

Adhesion Evaluation on Low-Cost Alternatives to Thermosetting Epoxy Encapsulants

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Abstract—The thermosetting epoxy curing systems have been widely used as encapsulants in the electronic packaging industry. With the continual evolving of electronic product markets, material suppliers have been challenged to provide more options to meet the requirements of advanced, yet cost effective, packaging solutions. In this paper, two low-cost alternative materials have been investigated experimentally regarding their adhesion and reliability performance, and these have then been compared with the thermosetting epoxy systems. One of the materials is thermoplastic bisphenol A epoxy/phenoxy resin, and the other is an interpenetrating polymer network composed of an epoxy curing component and a free radical polymerizable component. Some formulations of the materials being studied could exhibit excellent adhesion, durability and application reliability. While reworkability is expected for these materials, they are promising as cost effective encapsulants for electronic packaging, and may be applied with appropriate processing techniques.

Index Terms—Adhesion, durability, encapsulant, epoxy resin, interpenetrating polymer network, phenoxy resin, reliability, reworkability, thermoplastic, thermosetting.

I. INTRODUCTION

ORGANIC polymeric materials have found extensive applications in the microelectronics packaging industry [1]–[3]. This has mainly been driven by their low cost compared to other packaging material options, while offering the performance and reliability required for most applications. For example, among the different plastic packaging encapsulants, underfill is of significant interest for flip chip assembly [4]–[6]. Due to the mismatch in coefficient of thermal expansion (CTE) between the silicon die, solder bump, and organic substrate, underfill reinforcement is essential in improving the integrity and reliability of the solder joints by up to an order of magnitude.

Although the underfill material/process has evolved from conventional to fast flow and/or snap cure, and no flow or wafer level versions [7]–[9], the chemistry for underfill has been based on thermosetting epoxy curing systems. The same holds true for epoxy molding compound, glob top, and die attach adhesive, etc. The necessary curing process could transform the formulation into infusible three-dimensional network. The cured epoxy materials provide chemical resistance, electrical

insulation, good mechanical property and adhesion to various components and thermal stability. Starting from the different kinds of oligomer resins and curing systems available, the epoxy chemistries demonstrate the unique capability for tailoring and processing. However, the disadvantages include a rather poor shelf life in the pre-mixed single package, a rather high modulus if lower CTE is intended by pre-filling with silica filler, a generally long cure cycle under typical curing condition, and also the issue of reworkability.

While electronic packaging has been advancing on an “exponential” pace toward increasing functionality and miniaturization, cost consideration has become much more important, especially for the consumer electronic product markets. Material suppliers must come up with cheaper, yet still competent, formulations for use in advanced electronic packaging. From the standpoint of polymeric materials, the category of thermoplastics could be of great potential to be employed, as the thermoplastic materials could basically avoid some of the disadvantages of their thermosetting counterparts, e.g., the curing process, lack of reworkability, etc. In this paper, we are exploring the possibility of using the thermoplastic materials and its combination with thermosetting materials [10] for electronic packaging applications, in terms of their adhesion, durability and reliability performance.

The thermoplastic epoxy/phenoxy resins based on poly(bisphenol A-co-epichlorohydrin), including the basic liquid resin (BLR) and the advanced solid resins of different molecular weight, and the so-called phenoxy resin of high molecular weight, were investigated and compared to the thermosetting epoxy resin system. The interpenetrating polymer networks (IPN) composed of thermosetting BLR component and thermoplastic free radical polymerization (FRP) component were also devised and studied. The results obtained so far indicate that both the candidates are promising for encapsulation applications in ultra-low cost next-generation electronic packaging.

