

Laporan Akhir Projek Penyelidikan Jangka Pendek

Low CTE Substrate to Improve Warpage and Solder Joint Realibility

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2008

Intel Research Grant – Low CTE substrate to improve warpage and solder joint realibility [304/PBAHAN/6050049/I104]

1. Introduction

In electronic packaging industry organic substrate materials have been introduced in market to meet the trends of low cost, high interconnect and better reliability. Epoxy resin is one of the wisely used material in electronic packaging industries due to their ease of processing and low cost [1-3]. Many researchers are agreed with the statement of epoxy resins have excellent heat, moisture, and chemical resistance [4, 5]. It is previously observed that organic substrate based on polymeric materials generates warpage during thermal cycling test [6]. Warpage is caused by the large different of coefficient of thermal expansion (CTE) between the substrate and silicon die. CTE of epoxy, normally at above 80ppm/°C, is relatively high compared to silicon chip (2-3 ppm/°C). With incorporation of inorganic fillers such as silica, make the CTE of epoxy composites change to lower value.

Commercial epoxy resins based on bisphenol A, in average have viscosities of 10,000-16,000 cP at 25 °C. Therefore, it has been an issue to produce high filled epoxy composites. Viscosities of epoxy increased rapidly with incorporation of fillers. To overcome this issue, most epoxy matrices are often diluted with diluent to suit with applications. According to Pourabas & Raeesi [7], there are two major approaches to reducing viscosity, either thinning the mixture with low viscosity organic solvents or adding low viscosity reactive diluent. Reactive diluent can participate, together with a resin and a curing agent, in crosslinking reactions, thus permitting the diluent to become chemically bound into the crosslinked network, while non-reactive will not take action in curing reactions.

The formation of C-C, C-N, C-O or C-S bonds via nucleophilic opening of epoxies has been extensively studied. Research on the opening of epoxies with alcohol have been done by Ollevier & Lavie-Compin [8]. They have found that hydroxyl compounds, such as alcohols, act as catalysts and accelerate curing, but are not serious competitors with amines for reacting with the epoxide ring as shown in Figure 1.

So far, there are many works on epoxy composites diluent systems have been wisely published [9-11]. However, there is no report on ethanol as the diluent in epoxy composites yet. In this study, we will examine the effect of ethanol as the diluent in epoxy resin. The composites are then produced by incorporating of mineral silica in the epoxy resin that has been cured by aromatic amine curing agent. Theoretical, ethanol will reduce the viscosity of resin. A lower viscosity resin will allow addition of a greater amount of fillers while maintaining the process ability of the epoxy resin. This diluent system is believed to produce highly filled epoxy composites and provide a great variety of resin properties suitable for wide applications, particularly in the electronic packaging application. The fused silica is the preferred fillers used in electronic packaging industry due to its low CTE. However, in this study, mineral silica with CTE of 2.64 ppm/°C is used as the filler to reduce the CTE of the epoxy resin. This filler is produced locally. It has uniformity excellent properties. The comparison properties between the fused and mineral silica filled epoxy composites were reported in our previous study [12].

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Figure 1. Chemical reaction between alcohol, amine curing agent and epoxy resin.

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2. Experimental

2.1. Materials

Epoxy resin (EPON 8281)- clear difunctional bisphenol A/epichlorohydrin, used in this study was a commercial product from Shell Chemical, USA. Ethacure A100 (chemical name: diethyltoluenediamine) used as curing agent was supplied by Albermarle Corp. Ethanol was supplied by Fluka (Malaysia) was used as diluent. Mineral silica was grinded from local sand by fine grinding technique. The average particle size of mineral silica is 10 micron.

