Thermal shock test of integrated circuit packages sealed with epoxy moulding compounds filled with spherical silica particles

Yoshinobu Nakamura* and Miho Yamaguchi

Central Research Laboratory, Nitto Denko Corporation, Shimohozumi, Ibaraki, Osaka, 567 Japan

and Atsushi Tanaka

Product Engineering Research Laboratory, Nitto Denko Corporation, Nakahara-cho, Toychashi, Aichi, 441-31 Japan

and Masayoshi Okubo

Department of Industrial Chemistry, Faculty of Engineering, Kobe University, Nada, Kobe, 657 Japan (Received 25 August 1992; revised 7 October 1992)

The effect of filler silica particle size on the properties of integrated circuit (IC) packages sealed with epoxy moulding compound was studied. For this purpose, four epoxy moulding compounds filled with spherical silica particles having mean sizes in the range $6 \sim 31 \,\mu$ m were prepared. Critical stress intensity factor (K_c) and flexural strength (σ) of the cured epoxy moulding compounds were measured. As the particle size increased, the K_c value increased and the σ value decreased. The thermal shock test was carried out: IC packages were repeatedly dipped alternately in liquids at -65° C and 150° C and the occurrence of package cracking was observed. The cracking took place at an earlier stage of the thermal shock test when the particle size was smaller. There was a good relationship between the thermal shock test result and the K_c value.

(Keywords: epoxy resin; encapsulating materials for integrated circuits; silica particle; fracture toughness; flexural strength; thermal shock test)

INTRODUCTION

Recently, in integrated circuits (IC) sealed with epoxy moulding compounds, the chip size has increased rapidly while the package dimensions have become smaller and thinner¹. These trends increase thermal stress in the device package system which often causes package cracking during thermal shock testing under accelerating ambient conditions¹⁻⁴. Therefore, it has become important to design the device package so as to reduce the thermal stress⁴ and to increase the strength and toughness of the cured epoxy moulding compounds².

We have recently investigated⁵⁻¹² the effects of particle shape and size on the fracture toughness^{5,9,10,12}, mechanical strength^{6,9,11,12} and impact properties^{8,11} of cured epoxy resin filled with silica. In these studies, irregular-shaped^{5-9,12} or spherical silica particles^{7,10,11} ranging from 2 to about 50 μ m in diameter were dispersed in the liquid-type epoxy resin⁵⁻¹¹ (bisphenol A-type epoxy resin and dicarboxylic anhydride hardener) and in the solid-type epoxy resin¹² (o-cresol novolac-type epoxy resin and phenol novolac-type resin as a hardener). The former was developed as a 'model system' and the latter

0032-3861/93/153220-05

© 1993 Butterworth-Heinemann Ltd.

was the actual cured moulding compound of the encapsulating material for IC. In these studies, as the particle size increased, the fracture toughness increased but the flexural strength decreased. That is, the particle size has an opposite effect on the fracture toughness and the flexural strength. Therefore, the relationship between these values of the cured epoxy moulding compound and the practical properties of the IC package sealed with the epoxy moulding compound must be clarified. In this article, it will be investigated using IC packages sealed with epoxy moulding compounds filled with spherical silica particles having four different particle sizes.

EXPERIMENTAL

Materials

Four kinds of spherical silica particles having different mean sizes (FB-30, FB-35, FB-48, and FB-74, Denki Kagaku Kogyo Co., Ltd) were prepared as follows. First, the irregular-shaped particles were prepared by crushing fused amorphous silica. Then, these particles were fused in a flame to make them spherical. *Figure 1* shows the size-distribution curves obtained using a laser beam size distribution analyser (Granulométre 715 type, Cilas

^{*} To whom correspondence should be addressed



Figure 1 Cumulative size-distribution curves for spherical silica particles. Mean particle size: (\bigcirc) 6, (\bigcirc) 12, (\bigtriangleup) 15, (\square) 31 μ m

Alcatel). As described previously^{5,7,10}, the particle sizes at which the cumulative distribution values reached 50% were defined as the mean particle sizes.

The epoxy resin used was *o*-cresol formaldehyde novolac-type epoxy resin (ESCN-195XL, Sumitomo Chemical Co., Ltd, equivalent weight per epoxy group: 195, softening point: 85° C). Phenol formaldehyde novolac resin (P-180, Arakawa Chemical Industries, Ltd, equivalent weight per hydroxyl group: 104, softening point: 80° C) and 1,8-diazabicyclo(5,4,0)-7-undecene (DBU) were used as hardener and accelerator for curing of the epoxy resin, respectively.