II. EXPERIMENTAL

A. Materials

A series of epoxy resin oligomers based on poly(bisphenol A-co-epichlorohydrin) with two ends glycidyl-capped were designated as EPR (epoxy resin), followed by the nominal number average molecular weight (M_n), EPR377 through EPR6100 as listed in Table I. They were unmodified versions supplied by Aldrich Chem. Co. From the same source was the sample of EPRPH, which was a very long chain of poly(bisphenol A-co-epichlorohydrin), i.e., the so-called

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phenoxy resin, an actual thermoplastic polymer with nominal weight average molecular weight (Mw) of about 40 000 found by light scattering measurement (or Mn of about 8000). A combination of EPR6100 and EPRPH at a weight ratio of 3:4 gave the sample of EPR61PH with a calculated Mn of about 7000. The sample EPRRD is a mono-functional reactive diluent for epoxy resin from Shell Chem. Co., 4-tert-butyl phenyl glycidyl ether, under the trade name of Heloxy 65.

To minimize the difference between the chemical compositions of the thermoplastic and thermosetting epoxy resin systems, imidazole was used to act as a curing agent for the thermosetting ones. Imidazole, compared with other typical curing systems for epoxy resin, can be used at a relatively lower weight ratio against the resin part. As a curing agent, imidazole can give sufficient curing rate and distinctive degree of cure as well as high heat distortion temperature of the product [11]. A liquid imidazole, 1-cyanoethyl-2-ethyl-4-methyl-imidazole or 2E4MZ-CN, from Shikoku Chem. Corp. Japan, was used throughout the study at 4 phr of the epoxy resin, i.e., 4 parts by weight of the curing agent per 100 parts of the resin [12].

Both the solution and the hot melt approach were applied to the above-mentioned chemical systems in the test vehicle assembly for the adhesion measurement. The carrying solvent for the solution approach was 2-butanone or methyl ethyl ketone (MEK) from Aldrich Chem. Co., where a solid content of 30% by weight was always employed.

The free radical polymerizable component in the interpenetrating polymer network (IPN) samples was based on liquid mono-functional unsaturated monomers with certain free radical initiators. The FRP component would thus be thermoplastic after polymerization.

B. Instrumentation

The adhesion performance of the two candidate systems was characterized primarily with the die shear test. In either the solution or hot melt approach, about 10 to 20 small pieces of SiN passivated silicon die (2×2 mm in size) were assembled onto the same but larger wafer stand (15×15 mm) with the epoxy/phenoxy resins applied between the interface as adhesive. The solution assembly was performed at room temperature, and after assembly the test vehicles were allowed to stay at room temperature (RT) for 24 h; For the hot melt assembly, the operation was conducted at 175 °C, followed by cooling down to ambient temperature. The assembly was then subjected to drying/curing at 80 °C/125 °C/175 °C for 1 h/1 h/0.5 h, respectively. The IPN samples of BLR/FRP combinations were assembled for die shear testing at room temperature and cured with the appropriate temperature schedule corresponding to the free radical initiator used in the formulations (see details in Results and Discussion section).

The die shear tests were performed on Royce Instruments model 552 100 K at room temperature (RT) or at specified higher temperatures (below and above the Tg of the materials under study) using the hot stage monitored with the attached TPH14 temperature controller. In some cases, the test vehicle assembly first passed through an accelerated aging process, such as 85 °C/85%RH conditioning up to 1000 h or pressure cooker test (PCT) at 121 °C and 100%RH under 2 atmosphere

TABLE I
THERMOPLASTIC AND THERMOSETTING EPOXY/PHENOXY RESINS

Sample ID	Mn	Mw	Softening point, °C	As T.P.	As T.S.
EPRRD			Liquid	✓	
EPR377 (BLR)	377		Viscous liquid	✓	✓
EPR1075	1075		75~80	✓	✓
EPR1750	1750		88~95	✓	✓
EPR4000	4000		120~130	✓	✓
EPR6100	6100		130~140	✓	✓
EPR61PH	(7000)			✓	✓
EPRPH		40,000	Solid	✓	✓

T.P.: tested as Thermoplastic; T.S.: tested as Thermosetting.

pressure up to 96 h, before the die shear testing was conducted at room temperature.

