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# Evaluating the dimensional stability of optelectronic adhesives

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# EVALUATING THE DIMENSIONAL STABILITY OF OPTOELECTRONIC ADHESIVES

by

Thomas R. Daugherty

A Thesis

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of Lehigh Unversity

In Candidacy for the Degree of

Masters of Science

in

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May, 2008

# CERTIFICATE OF APPROVAL

This thesis is accepted and approved in partial fulfillment of the requirements for the degree of Master of Science in Polymer Science and Engineering.

May 2, 2008
Date

Thesis Advisor,

Prof. Raymond A. Pearson

Head of Polymer Education Committee,

Prof. James E. Roberts

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# Evaluating the Dimensional Stability of Optoelectronic Adhesives

*by*
*Thomas R. Daugherty*

## ABSTRACT

The viscoelastic behavior of four adhesives used to assemble optoelectronic packages has been characterized in terms of a distribution of relaxation times and the strength of the relaxations. The effect of temperature on the viscoelastic behavior of these materials was found not to be rheologically simple but could be easily modeled by allowing the distribution of relaxation times to vary with temperature. In addition to characterizing the adhesives, the performance of the adhesives were evaluated using test vehicles and the dimensional stabilities were ranked. The range of adhesive bond gaps from 5 to 200 um was examined for dimensional change resulting from environmental aging. The thermally cured adhesive was the most stable and the UV cured adhesives displayed a range of behavior.

## 1. INTRODUCTION

The dimensional stability of adhesive bonds in optoelectronic packages is of paramount importance [1]. The wear-out mechanism of optoelectronic packages often involves the movement of optical components that can greatly diminish the optical signal. Components joined using adhesives are often at risk. Therefore, choosing the appropriate adhesive (s) is crucial to ensure reliability.

The usual methodology for choosing an adhesive is based on previous experience or manufacturer's recommendations and then building some prototype optical systems to evaluate performance. Adhesive selection and prototype designs are then optimized until acceptable performance is achieved. Often accelerated aging techniques are employed to assist in the optimization. Such a methodology is expensive and time consuming but is also a valuable learning experience.

Displacements of bonded elements after environmental and accelerated aging that are on the order of a micrometer or less are common. Unfortunately, the selection of an adhesive that satisfies all of the manufacturing, design and performance conditions is often difficult.

Clearly, developing a methodology for characterizing the dimensional stability adhesives and the ability to input this behavior into a finite element code to model dimensional changes should allow the packaging engineer to avoid some of the iterative steps and shorten the development cycle and reduce development costs.

In general, all qualified passive optoelectronic devices must pass a series of rigorous tests detailed in the Telcordia standards 1221 and 1209 [2]. Overall a population of 11 or 21 test units must have less than .5 dB insertion loss after a regiment of test. Population of 11 test units may have at most 1 failure and a population of 21 test units may have at most 2 failures in order to pass this requirement. Insertion loss is the signal optical power loss as a result of an optical element being inserted into the optical light path.

Failure can occur via two mechanisms; the loss of adhesion or the movement of the bonded element resulting in altering the direction of the optical beam. The absorption of moisture into an adhesive joint typically leads to swelling of the adhesive and hence movement of the optical element.

In the building of a passive optoelectronic device, optical elements must be precisely aligned within the optical light path and then held in place during the adhesive curing process. This involves constraining the optical element from any movement. During the curing of the adhesive, the constrained optical element leads to the development of stresses within the adhesive bond. By exposure to a sufficiently high temperature or after a sufficiently long period of time, these stresses are driven toward relaxation. The relaxation of this stress is achieved by the movement of polymer chains within the adhesive from a higher, stressed, energy state to that of a lower energy state. The result is deformation of the adhesive and subsequent movement and misalignment of the optical element.

The source of these stresses come from a variety of causes, some of these are:

1. The volumetric contraction of the adhesive during its curing
2. The extent of cure of the adhesive which leads anisotropic of the material due to zones of cured and zones not fully cured material
3. The thermal history including maximum temperature, minimum temperature and the thermal ramp rates
4. CTE mismatch between the adhesive and the materials being bonded

5. Moisture being absorbed leading to swelling or being drive off leading to contraction
6. The material properties and the chemical make-up of the adhesive.

