Epoxies for OptoElectronic Packaging; Applications and Material Properties

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Abstract

Epoxies are used in the packaging and assembly of three opto-electronic devices, including Fiber Optics, LEDs, and LCDss. In fiber optics, you need epoxy for the cable and for the opto-packaging. The fiber optic components are usually packaged in hybrid technology, which mean several micro-electronic grade joining materials will be used. For LEDs, the silver epoxy is needed for both electrical and thermal properties. Optically clear epoxy is used for the potting, molding, or encapsulation of the LED. In LCDs, there are many different applications of epoxy of which 2 are presented. These include silver filled for interconnecting the ITO conductive layers to the circuitry on the substrate, while the second is the optical epoxy for making the glass sandwhich. This paper will discuss the epoxies which are used, the key design attributes which make them ideal, and the processing of them for assembly and manufacture. Analytical techniques for characterization and optimization of the epoxy properties will be discussed. Material properties such as Tg, outgassing, thermal conductivity, thermal resistance, hermeticity, strength, and fluorescence will be the focal point of the discussion.

Key words; LEDs, LCDs, Fiber Optics, optical epoxies, glass transition, fluorescence

1.0 Introduction

It is widely known about the use of epoxies for microelectronic packaging. Since they were introduced greater than 30 years ago, they have enjoyed success in many adhesive applications including die-attach [1-2, 4-6], bonding SMDs, [3] substrate attach, and hermetic lid sealing [7]. These packages, also known as hybrids, were primarily used in MILITARY / AEROSPACE applications for Rf and Microwave communications devices.

More recently, Fiber Optic packaging has adopted some of the basic principles as described above with the MIL and AERO devices. Unlike coaxial cables moving electrons down copper wires, fiber optic cables are messengers of information by using photons of light. Light is both a wave and a particle, so it is very useful as a source of heat, and illuminator of objects [8], but also makes it hard to control. The paper describes a typical fiber optic grade epoxy, and the material properties that makes it ideal. Data including DSC, Tg, CTE, hermeticity and percent cure is presented and discussed.

Unlike above, LEDs typically do not follow the same microelectronic packaging structure using hybrids. Rather, lower cost packaging is commonly employed. For LED packaging, 2 epoxy applications will be discussed. The first and most obvious is the silver epoxy for the die-attach. Not only does it provide the electrical pathway, but it is needed for the heat-sinking as well. Thermal resistance calculations are shown, and thermal conductivity is discussed. The 2nd application is the encapsulation or protection of the LED. It might be described as a clear, colorless, non-yellowing encapsulant. Fluorescence data is presented about epoxies "tending to yellow" under light- aging conditions

Lastly, LCD assembly uses many different classifications of epoxy resins for joining, protecting and sealing. Two applications of epoxy will be discussed. Like LED applications mentioned above, the LCD needs silver epoxy for electrically interconnecting the LCD to the circuit. Conductivity measurements are presented and discussed along with the role of ITO metallization. Optical epoxy resin is used for laminating and gluing the many glass layers used in the LCD sandwich. It must be clear and colorless due to it being in the light pathway. Results are shown that material properties are greatly affected by mix ratio.

2.0 Experimental

2.1 Fiber Optics

The adhesive for the fiber optics portion of the study may be described as a 2 component, high Tg, , catalytically cured epoxy resin adhesive, obtained from Epoxy Technology, Inc. All curing was done in a Scientific Products Model DK-43 convection oven. All samples for DSC were kept to 10-40 mg mass. The DSC was TA Instruments