Liquid-liquid thermal shocking (LLTS, -55~125 °C with 5 min dwell time) and air-air thermal cycling (AATC, -55~125 °C with 10 min dwell time) tests, both up to 1000 cycles, were also conducted on the materials under study as underfill for the assembly of 10×10 mm SiN passivated silicon die on FR-4 substrate coated with solder mask, where glass beads of 75 μ m diameter were loaded in the adhesive materials at 0.5 wt%. This was to simulate the typical die/substrate gap in flip chip assembly, and the appearance of underfill delamination or fillet crack was inspected by both the naked eye and microscopy.

III. RESULTS AND DISCUSSION

A. Epoxy/Phenoxy Resin: Solution Approach

To facilitate the application of all the interested materials to the die shear test vehicle assembly, we first resorted to the solution approach. Referring to Table I, the thermosetting systems studied in this section covered all cured products of EPR377, EPR1075, EPR1750, EPR4000, EPR6100, EPR61PH and EPRPH with the 2E4MZ-CN curing agent, while the thermoplastic systems included all corresponding epoxy oligomers without the curing agent. Specifically, one thermoplastic sample was the phenoxy resin (EPRPH) without the curing agent and was referred to as the pre-formed thermoplastic material; another was the EPRRD reactive diluent mixed with the curing agent, because of its mono-functionality, a thermoplastic material could be theoretically in-situ formed. The die shear strength data for the systems studied was shown in Fig. 1.

For the thermosetting systems of epoxy oligomer of Mn from 377 through 6100, the die shear strength exhibited a gradual increase and then leveled off with the increase of oligomer molecular length. This result can be attributed to at least two points. First, with the increasing chain length of the epoxy oligomer, there are more pendent hydroxyl groups along the backbone, and they could promote the adhesion by acting as the anchoring sites toward SiN passivated adherents. Secondly, with the decreasing crosslinking density for higher epoxy oligomer molec-

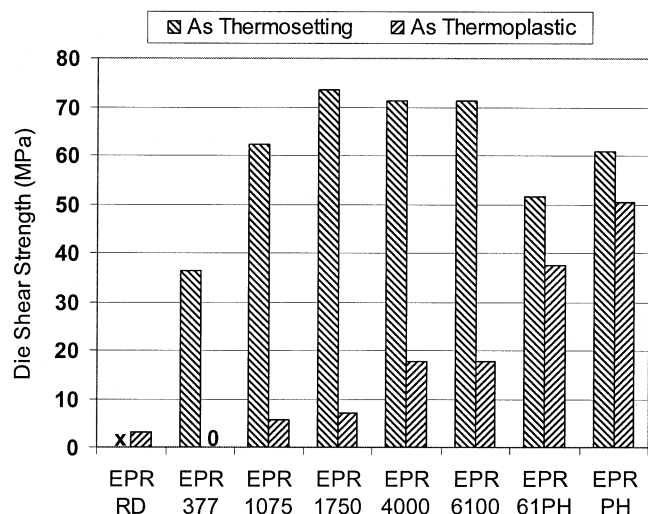


Fig. 1. Die shear strength at RT for thermosetting and thermoplastic epoxy/phenoxy resins, where X: N/A for EPRRD, not as a thermosetting material, and 0: zero for thermoplastic EPR377 (being liquid at RT).