The problem of identifying a dimensionally stable adhesive arose from work during the development and qualification of terrestrial and submarine passive devices by the optoelectronics division of Lucent Technologies. The design was to incorporate a wide range of optical elements onto a base platform. The basic platform is illustrated by a three pump combiner shown in Figure 1.

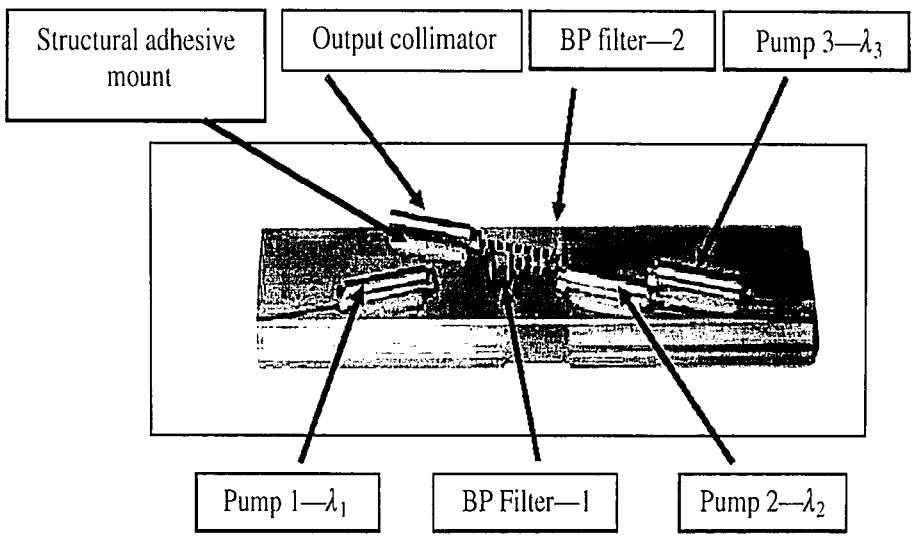



**Figure 1.** Photograph of a three pump combiner developed by Lucent Technology. Three different pump lasers were input via collimators and through reflection and transmission by two optical elements were combined into one output collimator.

The optical elements were bonded using an automated robotic pick and place system. A sweet spot in the center of each optical element was defined as the location of lowest optical power loss. A wide range of different functions could be achieved using the same platform and attachment method while varying the optical coating put on the element.

The collimators were first sheathed in a 303 stainless steel sleeve, the same material as the base of the platform. This helped to reduce the magnitude of any CTE mismatch. A precise six-axis alignment station was developed to, hold the collimator, align it within the optical path for optimal coupling and then hold it in place which it was being bonded. The typical bondline thickness was 225 um +/- 175 um, depending on the degree of tomb-stoning (tilting) of the optical elements and collimator pointing angle.

## 2. OBJECTIVE

The goal of this paper is to report on our progress on the viscoelastic characterizations of optoelectronic adhesives and correlate these characterizations with test vehicle behavior.

## 3. APPROACH

Our characterization of optoelectronic adhesives consists of stress relaxation measurements of selected adhesives in order to develop constitutive equations that can be fed into finite element models [3,4]. Previous studies used test vehicles to verify the constitutive equations. In this study a simple test vehicle was used to rank the dimensional stability of the adhesives as a function of bond line thickness (5 – 200 microns). The cure of these adhesive followed the manufactures

recommendation and not the optimized cure identified during the adhesives characterization.

### 3.1 Optoelectronic Adhesive Selection

Four different adhesives were studied. All of the adhesives are epoxy based. The first adhesive was thermally cured (Adhesive A – Epo-Tek 353ND) and the other adhesives were cured by UV radiation (Loctite ZETA 7411 UV Flood system) and then post-cured with heat. Adhesive B was a model UV-cured epoxy with known chemical composition (DGEBA with 4 phr sulfonium salt). Adhesive C was Loctite and Adhesive D was a Zymet

### 3.2 Cure Characterization

A TA Instruments DSC was used to ensure that all of the adhesives were completely cured. Such characterizations proved important when evaluating UV cured epoxies since the post cured conditions used were significantly different than the recommended conditions provided by the suppliers. Sample size was nominally 10 mg and the samples were scanned at 10 °C/min from 25 °C to 225°C. All samples were scanned twice.