2.2. Sample preparation

Epoxy resins were added with ethanol at 10% by weight, and then the mixtures was fully mixed with the mineral silica at 0-40vol% for non-diluent system and 0-60vol% for diluent system. Based on our research, it was found that for non-diluent and diluent systems, the maximum addition of filler contents in epoxy resin are 40vol% and 60vol%, respectively. The mixtures were then added with curing agent, Ethacure A100 at 24 phr. The mixtures were stirred by a mechanical stirrer at 20 rpm for 20 min and then degassed in a vacuum oven to eliminate air bubbles before pouring into a mould. Then, the mixtures were precured at 100°C for 2hr and post cured at 175°C for 4hr. Table 1 displays the epoxy composites produced along with their coding.

Table 1. Designation and composition of the mineral silica filled epoxy composites.

Filler content	Code	
(vol%)	Non- diluent	Diluent
0	END	ED
10	10END	10ED
20	20END	20ED
30	30END	30ED
40	40END	40ED
50		50ED
60		60ED

2.3. Thermal properties

2.3.1. Dynamic mechanical analysis

Glass transition temperature and dynamic flexural modulus were measured by using a Perkin-Elmer dynamic mechanical analyzer at a scan rate of 2 °C/ min under three point bending mode with the sample dimension of 10 X 50 X 3 mm. Crosslink density, ν is calculated based in equation (1), ν is expressed in moles of elastically effective network chains per cubic meter of sample [13].

$$\nu = \frac{E'}{3RT} \tag{1}$$

where E' is the elastic modulus, R is the universal gas constant and T is the temperature in K.

2.3.2. Coefficient of thermal expansion (CTE)

CTE measurements of the composite samples were performed on a dilatometer. These samples had a dimension of 5 X 2 X 10 mm. The samples were mounted on the dilatometer and heated from -50 to 300° C at a heating rate of 10 °C/min. The coefficient of thermal expansion was determined from the slope of the plot between thermal expansion and temperature. The CTE measurements were carried out for two different samples with the same composition, and the average value is reported.

2.3.3. Thermo-gravimetric analysis

A Perkin-Elmer thermo gravimetric analyzer (TGA) was performed to investigate the thermal stabilities of the composite samples from 30 to 600°C at a heating rate of 10 °C/min. The weight loss provides an indication of decomposition of the composite materials. The observed weight loss was analyzed.

2.4. Mechanical properties

Flexural measurements were carried out using an Instron-3366 machine at 23°C, according to ASTM D790 using 3-point bending configuration at 2.38 mm/min deformation rate. Fracture toughness measurements were tested using the Instron-3366. The single-edge notched samples were cut out and subjected to static tensile loading (SEN-T samples). The sample notching is done by sawing and sharpening with a razor blade. SEN-T samples were tested at 1 mm/min at 23°C. The geometry of the sample is shown in Figure 2. The fracture toughness was determined according to ISO 13586 with fracture toughness parameters, which are calculated by the following equations, equation (2) and (3) [14]:

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$$K_{C} = \frac{F_{\max} \sqrt{a}}{BW} f\left(\frac{a}{w}\right) \quad \text{(2)}$$

 $f\left(\frac{a}{W}\right)$ is the geometry correction factor which is given by:

$$f\left(\frac{a}{W}\right) =$$

$$1.99 - 0.41 \left(\frac{a}{W}\right) + 18 \cdot 7 \left(\frac{a}{W}\right)^{2} - 38 \cdot 48 \left(\frac{a}{W}\right)^{3}$$

$$+ 53 \cdot 85 \left(\frac{a}{W}\right)^{4}$$

$$(3)$$

Where: F_{max} is the maximum force in the force-deflection trace; *B* is the thickness of the sample; *W* is the width of the sample; and *a* is the total notch length.

3. Results and Discussion

3.1. Mechanical Properties

Flexural properties are important in understanding the materials mechanical behavior in flexural mode at room temperature, since it combines effects of compressive deformation (adjacent to the point of applied load) and tensile deformation (on the opposite side of the specimen). Flexural strength and modulus results are shown in Figure 2(a) and (b), respectively, for both non-diluent and diluent systems. In both systems, it was found that the flexural strength and modulus increases as the filler content increased as shown in both figures. As the filler content increases the mobility of composite system will be limited and thus, gives stiffer behavior. On the other hand, higher values of flexural strength and modulus were found in non-diluent system at the same filler loading. In other words, the system with ethanol used as diluent exhibited lower

flexural strength and flexural modulus than those of the non-diluent system. However, it should be reiterated that the advantage of diluent system is the ability to load the higher filler content, i.e. 60vol% of filler content, which shows flexural modulus and strength of 8872 and 95.90 MPa, respectively.