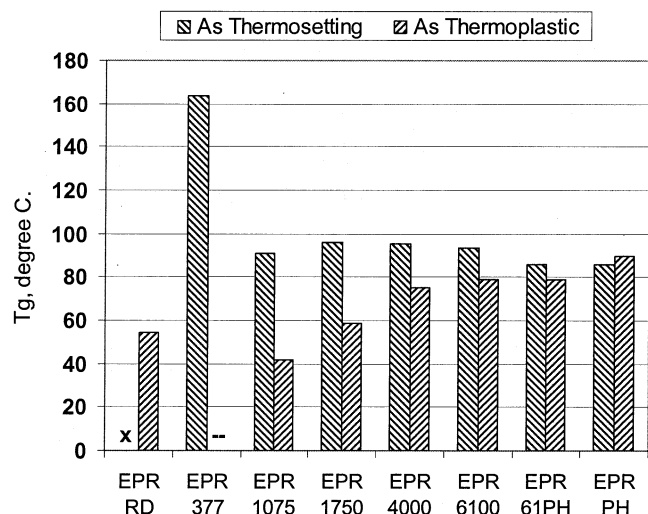


Fig. 2. Tg from modulated DSC run for thermosetting and thermoplastic epoxy/phenoxy resins, where X: N/A for EPRRD, not as a thermosetting material, and -: -18°C as measured for EPR377 (BLR).

ular weight, the role of the material flexibility and/or toughness could play a more important role for the die shear tests. The more flexibility/toughness of the cured system, the less residual stress after curing, and the more capability to prevent any delamination or crack from propagating. Thus, the adhesion data shown here resulted mainly from the enhanced anchoring effect from the hydroxyl groups as well as the increased flexibility/toughness of the cured matrix for the thermosetting epoxy resin from a higher molecular weight oligomer [12]. This was further supported by the Tg data for the thermosetting systems as depicted in Fig. 2, where under the current curing condition (the loading level of curing agent and curing program), the thermosetting EPR377 sample possessed a much higher Tg, while the rest of the thermosetting ones had only medium values of Tg.

The thermoplastic materials from epoxy resin oligomers of lower molecular weight typically demonstrated inferior die

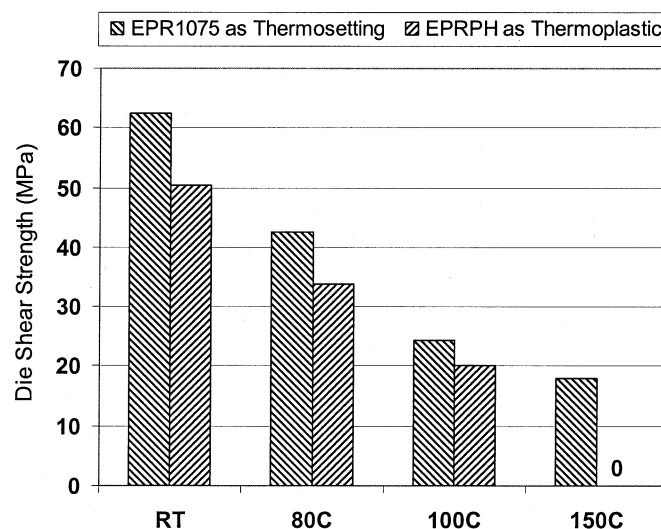


Fig. 3. Die shear strength for thermosetting and thermoplastic epoxy/phenoxy resins below and above Tg, where 0: zero for thermoplastic EPRPH at 150°C .

shear strength compared with those from the corresponding thermosetting systems, until we moved to a sufficiently high molecular weight of the oligomers. For example, the pre-formed thermoplastic phenoxy resin (EPRPH) exhibited somewhat higher die shear strength (>50 Mpa, which would typically be good enough for underfill encapsulation in electronic packaging) than the thermosetting basic liquid epoxy resin (EPR377). When combined with 2E4MZ-CN, it showed even higher die shear strength, but not comparable to the thermosetting systems from other lower Mn oligomers. This might be due to the curing agent used, because the phenoxy resin would be more readily reactive toward typical crosslinkers for hydroxyl functional resins, such as melamine, urea-formaldehyde, phenolics and isocyanate prepolymer, etc.

However, the reactive diluent EPRRD did not give much die shear strength as an *in-situ* formed thermoplastic material. This was reasonably normal because most common diluents could not give rise to high molecular weight product upon polymerization, especially for the mono-functional ones. The *in-situ* formed material only showed a very limited Tg value round 54°C as in Fig. 2.

B. Temperature and Aging Effect on Adhesion

For comparison purpose, the thermosetting EPR1075 (with the imidazole curing agent) and the thermoplastic EPRPH system were selected with the closest Tg value (91.1°C and 90.0°C , respectively) to understand the effects of temperature and temperature/humidity aging upon the adhesion durability.