### 3.3 Viscoelastic Characterization

A Rheometrics ARES system was used to perform dynamic mechanical analyses (DMA). The DMA experiments consisted of forced oscillation with a fixed strain amplitude of 0.1% while the frequency was varied logarithmically from 0.1 to 10 Hz and the temperature was increased in increments of 5°C with a 5 minute dwell.

Both the ARES system (torsion) described above and a TA Instruments DMA 2980 (bending) were used to perform stress relaxation tests over a 10,000 second interval and as a function of temperature. The principle of time-temperature superposition was used to extrapolate the data to extremes periods of time and generate master curves.

The viscoelastic behavior can be model either using a stretched exponential or a Prony Series fit. A Prony Series fit is often used in finite element codes. Such fits will be presented and have been discussed in reference 5.

### 3.4 Test Vehicle and Reflected Laser Measurement System for Dimensional Stability

In the past, we have evaluated several types of test vehicles to evaluate both dimensional stability and our ability to predict dimensional stability. In this study, a simple test vehicle was used to study dimensional stability as a function of bondline thickness. See Figure 1a. This test vehicle was closely akin to the three pump combiner outlined above and used actual piece parts from that design including the optical elements and the stainless steel base of the platform. All of these piece parts were manufactured to very tight and consistent specifications including surface roughness, dimensions, clean treatments and storage methods. This greatly reduced variations which might have come from piece parts differences.

Bondline thickness was varied from 5 to 200 microns. The test vehicles were either subjected to a thermal soak of 19 days at 120 °C or to 40 thermal cycles (-45 to 85 °C). Dimensional stability was assessed via the reflection of a laser from the optical surface.

See Figure 2. Rotation of both pitch and roll can be determined by changes in the position of a reflected laser.

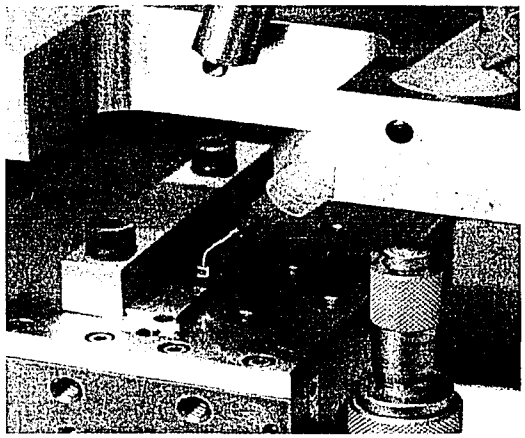


**Figure 2**. Test vehicles containing tetragonal prisms were assembled with controlled bondline thickness.

A system for measuring changes in tip and tilt of the optical elements was developed which incorporated the reflection of a laser from the surface of the element and recording its position on a target. Changes in the yaw of the optical element would move the beam spot left or right while changes in the tilt (tomb-stoning) would move the beam spot up and down. An optical profilometer was available for measuring any translations changes. However it was previously found that translations motion is closely associated with the presence of adhesive fillet around the base of the optical element and that these fillets needed to extent up onto the sides of the optical element. During the assembly process, care was taken to reduce this as much as possible.

Figure 3 shows an overview the measurement method associated with the reflective laser technique.