Model 2010. Scan rates for DSC were 10 C / min for uncured epoxies, and 20 C / min for all cured samples. For outgassing measurements, the TA Instruments TGA 2950 was used, with 20 cc/min purge of air gas. All outgassing samples were controlled in the 10-40 mg mass. For lap shear specimens, ASTM D1002-01 procedure was used. Tensile Lap Shear was measured by Instron Corp. Model 1130 at 23 C +/- 2 C. The CTE was plotted using Perkin Elmer model TMA7, while the storage modulus was plotted using Perkin Elmer DMA7. The former used samples that were 0.250" diameter and 40-60 mils height, while the latter used samples that were 18mm x 3 mm x 1mm tall. The TMA used static force of 20 – 50 mN, while the DMA had both static and dynamic stresses applied. Both TMA and DMA used a 5 C / min scan rate. A hermeticity plot of the adhesive seal has been included, to illustrate the importance of Tg. However at this time, it is not known the make or model of the leak detector.

2.2 LEDs

For the LED portion of the study, thermal resistance was calculated from the silver epoxy die attach, and fluorescence of the optical epoxy encapsulant was measured. A TO-3 package test vehicle was chosen for thermal resistance. It used bipolar transistor chips from Motorola, part # 2N-3055. The die were 120 mil and 140 mil. Six commercially available silver filled epoxies were chosen for the study against solder controls. The bond-line thickness (BLT) of the die attach was varied. A burn-in process of 150 C / 1000 hrs was chosen. Thermal resistance from junction-to-case, (θJc) was measured and plotted during the burn-in. The measurements were obtained using IR Thermograph from Agema Thermovision 782. A color coded temperature gradient, accurate to 0.1 C, was produced and recorded on the thermogram [9].

For LED encapsulants, the relative fluorescence was measured for more than a dozen optical epoxies. An argon laser at 488nm was used to excite the fluorescence of the epoxies in a cuvet at room temperature. The cuvet was made from methacrylate by Fisher, catalogue # 14-386-21. The fluorescence intensity was detected by a photomultiplier, Hamamatsu R928, using 2 filters, one at 488 nm notch filter from Kaiser Optics and 535 short cut-off glass filter. The data of fluorescence intensity was displayed and stored in a computer after digitizing with an A/D board. [10]

2.3 LCDs

For LCD glass laminations, the optical epoxy performance was quantified by varying its mix ratio and cure schedule. The cure was done using the Scientific Products Model DK-43 convection oven, the same one used for the fiber optics portion of the paper. The DSC and TGA were also the same, TA Instruments DSC 2010 and TGA 2950, respectively speaking.

For the LCD die attach, 4 silver epoxies were tested, independent of the LCD circuit. It was found that the silver epoxy electrical connection to the ITO was being lost at the cantilevered edge only; all 3 other ITO connections were fine. Therefore, bus bar stripes of silver epoxy were made on dummy glass substrates and blank FR4 boards. The stripes were about 0.2 mm long, 0.01 mm tall, and the length was 15 cm on FR4 while 2.54 cm on glass. A Keithly 197A Autoranging Microvolt Ohmmeter was used with Trebor Instruments Corp 4-pt probe for volume resistivity calculations on substrates. The prior mentioned DSC was used to determine Tg.

3.0 Discussion of Results

3.1 Fiber Optics

Table I. shows the results of the cure study for the fiber optic adhesive. Basically, the goal was to find which temperature and time combination is the best to make 100% full cure using DSC thermodynamics. The cure temperatures chosen were 80 C, 100 C, 120 C, and 150 C. At each temperature, the epoxy was allowed to cure at 30 minutes, 1 hr, or 2 hrs total dwell. Some mixed cured schedules were used. Some 15 different cure cycles were tested for completeness of cure and the final Tg.