Sample preparation

Table 1 shows the formulation of an epoxy moulding compound. These materials were mixed in a mixing roll at 110° C for 5 min, then cooled and crushed. The test specimens were prepared by transfer moulding at 175° C for 2 min and cured in an oven at 175° C for 5 h.

Fracture toughness test

The critical stress intensity factor (fracture toughness, K_c) and critical strain energy release rate (fracture energy, G_c) were measured by a Single Edge Notched Beam loaded in three-point bending (SENB) test according to ASTM¹³. The specimen size was 10 mm × 4 mm × 44 mm with a support span of 40 mm having a slot at the centre. A sharp initial crack was introduced at the base of the slot by pressing in a new razor blade. The displacement rate was 10 mm min⁻¹. The conditions were explained in detail in previous articles^{5,10}.

Flexural test

The specimen size was $10 \text{ mm} \times 4 \text{ mm} \times 80 \text{ mm}$ with a support span of 64 mm. The displacement rate was $5 \text{ mm} \text{min}^{-1}$ (ASTM D790). The conditions were explained in detail previously⁶.

Thermal mechanical analysis

Thermal expansion coefficients below and above the glass transition temperature (T_g) of the cured epoxy moulding compounds and the value of T_g itself were measured by a thermal mechanical analyser (TMA/SS-100, Seiko Instruments Inc.). The conditions were detailed in a previous article¹⁴.

Thermal shock test

The package used in this study was an 80 pin Quad Flat Package (QFP, 20 mm × 14 mm × 2 mm) with an Alloy 42 (alloy of Fe and Ni (Ni: 42 atom %)) lead frame. The chip and the chip pad sizes were 7.5 mm × 7.5 mm and 8 mm × 8 mm, respectively. The thermal shock test (TST) was carried out using a liquid-bath thermal shock chamber (TSB-1L, Tabai Espec Corp.) in which test packages were repeatedly dipped alternately in liquids at -65° C and 150°C for 5 min each with 30 s in transition. There were 20 test packages for each epoxy moulding compound.

RESULTS AND DISCUSSION

Figure 2 shows scanning electron microscopic (SEM) photographs of the polished surfaces of cured epoxy resins filled with spherical silica particles. In these systems, particles were well dispersed in the cured epoxy matrix.

Table 2 shows some thermal and mechanical properties of cured epoxy moulding compounds. As the particle size increased, the fracture toughness (K_c and G_c) values increased, while the flexural strength decreased. The effects of particle size on the K_c and flexural strength values are shown in *Figure 3*; the particle size had an opposing effect on the fracture toughness and the flexural strength, as described in previous articles^{10,11}.

Oizumi *et al.*¹⁵ reported the failure phenomena during TST. Before TST, the chip pad and the encapsulating material (cured epoxy moulding compound) were completely adhered. As the number of the dipping cycles in the TST increased, delamination between the chip pad

Table 1 Formulation of epoxy moulding compound (wt%)

Epoxy resin (o-cresol formaldehyde novolac resin)	27.03
Hardener (phenol formaldehyde novolac resin)	17.83
Accelerator (1,8-diazabicyclo(5,4,0)-7-undecene (DBU))	0.14
Spherical silica particles	70.00

 Table 2
 Some thermal and mechanical properties of epoxy moulding compounds filled with spherical silica particles

	Mean particle size of filled silica (μ m)			
	6	12	15	31
Thermal mechanical analysis				
$\alpha_1 (\times 10^{-5} \text{ deg}^{-1})^a$	2.0	2.0	2.0	1.9
$\alpha_2 (\times 10^{-5} \text{ deg}^{-1})^b$	6.4	6.5	6.5	6.5
$T_{\mathbf{s}}$ (°C)	168	170	169	169
Flexural test ^c				
Flexural modulus (GPa)	13.5	14.0	14.3	14.4
Flexural strength (MPa)	138	138	135	127
Displacement at break (mm)	1.9	1.8	1.8	1.7
Fracture toughness test ^d				
$K_{\rm c} ({\rm MPa}{\rm m}^{1/2})$	1.6	1.9	2.0	2.2
$G_{\rm c}$ (× 10 ⁻¹ kJ m ⁻²)	2.1	3.0	3.1	3.5
Thermal stress during TST				
Calculated thermal stress (MPa) ^e	435	452	461	433

"Thermal expansion coefficient below T

^b Thermal expansion coefficient above T_{a}

^cThree-point bending flexural test. Displacement rate: 5 mm min⁻¹

^d Single edge notched beam loaded in three-point bending (SENB) test.