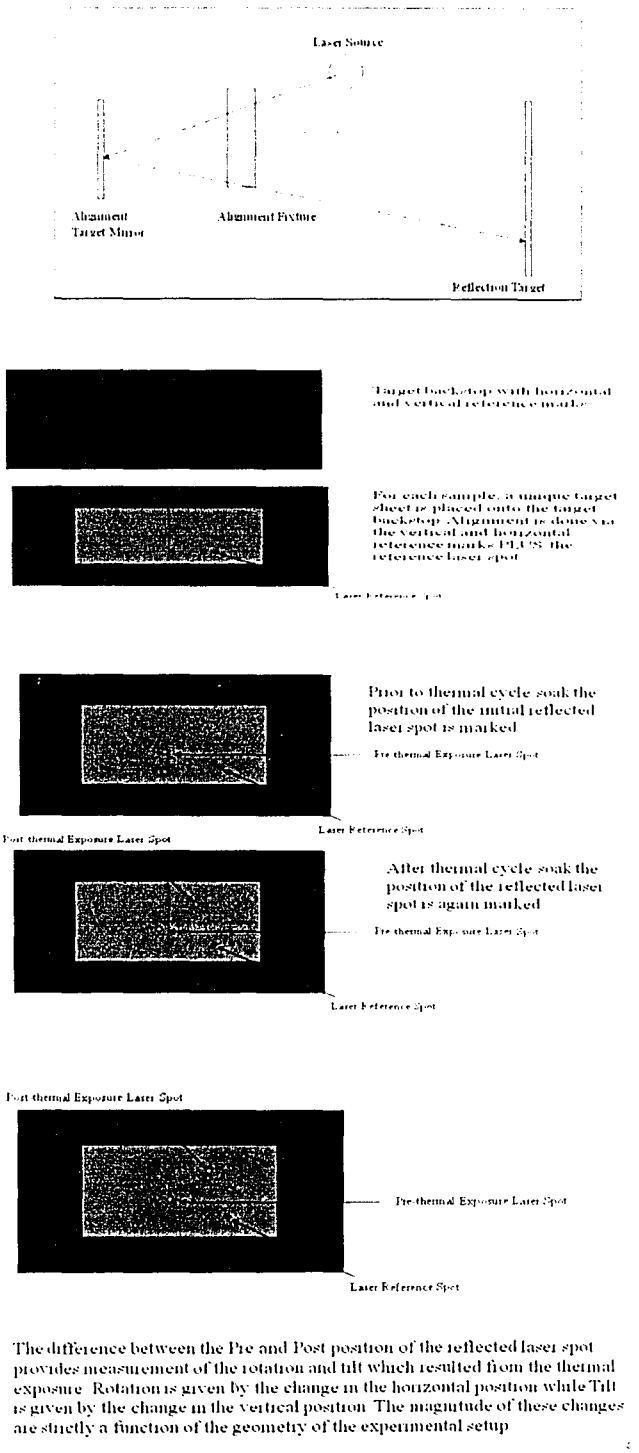



**Figure 3**. Experimental method developed to measure the rotation and tilt of the tetragonal prisms by the movement of the laser spot on the reflection target.

Figure 4 shows a schematic of the test vehicle used in this study. Each test vehicle consisted of one type of adhesive and one bondline thickness.

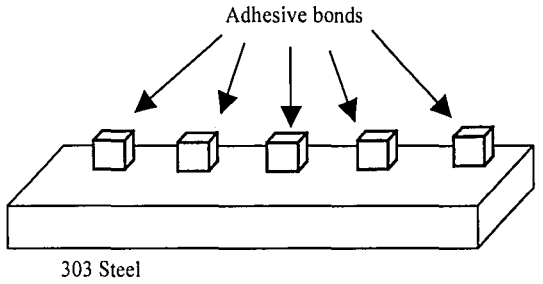



**Figure 4.** Schematic diagrams of the test vehicle used in this study.

Challenges were identified during the assembly process of the test vehicle. It was found that slight differences in the amount of force used to clamp the test vehicle to the measurement system could lead to slight deformation of the platform base thereby altering the location of the reflected laser spot on the downstream target. A torque controlled clamp was developed to apply a known, repeatable amount of force during clamping and thereby remove this potential error. Figure 5 shows this torque controlled clamp.

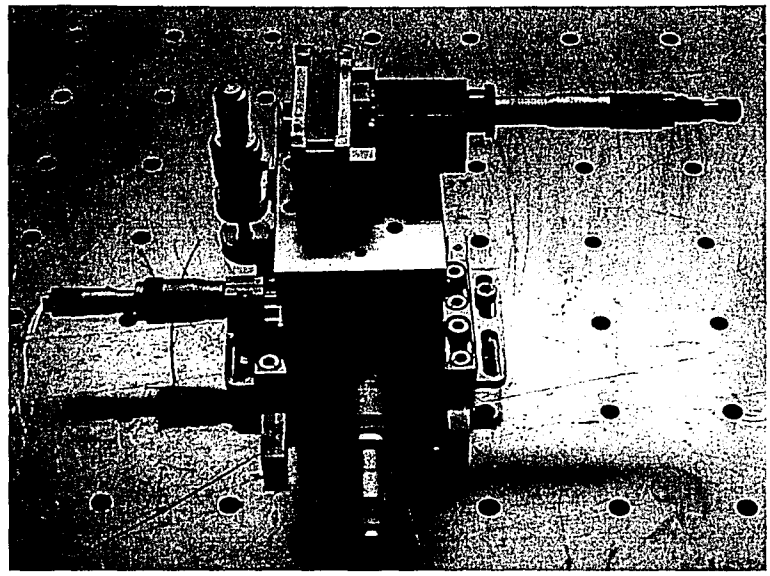


**Figure 5.** Torque controlled clamp to hold test vehicle.

In order to verify the reflected laser measurement method, an optical profilometer was also incorporated. This is illustrated in Figure 6. The location of the four corners of the optical prisms were measured from a know origin and any changes in the position of the prism were thus calculated. The limitation of the optical profilometer was that it had less sensitivity to tilt due to its downward looking perspective.