Cure condition	Residual Exotherm (J / g)	Peak exothermic Temperature (C)	% NOT cured (% unreacted)	Calculated Tg (C)
80 C / 30 minutes	328.5	134.6	65	87
80 C / 1 hr	253	137.4	50.7	87
80 C / 2 hr	212.3	138.8	42.5	87
100 C / 30 minutes	83.1	155.2	16.6	103
100 C / 1 hr	86.14	155.5	17.3	103
100 C / 2 hr	51.94	155.1	10.4	103
120 C / 30 minutes	20.16	158.2	4	110
120 C / 1 hr	8.67	159	1.7	110
120 C / 2 hr	0.8	155.6	0.2	110
150 C / 30 minutes	N/A	N/A	N/A	120
150 C / 1 hr	N/A	N/A	N/A	119.4
150 C / 2 hr	N/A	N/A	N/A	116.4
80 C / 1 hr + 120 C / 1 hr	19.9	158.4	4	110
80 C / 1 hr + 150 C / 1 hr	N/A	N/A	N/A	129.4
80 C / 1 hr + 120 C / 1 hr + 150 C / 1 hr	N/A	N/A	N/A	128.8
150 C / 15 second hot plate cure	N/A	N/A	N/A	137.3
totally uncured exotherm	498.9	118		

Table I. DSC cure results of Fiber Optic Adhesive

From the table you can notice that all cures < 120 C yielded incomplete curing. In general, any combination of cure that used 120 C or greater, yielded 100% cure. The step cure of 80 C + 120 C post cure did not really influence the cure compared to using 120 C cure alone. The residual exotherm is the amount of epoxy that still needs to become cured. The cure percentage is expressed by the ratio of [

residual exotherm / totally uncured exotherm]. Basically, the larger the residual exotherm, the more un-cured the epoxy. The peak exothermic temperature is the temp at which the remaining un-cured groups will become polymerized finally yielding 100% cure.

Plotted in Figure 2 are the actual DSC curves which have been summarized in Table I. The curves show the region where the epoxy is endothermic or exothermic. Epoxy resin chemistry, by definition, is an exothermic process which is non reversible polymerization. It can be thought of as

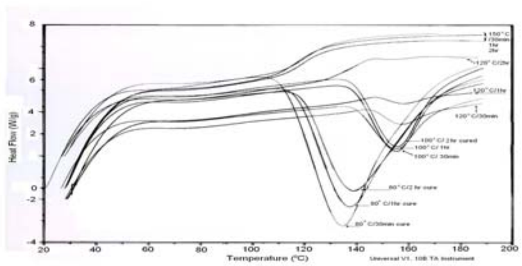


Figure 2. Actual DSC overlay curves from Table II. revealing exothermic regions, and Tg,

kinetic potential, or a simple reaction coordinate. Therefore, any portion of the curve which is exothermic, represented by the curve heading in the "down direction", means that range of temperature is the un-cured epoxy groups. Conversely, any portion of the curve which is represented by endothermic behavior, represented by traces in the "upwards direction", means the epoxy has been kinetically favored meaning good cure.

A comparison of the 80 C, 100 C, and 120 C curves from Figure 2 shows two trends about

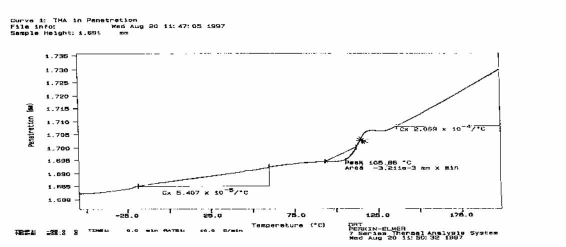


Figure 3. TMA /CTE curve, showing Tg region

increasing the cure cycle; namely the exothermic region becomes smaller in magnitude, and also it falls at a higher temp range. This was also expected, and it supports the rule of thumb; " the higher the cure temp, the more complete cure; or the 2nd trend of the longer the cure time, the better the cure"**.** Figure 3 shows the TMA, or CTE curve for the fiber optic adhesive. Notice the Tg range near 120 C, which agrees very well with the Tg data obtained from DSC shown in Figure 2.

