particles of polysiloxanes in the S03 and S04 formulations are thought to be too large to improve the fracture toughness as shown in Figure 12. In general, smaller particle size of the second phase is more desirable than larger one for higher fracture toughness. During crack prop-

agation of the toughened epoxy, a few mechanisms are included, and they are combined and interact with. Among them, local shear deformation around toughening particles has been thought to be one of major mechanisms. The epoxy region surrounding those particles as well as the particle itself can absorb the crack propagation energy. The smaller particle has the larger surface area. Therefore, the formulation with smaller toughening particles would have higher toughness.

The S02 and S05 formulations show much higher toughness. From the SEM observation, the particle sizes of S02 and S05 were found to be 0.5 and 0.1 ~0.2 μm, respectively, which are presumed to be appropriate for toughening. Although the particle size of S05 was smaller than that of S02, the toughness was comparable. For G_{Ic}, which takes elastic modulus values into account, S02 and S05 also show considerably high G_{Ic}, especially S05 shows slightly higher value. Considering both K_{Ic} and G_{Ic}, it was obvious that the S02 and S05 formulations have high fracture toughness.

Reliability test

The above formulations were prepared and applied to the test vehicle assembly for the thermal shock test. The results of LLTS are listed in Table V, where S00 ~ S04 formulations showed the fillet cracking behavior before the required 1000 cycles. S03 and S04 showed lower cycling number, since those formulations are believed to have much lower fracture toughness compared with others. S02 showed a failure before 700 cycles, although high cycle number was expected due to its high toughness. Although S02 has a high fracture toughness, its adhesion strength was not so high as shown in Figure 10. The low adhesion strength of S02 is thought to cause its low reliability cycling performance. On the other hand, S05 surpassed all other formulations in the LLTS test. It was observed that many cracks propagated along the lateral plane of the fillets of S00, S01, S02, S03, and S04 formulations before 1000 cycles, while S05 showed no crack on the underfill for 1000 cycles, which was believed due to the comparably lower modulus, higher

TABLE V
The Number of Cycles at Which Fillet Cracking Occurs in LLTS (Test Was Done Until 1000 Cycles)

Formulation	The number of cycles for first fillet cracking observed
S00	<600
S01	<100
S02	<700
S03	<400
S04	<600
S05	No fillet cracking

strain and adhesion strength, and excellent fracture toughness although its CTE is similar to other formulations.

CONCLUSIONS

The effect of polysiloxane toughening agents of fillet cracking of underfills was investigated. The polysiloxane used were phase separated in the epoxy resin and rubbery particles formed. The second-phase particle of amine-terminated polysiloxane was formed with very fine (less than $0.1 \sim 0.2 \ \mu$m) size; as such, this no-flow underfill has passed 1000 cycles in LLTS with no fillet cracking.

From the physical properties measurement, it was concluded that in order to obtain an excellent thermal cycle performance of no-flow underfills in terms of fillet cracking, the suitable toughening agent that has well-controlled physical and thermal properties would be required. These underfills also should have high die shear strength, comparable CTE, high elongation, low modulus, phase separation of very fine particles with the matrix resin, and high fracture toughness.

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