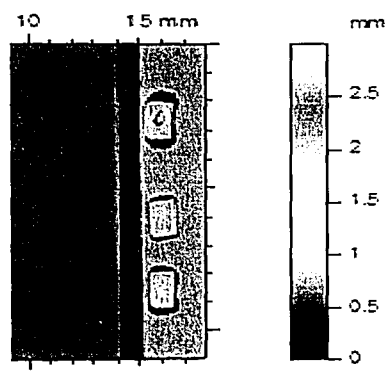



**Figure 6.** Example of an optical profilometer measurement which was used to verification the reflective laser measurement method.

### 3.5 Acceptable Range of Dimensional Change for an Optoelectronic Device

In order to obtain some understanding of the range of acceptable dimensional change on the performance of an optical system, the loss of optical power as a function of mis-alignment was carried out. This involved using optical collimators which were systematically misaligned in first rotation and then in translation. A two stage, six-axis system was developed for this purpose. The accuracy of the stages was .001 degree in pitch, yaw and roll and .1 um in X, Y and Z. The experimental setup is illustrated below in Figure 7.

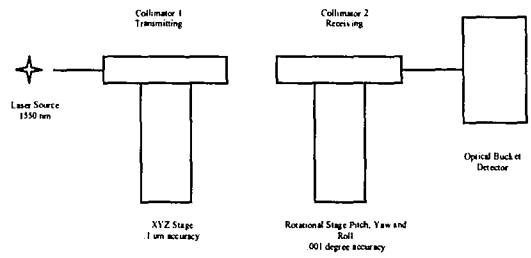



**Figure 7.** Schematic setup of measurement system

A 1550 nm laser source is spliced into fiber of one of the collimators and attached via a vacuum clamp to an adjustable XYZ stage. A second collimator is attached via a vacuum clamp to a 3 axis rotational stage and connected to an optical bucket detector. The collimators are placed at a nominal distance apart and adjust to obtain optimal optical power coupling. The nominal distance Z was found to be a function of the specific collimator pair used and was investigated in an earlier study. The receiving collimator was then mis-aligned by .02 degrees. The XY stage was adjusted to re-peak the optical

power. This was repeated up to a total of 2 degrees. A similar procedure was used for the translation mis-alignment. In addition, the variation in power reading across the optical face of the bucket detector was measured.

Figure 8 is a measure of the optical power variation (blue) and the PDL polarization dependent loss (yellow) across the active area of the optical face of the bucket detector. The maximum power variation was measured to be in the range of .025 dB.

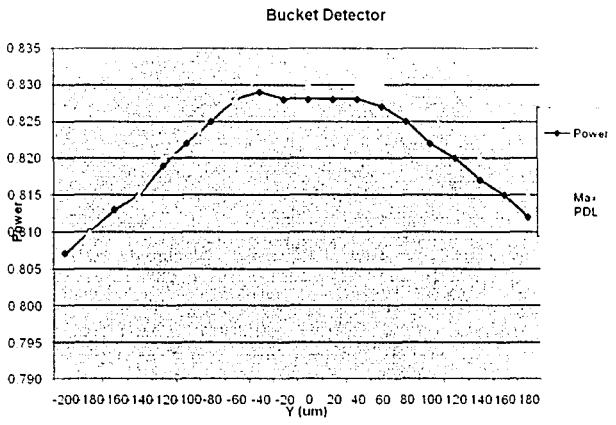



**Figure 8.** Varition in the response of the optical bucket detector across the active area.

The loss of optical power due to mis-alignment up to 2 degrees rotation and 4 mm translation is illustrated in Figure 9.

The results indicate that at or very near optimal/peak alignment, the system is less sensitive. This is demonstrated by the curve of IL versus mis-alignment being relatively flat near peak alignment. The further one moves from peak alignment the faster optical power tends to drops off for a given step.