As shown in Fig. 3, the die shear strength of both the thermosetting and thermoplastic materials decreased gradually with the increase of temperature. The thermoplastic phenoxy resin did not show any worse situation than the thermosetting one, even when the temperature was raised through their Tg. When the temperature went much higher than the Tg, the thermosetting EPR1075 still had some reasonably good die shear strength retained, while the thermoplastic phenoxy resin simply softened/melted to yield a zero value for die shear strength. Nevertheless, the high temperature of 150°C is rare for typical micro-

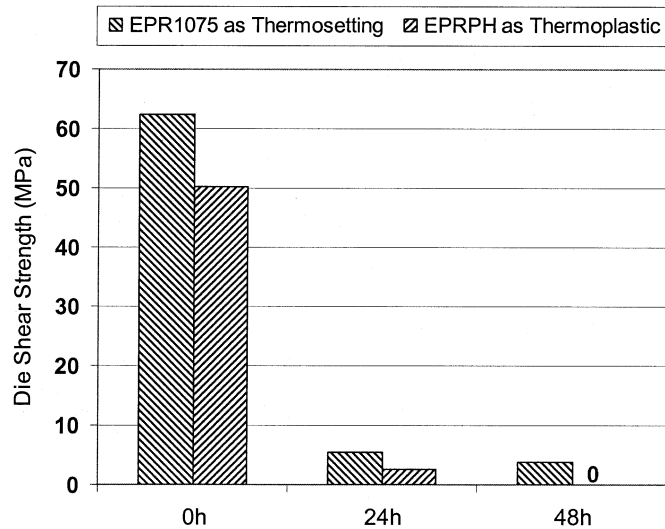


Fig. 4. Die shear strength at RT for thermosetting and thermoplastic epoxy/phenoxy resins after PCT (at 121 °C, 100%RH, 2 atmosphere) aging, where 0: zero for thermoplastic EPRPH after 48 h of PCT test.

electronic devices, especially for the low cost consumer product applications (normally $\ll 100^\circ\text{C}$).

The deterioration of die shear strength of the systems with pressure cooker test aging was plotted in Fig. 4. Both samples showed dramatic depletion of die shear strength after PCT aging for 24 h. The thermosetting material retained some positive die shear strength for further PCT aging time, while the thermoplastic one was completely consumed at 48 hours. Fig. 5 depicted the die shear strength of the systems along with the 85 °C/85%RH aging time. It appeared that the 85 °C/85%RH aging was much milder than PCT, and a sharp decrease in die shear strength happened well before 500 h under 85 °C/85%RH condition. Again the situation was comparable for the thermosetting and thermoplastic materials.

C. Epoxy/Phenoxo Resin: Hot Melt Approach

All the above-mentioned test vehicles were assembled using the solution approach, which may have some difficulty in several of the industrial applications. Thus, we are interested in exploring the possibility of assembly procedure by the hot melt method, which could also enable us to check whether the carrying solvent MEK could be driven out of the die/stand assembly interface (2×2 mm in area) following the specified drying/curing program.

It can be seen that, for the thermoplastic phenoxo resin EPRPH, both solution approach and hot melt approach for the current test vehicle assembly gave rather similar die shear strength (the right 2 bars in Fig. 6). For more information on processability, the thermosetting EPR6100 system by solution approach and the thermoplastic EPR6100 system by hot melt approach were also included. In short, both the solution and the hot melt assembly approach are of potential feasibility for both the thermosetting and the thermoplastic materials.