(b)

Figure 2. (a) Flexural strength and (b) flexural modulus of epoxy composites for both nondiluent and with diluent systems.

Fracture toughness is used to quantify the resistance of crack propagation of the materials [15]. Micro crack can be propagated under the repeating load and led to the failure of the substrate during thermal cycling. Figure 3 shows the fracture toughness of epoxy composites for both non-diluent and diluent systems. In Figure 3, non-diluent epoxy composites show increasing K_c values with increased of filler contents; this showed that the mineral silica could toughen the epoxy as filler loading increases and thus, act as the crack stopper in the matrix. However, when diluent are added into the epoxy systems, the K_c value of epoxy composites shows the reverse

trend. According to Gam *et al.* [16] and Plangsangmas *et al.* [17], one of the factors which might influence the toughness behavior of polymer is the crosslink density of the polymer network. Epoxies with higher crosslink densities will have lower fracture toughness values. (The crosslink density values will be discussed later in dynamic mechanical properties in section 3.2.). It is because high crosslinked epoxy resins have limited ability to deform by shielding and crazing, especially in the tri-axial stress field present inside the sample at the crack tip [18]. Therefore, addition of ethanol as diluent changes this situation and can significantly improve the fracture toughness. This explained why the higher values of K_c values showed in epoxy systems with diluent compared to those of non-diluent systems. The fracture toughness of epoxy resins is a complex phenomenon and it is affected by many factors, such as the volume fraction of fillers, properties of modifier such as diluent, compatibility between the filler and the epoxy matrix, or the curing conditions.



Figure 3. Fracture toughness of epoxy composites for both non-diluent and with diluent.

3.2. Dynamic Mechanical Properties

Dynamic mechanical testing can provide us the information of viscoelastic behavior polymer materials in the temperature range of -50° C - 300° C. The storage modulus and Tan δ of epoxy composites with diluent and non-diluent systems are shown in Figure 4(a) and (b), respectively. For substrate materials, high modulus is needed to effectively redistribute solder joints stress in order to prevent the warpage occur [19]. Higher modulus can be obtained by adding high content of silica filler. Therefore, higher content of silica filler is preferred, unless the viscosity of epoxy resin is too high and unable to provide a well dispersion of fillers in matrix. Based on Figure 3(a), in overall, all diluent epoxy composites exhibited lower flexural storage modulus than the non-diluent system at the same filler content. According to Wilkinson & Ryan [20], when the low molar mass diluent are added into the epoxy network, an effect called plasticizing will be

occurred. With incorporation of diluent, the network of the epoxy will become more flexible due to the introduction of addition free volume [21]. The addition of filler will increase the modulus of composite systems; for example, when the filler content in diluent system is up to 60vol%, the flexural storage modulus is increased up to 15GPa. This epoxy composites exhibited higher flexural modulus than that non-diluent system filled at 40vol%. It is believed that the fillers filled up the free volume space sufficiently and increased the stiffness of the epoxy.

The glass transition temperature and crosslink densities of the epoxy composites are shown in Table 2. In overall, all the epoxy composites systems have T_g at above 150°C. It is in the range of requirement for electronic packaging application as the maximum operation temperature at thermal cycling is 150°C. All the diluent systems showed lower T_g and crosslink densities compared to the non-diluent systems. As known that ethanol is consisted of aliphatic segments. The presence of significant amounts of aliphatic segments in the chemical structure of the cured epoxy network resulted the lower rigidity and lower T_g [20].



Figure 4. Dynamic (a) flexural storage modulus and (b) $Tan \delta$ of the epoxy composites for nondiluent and diluent systems.