A common post-cure IL spec during the assembly of a passive optoelectronic device is approximately .5 dB. Post cure IL refers to the difference in optical power from peak alignment and the adhesive is still uncured, to the after the adhesive is fully cured. This is the additional insertion loss resulting from the curing of the adhesive. It is not practical to tie up an alignment station while an adhesive fully cures. Typically an adhesive it "tack' cured while the device is on the alignment station via UV or some other method. Then it is then fully cured in an oven.

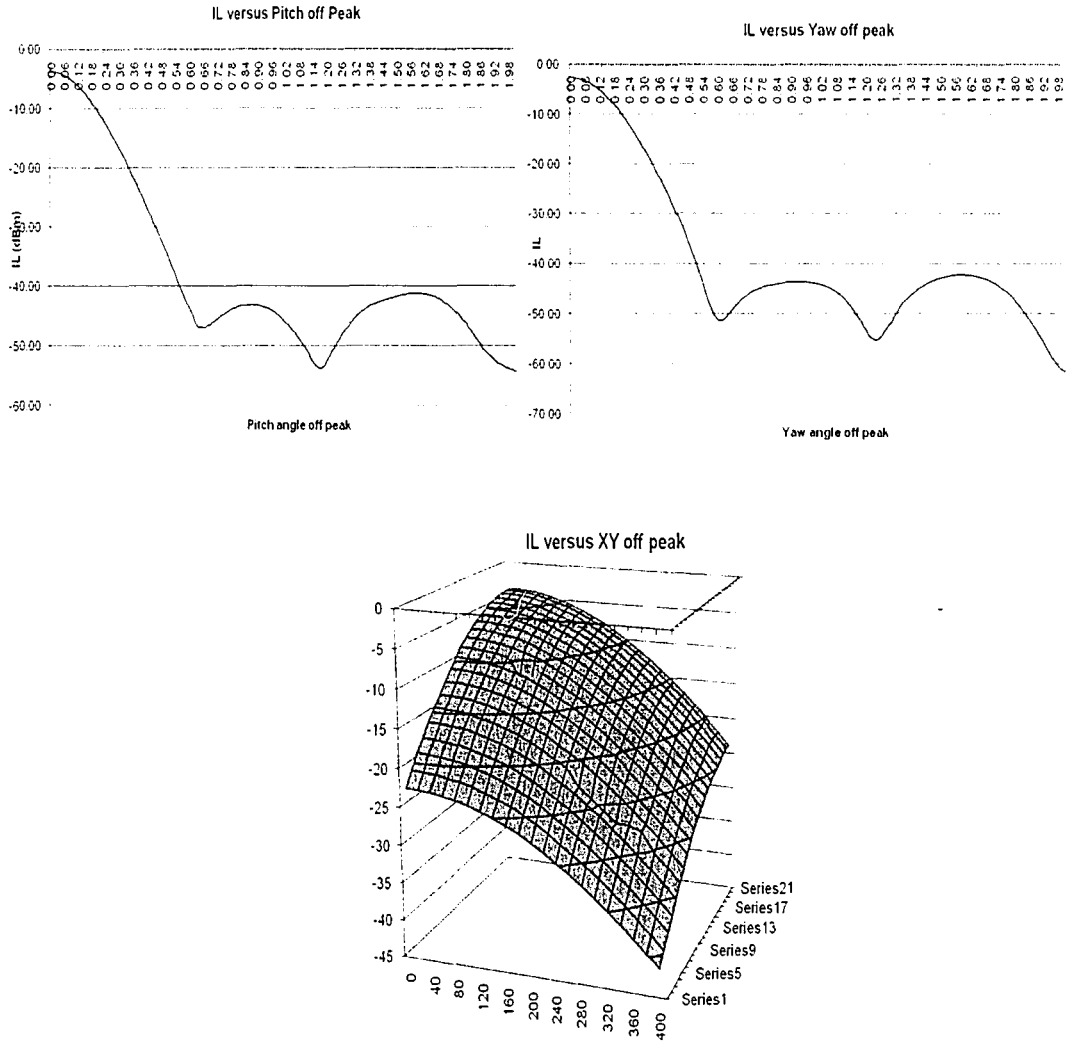



**Figure 9.** Optical poer loss (dB) as a function of mis-alignment in pitch, yaw and roll.

A common post-cure IL spec during the assembly of a passive optoelectronic device is approximately .5 dB. Post cure IL refers to the difference in optical power from peak alignment and the adhesive is still uncured, to the after the adhesive is fully cured. This is the additional insertion loss resulting from the curing of the adhesive. It is not practical to tie up an alignment station while an adhesive fully cures. Typically an adhesive it "tack' cured while the device is on the alignment station via UV or some other method. Then it is then fully cured in an oven.