The experimental results obtained so far indicated that the thermoplastic epoxy/phenoxo resins of high enough molecular weight could exhibit comparable adhesion performance to the thermosetting epoxy systems. The adhesion durability from

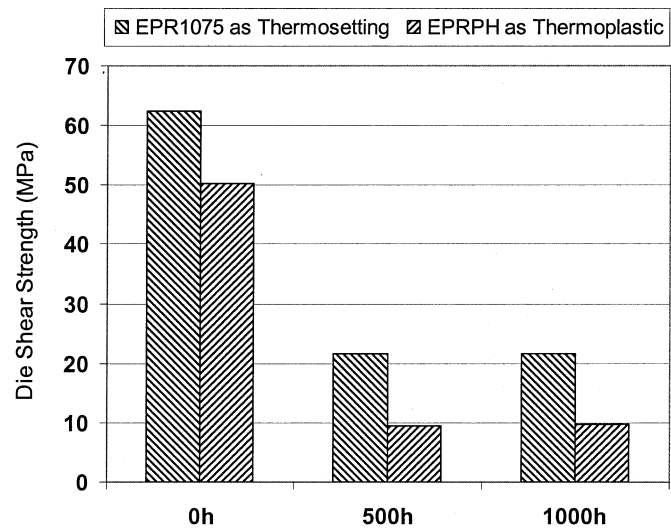


Fig. 5. Die shear strength at RT for thermosetting and thermoplastic epoxy/phenoxy resins after 85 °C/85%RH aging.

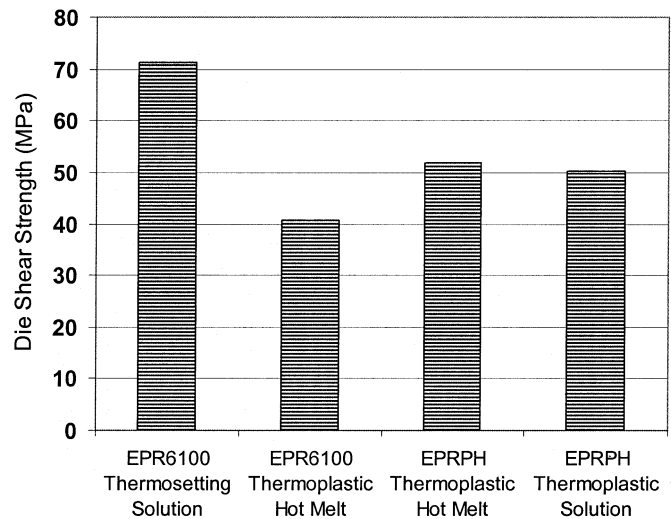


Fig. 6. Die shear strength at RT for thermosetting and thermoplastic epoxy/phenoxo resins with solution or hot melt assembly approach.

the thermoplastic systems could also be expected based on the results from temperature, 85 °C/85%RH, and pressure cooker aging tests. Such thermoplastic epoxy/phenoxo resins may find applications as in molding compounds for transfer molding and other molded encapsulation process (similar to hot melt), while the solution formulation may possibly be used for adhesive applications.

D. BLR/FRP IPN Samples: 100% Reactive Liquid Encapsulant

Considering the excellent adhesion normally provided by the epoxy resin, we would still employ the thermosetting BLR curing system in future formulation. Nevertheless, to formulate advanced encapsulants with lower cost but noncompromising or even-surpassing application performance, e.g., reworkability feature, we may take advantage of the interpenetrating polymer networks. The IPN concept of combining the thermosetting BLR component and the thermoplastic FRP component into a single compound could result in quite different characteristics from the epoxy/phenoxo resin systems as discussed above, i.e.,