Figure 4 shows the DMA curve, or modulus plot versus temperature. The DMA curve shown suggests Tg near 120 C, and this Tg regions also agrees well

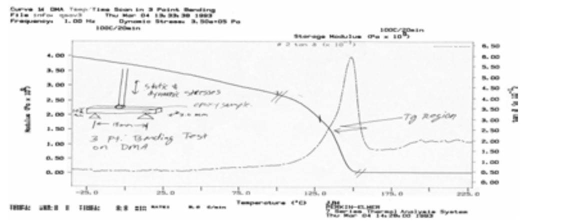


Figure 4. DMA curve plot of modulus and Tg

with the DSC and TMA curves shown. Figure 5 is a hemeticity plot of the adhesive. It reveals most loss of hermeticity near 125 C, which agrees with Tg ranges from Figures 2-4. A loss of hermeticity (but still very hermetic in terms of the Mil-STD 883, TM5011 definition) is expected

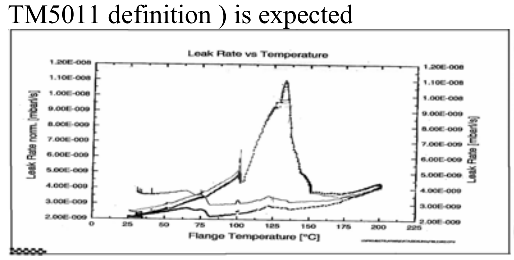


Figure 5. Leak Rate versus Temp showing Tg

near the Tg range, but the curve also shows that it is fully recoverable above and below the Tg. Thus in summary, the Tg could be explained in simple terms; Tg by DSC is recording thermodynamic changes or relaxation of the polymer chains; Tg by TMA is Tg by recording dimensional changes; Tg by DMA is Tg from physical deformation (or softening); and lastly Tg obtained from leak-detector values from hermetically sealed microelectronic package.

Table II shows the effect of cure on lap shear strength. The adhesive was allowed to cure at 80 C, 120 C, and 150 C for 1 hr duration in each case. Notice that the strength of 80 C cure compared with 150 C cure is about ½ the strength. This agrees very well with the DSC cure results of Table I where it was found that 80 C / 1 hr cure was roughly 50.7% not reacted.

Table II. Lap Shear Strength versus Cure

	cured 80 C / 1 hr	cured 120 C / 1 hr	cured 150 C / 1 hr
Lap Shear Strength (average, mix ratio of 10 to 1)	1280 psi	1533 psi	2533 psi

Table IV. is a summary of adhesives for fiber optics. Quite simply, the adhesives used for the packaging of fiber optic components, are the same traditional ones that have been used in military type of hybrids. I agree with Tom Green's guest editorial that an opto-device "is nothing more than a hybrid with a light pipe" [11]. The assembly and test industry of opto-devices has not reached its maturity level, as there are sub-micron or "nano" level alignments needed. To achieve the nano level, hand or manual assembly is being used during this opto-infancy, so manufacturing or automation challenges will need to be addressed when the economic situation presents itself. One groups effort says that "packaging and assembly still dominate the overall cost of any fiber optic device, largely because fiber optic alignment and attachement (i.e. nano level) are difficult and time consuming" [12]. A second group has developed a novel low cost opto-package using premolded plastic. [13] These lower cost packages will compete against the traditional hybrid ones as mentioned by Green, and reinforced by Dr Truzzi who similarly claims that "butterfly [and mini-DIPs] are still the most poplular opto-electronic package choice", and that they derived from military applications using coaxial connectors such as SMA or SMP, or K-types for the electronic signal entering / exiting the package. [14]

Table IV. Fiber Optic Grade Adhesive Classification

Adhesive Type	Comment
optical	Optical adhesive for light pathway, nano align, alignment fiber, lens, diodes, coupling of light
hemetic seal	non solder, non eutectic, optical seal, keep out moisture from package
lid seal	MIL / Hybrid, traditional micro-electronic grade
substrate attach	MIL / Hybrid, traditional micro-electronic grade
die-attach	for LD-PD chips, traditional MIL-microelectronic grade
thermal management	TEC's, LD, heat sinking, traditional MIL-microelectronic grade