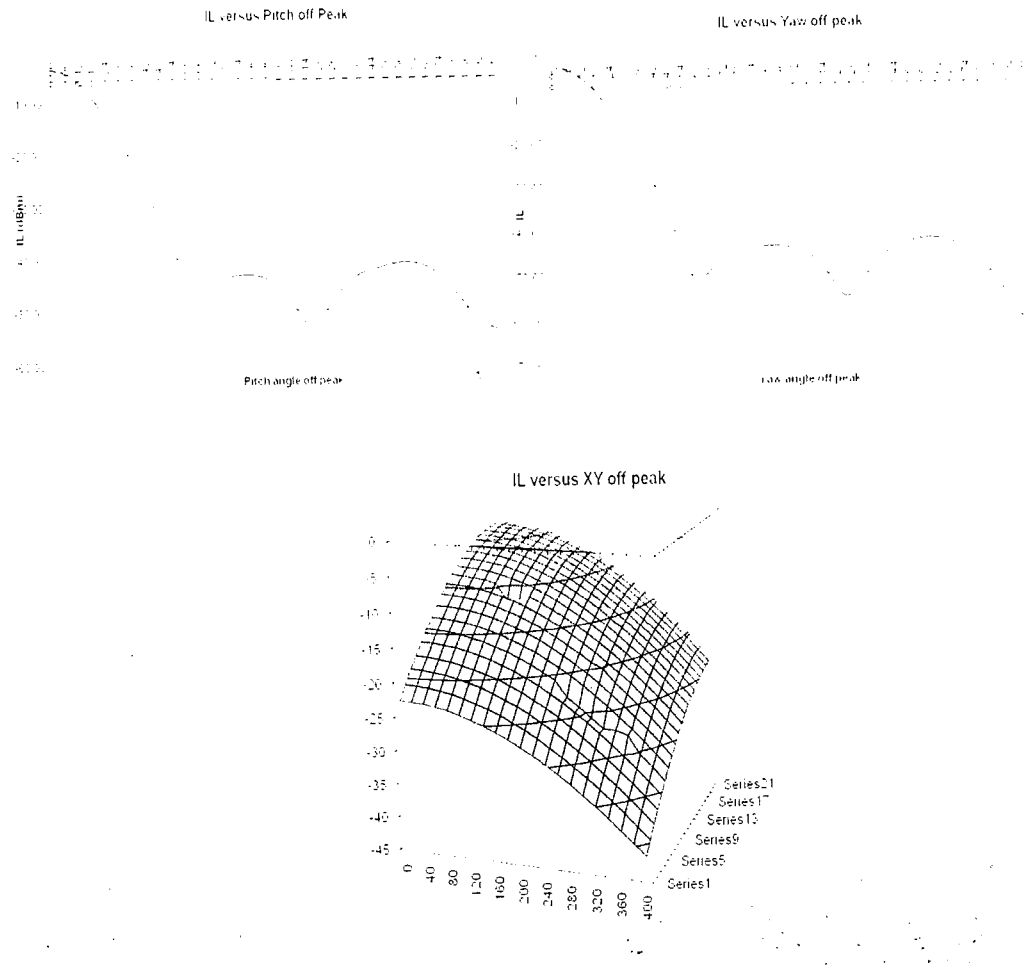



**Figure 9.** Optical poer loss (dB) as a function of mis-alignment in pitch, yaw and roll.

There are many factors which contribute to the sensitivity of an optoelectronic device to dimensional change of bonded elements. These include optical path length, the size of the optical "sweet spot" in optical filters or prism and the wavelength light, just to name a few. The preceeding study was done to allow an estimate of acceptable dimensional change in a bonded element. This is not intended to be an absolute specification but rather a general guideline. Given this, the maximum total movement of an optical element from optimal alignment is on the order of between .1 and .15 degrees for pure rotation or of 60 um for pure translation.