 Table 2. Glass transition temperature and crosslink density of selected epoxy composites for

 diluent and non-diluent systems

Formulations	Glass	Crosslink
	transition	density,
	temperature,	V
	<i>T_g</i> (°C)	$\times 10^3 (\text{mol/m}^3)$
END	179.78	76.28
40END	189.79	115.75
ED	165.10	17.51
40ED	168.08	38.04
60ED	177.01	52.88

3.3. Thermal Expansion

The thermal expansion behavior of packaging materials plays a critical role in affecting the thermo-mechanical reliability of devices. During the thermal cycling, the substrate materials are expected to contract more than the silicon chips and solder balls due to the CTE mismatch between the two materials. This could potentially cause deterioration in thermal performance. Therefore, the closer the CTE of the substrate with that of silicon chips or solder balls is the better properties of the substrate materials. Note that, the CTE of a typical solder ball is in the range of 24-29 ppm/°C [22].

Figure 5 exhibits the CTE and T_g value for the epoxy composites for both non-diluent and diluent systems. For any given filler loading, the filled epoxy composites showed lower CTE than that of unfilled epoxy resin. This is mainly due to the nature intrinsic low CTE of filler. This statement is in agreement with Powell and co-researchers[22]. For the same filler loading at 40vol%, the CTE of non-diluent systems showed lower CTE than with diluent systems. CTE of the materials is depends on the cross-linking. Cross-linking is the process in which the polymer chains are linked together chemically by short chemical bonds to form a network, therefore transforming the material from a viscous material to an elastic solid. According to Kornaski [23], CTE is depends on the chain stiffness. High cross-linking network will bring high chain stiffness and thus lower the CTE of the materials. However, when the filler loading is up to 60vol% for the

diluent system, the CTE of epoxy composite is reduced to 18.5 ppm/°C compared to 108.3 ppm/°C of the unfilled system. The CTE value of 18.5 ppm/°C shown by 60vol% of the diluent systems is close to the CTE of solder ball as mentioned before. This clearly indicates that the addition of high content fillers can reduce the CTE of the epoxy matrix significantly, and generally high content of fillers in epoxy resin is possible to be produced with the addition of diluent.



Figure 5. The CTE and T_g values of epoxy composites, for both non-diluent and diluent systems.

3.4. Thermal stability

Thermal stability is performed to understand how the materials stand against the wide range of the temperature. Thermal stability of the epoxy composites for non-diluent and diluent systems is displayed in Figure 6. It is clearly that all of the epoxy composites showed a typical one step weight loss and they are quite stable at before 150°C. This is a good indication since the maximum temperature for a normal device is normally does not exceed more than this temperature. When we focus into the weight loss of the epoxy composites at 150°C (Table 3), we noticed that the weight losses of less than 1.5% are observed in all the epoxy composites. The weight losses in this stage are related to the moisture on the sample surface and some high volatiles substance that is existed in the resin. Weight losses of diluent systems are slightly higher than the non-diluent system at the same filler content. It is believed that the diluent can bring to the plasticizing effect and produces loosen network structure, thus, provide low thermal stability [24, 25].



Figure 6. Thermal stability of epoxy composites for both non-diluent and diluent systems.

4. Conclusions

The substrate materials were successfully prepared from mineral silica filled epoxy composites with ethanol, as diluent. The epoxy filled with 60vol% of mineral silica can be produced in diluent system compared to that of maximum filler content of 40vol% in non-diluent system. The epoxy composites with diluent system filled at 60vol% was showed relatively high dynamic flexural modulus and low CTE over the wide range of temperature. The addition of either non-diluent or diluent into the epoxy composites increases both its thermal stability and dynamic flexural modulus but reduces its CTE compared to the epoxy resins. Comparatively at a fixed filler loading, systems with diluent exhibited lower mechanical and thermal properties compared to the non-diluent systems, due to its lower crosslink density obtained from elastic modulus.

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