When designing an opto-package, there are many challenges to consider, according to R. Irving. [15] Challenges like thermal, electrical, and optical design need to be met. Thermal challenges are common with laser diode packaging, which use TECoolers, and the design should use thermally conductive adhesives between the die, TEC, and package. One laser diode design does not use butterfly or DIP packages mentioned above, but rather uses hybrid similarities from TO-can packaging. [16]. For electrical challenges, one must choose how the electrical leads will pass through the package walls, while still being hermetic. Figure 5 supports the use of adhesive, whereas higher cost packages might use o-ring solder seals. The similar argument could be said about optical challenges, where light must get into, through, and out of the package, and inside the package it needs to be coupled correctly to fibers, lenses, and diodes, all at nano level. Figure 6 shows the hermetic optical alignment seal that is common in fiber optic packages.

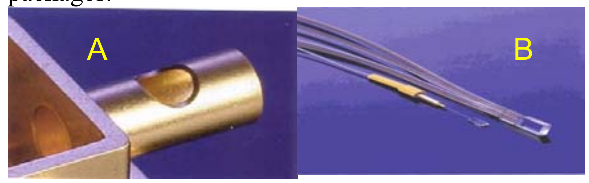


Figure 6. Examples Hermetic Optical seal. A. Fiber Feed –through seal in "opto-pipe". B. 48 F/O V-groove array glass seal.

3.2 LEDs

The research was done in 2 areas. The first was to find high thermally conductive silver filled epoxies that can handle high power LEDs, while the second area was to find clear encapsulants that would not yellow with time.

TableV. Thermal Resistance Measurements and Thermal Conductivity Calculations vs. Solder

Epoxy	Thermal Resistance (C / W)	Back Calculated Thermal Conductivity (W / m-K)
A	0.33	14.2
B	0.31	15.1
C	0.4	11.7
D	0.5	9.4
E	1.45	3.2
F	1.38	3.4
solder	0.3	15.6

It was widely reported in the LED 2002 Conference about the high power requirements for lighting technology. [17]. Six silver filled epoxies, obtained from a few common vendors, were analyzed and tested against solder controls. The best silver epoxy would be the ones that have the lowest thermal resistance compared to solder. Die attach using solder, in theory, would be the best heat sinking, since it is purely metal alloy, rather than polymer like epoxy. The results are shown in Table V.

Solder die attach turned out to be the lowest thermal resistance which was expected. Comparing it to epoxies A and B, it only had 0.03 C and 0.01 C, cooler improvement, respectively speaking. It can be said that epoxies A and B have nearly the same thermal conductivity, ***in package***, as the solder bond. The thermal resistance is plotted as a function of adhesive versus 1000 hr bake-out at 150 C. The results are shown in Figure 7. In general, the thermal resistance increased with time, which was expected. It is postulated that during burn-in, hydrocarbon materials are outgassed, which contribute to voiding or pin-holes, which will impede the thermal transfer. [18]

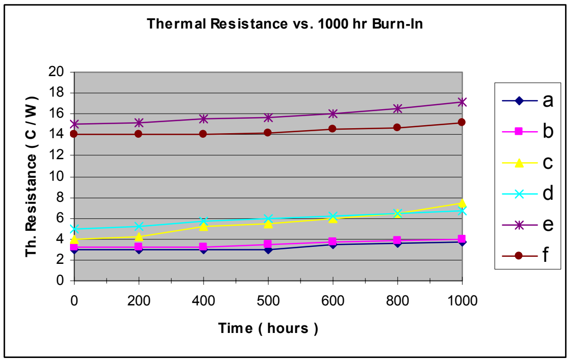


Figure 7. Thermal Resistance vs. 1000 hr Storage

OLEDs, or organic LEDs, are made from a plastic technology, rather than silicon or traditional semiconductor materials. They can offer more advantages from LED / Displays like; lighter weight printed on plastic; lower temp process means lower cost; they can be flexible for roll-up applications for space saving or transport; and they produce brighter images at less power. [19] The brighter image is enabled by the OLED producing more visible light, while filtering out IR light from glowing red sources. But more brightness of visible light, may cause the clear OLED (or high power LED) encapsulant to become discolored into yellowish tinge.