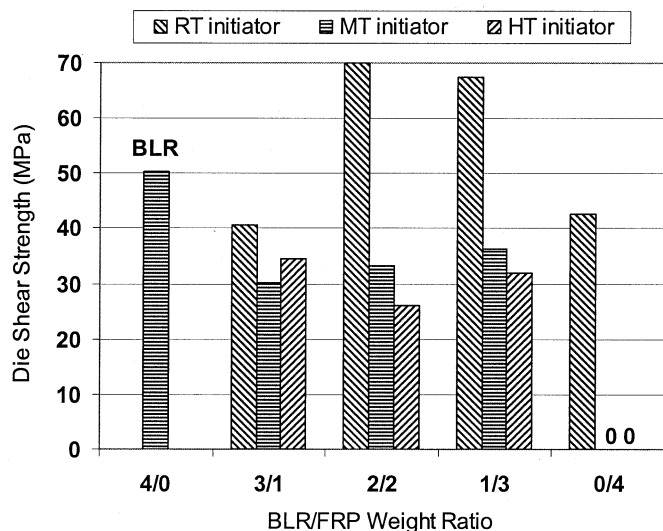


Fig. 7. Die shear strength at RT for BLR/FRP IPN formulations with various component weight ratios and free radical initiators, where 0: zero die shear strength for pure FRP component with MT or HT initiator.

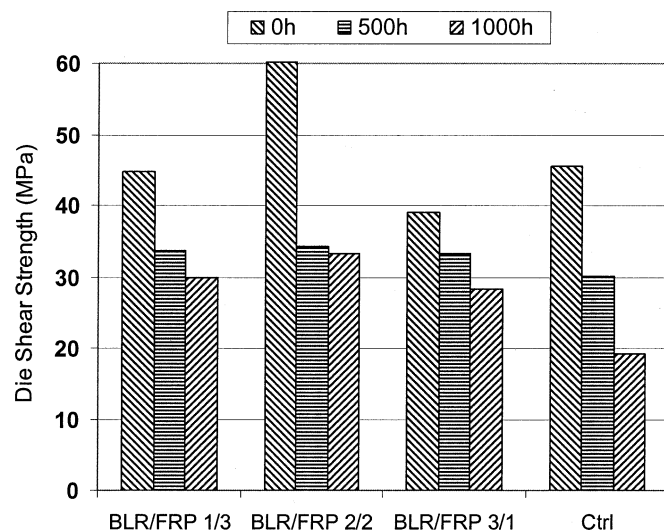


Fig. 9. Die shear strength at RT for BLR/FRP IPN formulation with various component weight ratios and RT free radical initiator after 85 °C/85% RH aging.

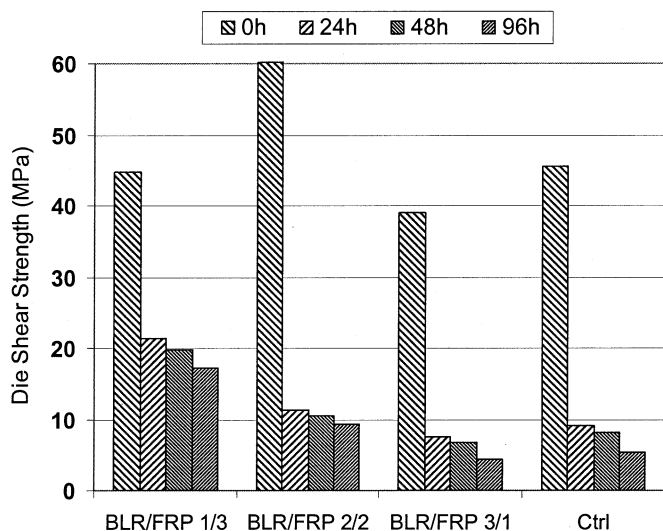


Fig. 8. Die shear strength at RT for BLR/FRP IPN formulation with various component weight ratios and RT free radical initiator after PCT (at 121 °C, 100% RH, 2 atmosphere) aging.

elimination of solvent use vs. the solution approach, and low viscosity of starting materials from liquid monomer/oligomer vs. highly viscous hot melt. All these IPN formulations could initially render to be drop-in substitutes for the current complete epoxy- based encapsulants and feasible with typical liquid dispensing process.

To qualify the adhesion performance of the BLR/FRP IPN samples, EPR377 was combined with a mono-functional unsaturated monomer at various weight ratios (4/0, 3/1, 2/2, 1/3, 0/4). 2E4MZ-CN was always used for the in-situ BLR curing, while one of the following four free radical initiators was employed for the FRP component, a room temperature (RT, 25 °C), a medium temperature (MT, 70 °C), a high temperature (HT, 120 °C) or a UV sensitive initiator.