Displacement rate: 10 mm min⁻¹

^e Calculated using equation (1) with $\alpha_L = 0.5 \times 10^{-5} \text{ deg}^{-1}$

Thermal shock test of IC packages: Y. Nakamura et al.



Figure 2 SEM photographs of polished surfaces of cured epoxy moulding compounds filled with spherical silica particles at a particle content of 70 wt%. Mean particle size of silica filler: (a) 6, (b) 12, (c) 15, (d) $31 \mu m$

and the encapsulating material progressed from edge to centre of the interface. Since the curvature radii of the edge tips are usually of the order of 1 μ m (ref. 2), after complete delamination of the interface, the crack originated from the edges of chip pad and propagated to the bottom surface of the package. In this study, to eliminate the influence of interfacial adhesion between the chip pad and the encapsulating material, the bottom surface of the chip pad was coated with silicone grease (deposited from toluene solution and dried in an oven). The interface was completely delaminated before test.

Figure 4 shows the result of TST. The degree of package cracking indicates the percentage of the packages where cracks were visible on the package surface. Cracks in the package's interior were not counted. Before TST, an initial crack of length $10 \sim 30 \,\mu\text{m}$ was observed at the lower edges of both sides of the chip pad in the packages sealed with the small and large particle-filled resins. These



Figure 3 Effects of particle size on (\bigcirc) flexural strength and (\bigcirc) critical stress intensity factor (K_c) of cured epoxy moulding compounds filled with spherical silica particles with a particle content of 70 wt%



Figure 4 Package cracks caused during thermal shock test of IC packages sealed with epoxy moulding compounds filled with spherical silica particles with a particle content of 70 wt%. Mean particle size of filler silica: (\bigcirc) 6, (\bigoplus) 12, (\bigtriangleup) 15, (\square) 31 μ m

cracks propagated to the bottom surface of the package during TST. In the smallest particle-filled system (\bigcirc) , most of the packages cracked within 50 cycles, while in the largest particle-filled system (\Box) , about half of the packages did not crack in over 100 cycles. Thus, the rate of occurrence of package cracking during TST decreased with increase in particle size.

As shown previously⁴, the thermal stress caused during TST at the edge of chip pad is calculated according to the following equation:

$$\sigma = \int_{-65}^{150} E(\alpha_1 - \alpha_L) \,\mathrm{d}T \tag{1}$$

where σ is the thermal stress caused during TST, α_1 and E are the thermal expansion coefficient below T_g and the flexural modulus of cured epoxy moulding compound, respectively, and α_L is the thermal expansion coefficient of the lead frame (chip pad, $=0.5 \times 10^{-5} \text{ deg}^{-1}$ was used³). It was also clarified that as the thermal stress decreased, the occurrence of package cracking decreased⁴.

The thermal stress values evaluated using equation (1) are shown in *Table 2*. Since the values were almost the same, the TST result seems to be caused by the difference of the mechanical properties of cured epoxy moulding compounds. That is, the TST result showed the same tendencies as the fracture toughness values (K_c and G_c) of the cured epoxy moulding compound, while it was independent of the flexural strength values.

Next, the crack propagation during TST was observed with a SEM. For this purpose, the dipping was stopped before the crack reached the bottom surface of the package, the IC package was cut into two vertically and the exposed surface was polished.

Figure 5 shows the cross-sections of the test packages. In the smallest particle-filled system (Figure 5a, mean size, 6 μ m), the crack propagation was linear. On the other hand, in the largest particle-filled system (Figure 5b, mean size, 31 μ m), the crack propagation was deflected markedly by the large particles. Furthermore, debonding of particle/matrix interfaces, divergence of cracks and the particle fracture were observed along the propagated crack path. Such a microfracturing seems partially to release the stored strain energy at the crack tip, and obstruct the crack propagation. This region in which microfracturing occurred is called the 'damage zone'^{16,17}. In the smallest particle-filled system (Figure 5a), no damage zone was observed.

The initiation point of fracture at fractured surfaces of the flexural test specimen was observed by SEM as described previously^{6,11}. In the small particle-filled resin, the fracture was initiated from the defect which had existed on the specimen surface or edge. However, in the large particle-filled resin, the fracture was initiated from the particle fracture in all tested specimens. As mentioned earlier, the spherical silica particles used in this study were fused in a flame during the preparation process. In this process, a portion of the particles agglomerated, resulting in 'irregular' particles. Such agglomerated irregular particles may be cracked easily when the stress is concentrated. The same observation was made in the similar spherical silica-filled systems described in previous articles¹¹.