For the best products not to yellow, fluorescence of more than a dozen epoxies was measured. The cured and un-cured epoxies were irradiated with 488 nm light, and the fluorescent emissions were collected. Table VI. is a summary of the results.

Table VI. Relative Fluorescence Intensities

epoxy	part A resin	part B cure agent	cured
water	3.94	NA	NA
1	472	7.14	400
2	325	209	233
3	430	7.8	390
4	171	17	170
5	237	740	NA
6	276	446	833
7	646	10	NA
8	287	saturated	saturated
9	148	411	530
10	NA	8.7	NA
11	255	saturated	saturated
12	345	saturated	saturated
13	405	saturated	saturated
14	400	NA	saturated
15	125	NA	NA
16	100	NA	700

The data above shows that some epoxy cure agents can be as clear and colorless as water, and tend not to yellow. Epoxy sample 1, 3, and 10 fall into this category. It is also interesting to note that most part B's studied tended to be saturated, or of very high fluorescence. If the part B was high, then the cured product similarly speaking was high value. The part A's were all capable of being measured, and some trends could be made among them. Generally speaking, the lowest part A values, did not make the lowest cured values, since the part B was too high, represented by samples 5, 6, 8, and 11. Sample 16 was very low un-cured, but after UV exposure, it became high fluorescent.

3.3 LCD's

The focus of the study was 2 fold; first it was to find the best properties of optical adhesive for laminating the stacks of glass in LCD sandwich, and the second was to evaluate 4 silver epoxies for best electrical connection with ITO. A typical LCD schematic is shown in Figure 8.

The epoxy used to bond the glass plates together is described as a 2 component, optically clear and colorless adhesive capable of laminating, bonding, sealing, and potting any portion of the LCD. It was also epoxy #4 from the fluorescent portion of the study summarized in Table VI. The mix ratio was varied to simulate what would happen if static mixing was incorrect during automated dispensing and manufacturing. The results are presented in Table VII. The control mix was 100:35 b/w, and we decided to see the trends at lower mix ratios of 100:28 and 100:25 respectively.

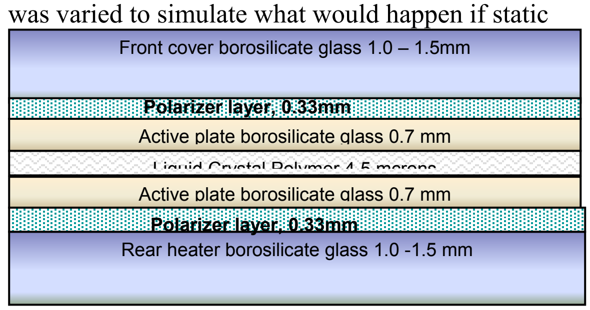


Figure 8. Schematic of LCD glass sandwich

Table VII. Mix Ratio vs Properties for LCD Epoxy

	100/35	**100/28**	**100/25**
viscosity (cps)	287	410	471
Tg (C)	80	64	57
Outgass% at 200 C	0.008	0.12	0.38
TGA 10% weight loss (C)	354.7	349.8	350.7