When moving from the RT to MT and to HT initiator for the FRP component, the curing temperature for the IPN samples went up accordingly, but the die shear strength decreased for

TABLE II
THERMAL RELIABILITY TESTS ON CANDIDATE FORMULATIONS APPLIED AS UNDERFILL ENCAPSULANTS

Sample	Tested as	Assembly by	Delamination or crack after 1000 LLTS cycles	Delamination or crack after 1000 AATC cycles
EPR6100	Thermoplastic	Hot melt	None	None
EPR6100	Thermosetting	Solution	None	None
EPRPH	Thermoplastic	Solution	None	None
BLR/FRP IPN	1/3 weight ratio	Dispensing	None	None
BLR/FRP IPN	2/2 weight ratio	Dispensing	None	None
BLR/FRP IPN	3/1 weight ratio	Dispensing	None	None

most composition ratios studied, as shown in Fig. 7. This could be explained by the fact that, the higher temperature needed to start the free radical polymerization with MT and HT initiator, the more the evaporation of the free radical monomer became an issue (there were no measurable die shear strength for pure FRP component with MT and HT initiators), while we could ignore the evaporation of BLR oligomer and the temperature effect on its curing with 2E4MZ-CN curing agent.

Similar to the RT free radical initiator, the BLR/FRP IPN samples at 1/3 weight ratio with the UV initiator for the FRP component (UV polymerization took place at RT) could exhibit rather high die shear strength, 68.4 MPa versus 67.4 MPa from RT initiator, both well above 50 MPa die shear strength which is typically considered necessary for underfill encapsulation applications.

In order to evaluate the adhesion durability of the BLR/FRP IPN samples against temperature/humidity aging, the IPN samples from Table II (see details in next section) were benchmarked with a typical commercially available thermosetting epoxy based sample (ctrl), as displayed in Figs. 8 and 9. It can be seen that most of the IPN samples studied revealed

better adhesion retention than the ctrl sample, which was quite encouraging.

E. Reliability Test: Application as Underfill

Thus, so far we have observed that both the thermoplastic epoxy/phenoxy resins and the BLR/FRP IPN formulations could be potential alternatives to thermosetting epoxy systems for encapsulation uses regarding their adhesion and durability. We are further interested in their application reliability performance, for example, when they were used as underfill encapsulants, adhesion of encapsulant toward die and substrate as well as their overall mechanical properties could be examined with LLTS and AATC tests. The underfill samples in Table II were prepared from the respective candidate systems, where formulation techniques were applied to the IPN samples. The results from the reliability tests were positive and exciting, no delamination or fillet crack was found with all the samples after 1000 thermal cycles of both LLTS and AATC. This indicates that these alternative encapsulants are highly potential for electronic packaging applications, even for assemblies of dies of relatively larger size (10×10 mm silicon used in the experiment) on cost-favored FR-4 substrate.

IV. CONCLUSION

In an attempt to identify potential alternative polymer materials to thermosetting epoxy resin systems for encapsulation applications in electronic packaging, the adhesion performance of thermoplastic bisphenol A epoxy/phenoxy resins as well as interpenetrating polymer networks composed of basic liquid epoxy curing component and free radical polymerizable component was investigated in terms of the die shear strength. The effect of temperature and temperature/humidity aging upon the adhesion durability was also studied. Both the candidate systems exhibited comparable or surpassing adhesion and durability against the thermosetting epoxy systems. The results from the initial application reliability test was positive too. While those materials are fully or partially thermoplastic in nature, the reworkability feasibility could thus be expected [13]. The knowledge obtained herein could provide necessary starting information and more options toward material development for next generation ultra-low cost microelectronics packaging, and would be of significant interest for potential encapsulant formulations, such as underfill, epoxy molding compound, glob top, and die attach adhesives, etc.

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