The crack tip regions of the fracture toughness test specimens before and after the test were observed as before^{5,10}. The observed phenomena were exactly the same as the above-mentioned effect of the particle size on the crack propagation during TST and on the fracture toughness obtained in the similar spherical silica-filled systems shown previously¹⁰.

The opposing effect of the particle size on the flexural strength and the fracture toughness, which was clarified in our previous articles^{5,6,10,11}, was explained as follows. In the flexural test, the strength depends on the size of the inherent flaw in the specimen^{6,11}. On the other hand, in the fracture toughness test, the toughness depends on the resistance to crack propagation from the constant-sized flaw (the starter crack) which had been introduced in all specimens using a fresh razor blade^{5,10}. As described above, as the particle size increases the inherent flaw of each particle increases, and the resistance increases because of the energy absorption by the crack deflection and the formation of the damage zone. Therefore, in our system, the particle size reacts negatively on the flexural strength but positively on the fracture toughness.

From the above results, it is concluded that the package cracking during TST of the IC packages sealed with the

Thermal shock test of IC packages: Y. Nakamura et al.



Figure 5 Polished revealed surfaces of cross-sections of IC packages during thermal shock test observed by SEM sealed with epoxy moulding compounds filled with spherical silica particles with a particle content of 70 wt%. Mean particle size of filler silica: (a) 6, (b) 31 µm. Dipping cycles before observation: (a) 30, (b) 110

epoxy moulding compounds filled with spherical silica particles is dependent on the fracture toughness values, while independent of the flexural strength values.

ACKNOWLEDGEMENT

The authors are grateful to Denki Kagaku Kogyo Co., Ltd for preparation of sample silica particles.

REFERENCES

- Kinjo, N., Ogata, M., Nishi, K. and Kaneda, A. Adv. Polym. Sci. 1 1989, 88, 1
- Nishimura, A., Tatemichi, A., Miura, H. and Sakamoto, T. IEEE 2 Trans. Components, Hybrids, Manuf. Technol. 1987, 12, 637 Nishimura, A., Kawai, S. and Murakami, G. IEEE Electron.
- 3 Components, Conf. 1989, 39, 524
- 4 Nakamura, Y., Uenishi, S., Kunishi, T., Miki, K., Kuwada, K., Tabata, H. et al. IEEE Trans. Components, Hybrids, Manuf. Technol. 1987, 12, 502

- Nakamura, Y., Yamaguchi, M., Kitayama, A., Okubo, M. and 5 Matsumoto, T. Polymer 1991, 32, 2221
- 6 Nakamura, Y., Yamaguchi, M., Okubo, M. and Matsumoto, T. J. Appl. Polym. Sci. 1992, 44, 151
- 7 Nakamura, Y., Yamaguchi, M., Okubo, M. and Matsumoto, T. J. Thermosetting Plast. Jpn 1991, 12, 1
- Nakamura, Y., Yamaguchi, M., Okubo, M. and Matsumoto, T. 8 Polymer 1991, 32, 2976
- Nakamura, Y., Yamaguchi, M. and Okubo, M. Polym. Eng. Sci. 9 1993, 33, 279
- Nakamura, Y., Yamaguchi, M., Okubo, M. and Matsumoto, T. 10 Polymer 1992, 33, 3415
- Nakamura, Y., Yamaguchi, M., Okubo, M. and Matsumoto, T. 11 J. Appl. Polym. Sci. 1992, 45, 1281
- Nakamura, Y., Yamaguchi, M. and Okubo, M. J. Adhes. Soc. 12 Jpn 1992, 28, 308
- ASTM Committee D-20 on Mechanical Testing, Project no. 13 X-10-128
- Nakamura, Y., Tabata, H., Suzuki, H., Iko, K., Okubo, M. and 14 Matsumoto, T. J. Appl. Polym. Sci. 1986, 32, 4865
- Oizumi, S., Imamura, N., Tabata, H. and Suzuki, H. Am. Chem. Soc. Symp. Ser. 1987, 346, 537 15
- Narisawa, I. Kobunshi Ronbunsyu 1988, 45, 683 16
- Pearson, R. A. and Yee, A. F. J. Mater. Sci. 1989, 24, 2571 17