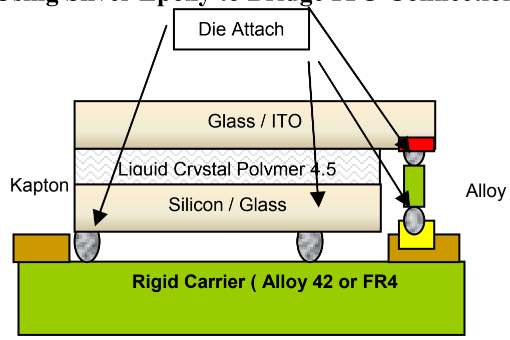
The data shows a few trends. First of all, the less cure agent yields the higher the viscosity. This makes sense, because the part A resin is like honey, while the part B is like water. The less part B added makes more high viscosity mixture. This would affect the wicking and capillary flow needed for LCD sandwhich. It was also shown that the Tg decreased with less part B, while the outgassing increased. We can theorize that part B is the "limiting reagent" while the unconsumed part A resin is being outgassed. The unconsumed part A resin could be reasoned as suppressing the Tg. The degradation temperature, defined as 10% weight loss, did not show any trends, however.

Table VIII. Results of 80 C Cure Study; Ag Epoxy for ITO Electrical Connection. VR and Tg Data.

epoxy	VR when cured 80 C / 4 hrs	VR 80 C/ 4 hrs + 16 hrs post cure	Tg 80 C / 4 hrs	Tg 80 C / 4 hrs + 16 hrs post cure
A	700 ohms glass 52 ohms FR4	83 ohms glass 31 ohms FR4	63	73
B	14 ohms glass 12 ohms FR4	<1 ohm glass <1 ohm FR4	35	38
C	< 1.0 ohms glass < 1.0 ohms FR4	<1 ohm glass <1 ohm FR4	43	49
D	500 ohms glass 1300 ohmsFR4	300 ohms glass 950 ohms FR4	NA	NA

Table VIII. shows the results of the electrical investigation. Since the application was for a micro-display using plastic housing, the cure temperature was chosen not to exceed 80 C. Four silver epoxy candidates were chosen for the investigation. The final product selected was the best balance of electrical conductivity with the highest Tg possible. A cure of 80 C / 4 hrs may or may not give the best Tg, so it was decided to see what happens with 16 additional hours of post-cure. Conductivity and Tg results are presented above. Product A was known to give the highest Tg, but we were unsure of its electrical conductivity with a 4 hr cure. The results show that 16 hrs post cure not only increases the Tg, but also makes better Volume Resistivity, which was expected. Epoxy sample C had the lowest VR condition which was expected, but we were skeptical about the Tg value. Comparing epoxy C to B, we had improvement in VR and higher Tg. Epoxy sample D was eliminated from the study due to very bad VR condition, and the Tg measurements were dropped from the study. Therefore, we chose epoxy sample C as the best product for LCD die attach electrical connection to ITO. The schematic of typical LCD assembled to PCB is shown in Figure 9.

Figure 9. Schematic of LCD Die Attach to PCB Using Silver Epoxy to Bridge ITO Connection



4.0 Conclusion

The paper focused on 3 applications of optical epoxies. We discussed Fiber Optic materials and packaging, LED die attach and encapsulation, and lastly LCD lamination and die attach to ITO.

For fiber optics, a cure study was presented showing % cure and Tg. The Tg was confirmed by DSC, TMA, DMA, and a hermeticity plot of leak-detector. The lap shear at 80 C / 1 hr was about ½ the full strength, which agreed with the DSC results of 50% not cured.

For high power LEDs, thermal resistance measurements were presented alongside solder controls. The soldered attach devices were not any cooler than the epoxy die attach. Calculated thermal conductivity of a few silver epoxies was the same thermal conductivity as solder. More than a dozen LED encapsulants were tested for relative fluorescence. Some trends were discovered about which resins and cure agents tend to become yellow. Yellow tinge encapsulants should be avoided in high power, bright LEDs.

For LCD packaging, 2 schematic cross sections are shown. The optical epoxy for the LCD lamination sandwich will have varying material properties affected by mix ratio accuracy. Four silver epoxies were tested for best electrical connection to ITO using cure not to exceed 80 C. A final selection could be made for a micro-display.

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