## 4. RESULTS

Viscoelastic characterizations and movement of the adhesives in the test vehicles are discussed below. A general overview of the characterization of the adhesive will be given. A detailed presentation of the characterization of all four adhesives is beyond the scope of this paper.

### 4.1 Adhesive Characterization

DSC was performed to determine the proper cure procedure such that the adhesive is completely cured. To achieve thermal stability of an epoxy it is necessary to achieve a cure of at least 97 %. [6].

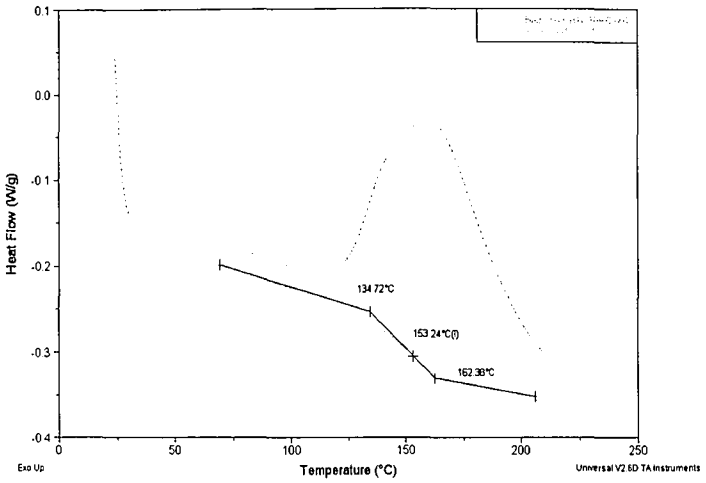



**Figure 10.** DSC traces (1st and 2nd scans) for Adhesive B post-cured for 10 minutes at 120 °C.

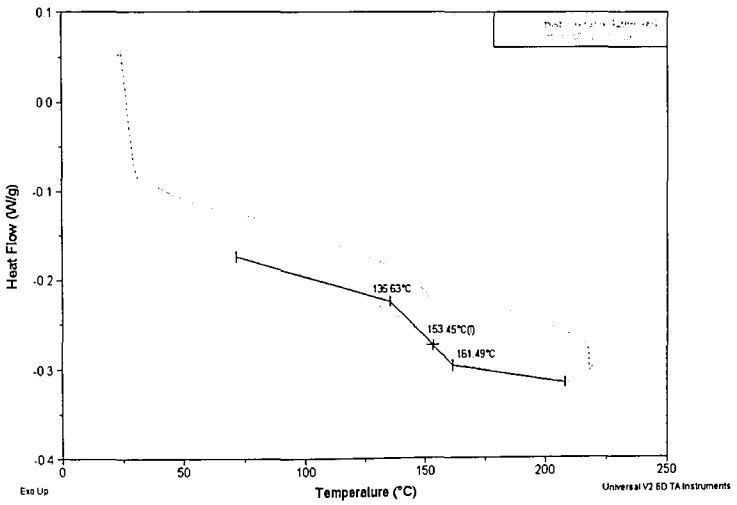
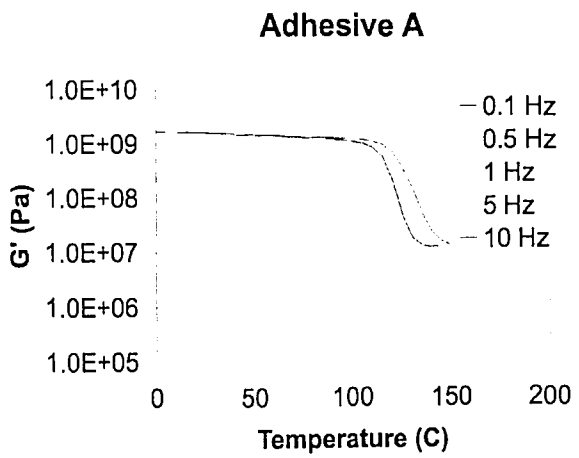



**Figure 11.** DSC traces (1st and 2nd scans) for Adhesive B post-cured for 120 minutes at 120 °C.

The cure procedures for the UV cured adhesives were significantly different than the manufacturer's recommendation. This may be partly due to the thickness of our tests specimens (>250 microns). Figure 3 contains DSC thermograms of Adhesive B cured under recommended conditions (Fig. 10) and cured under conditions that gave complete cure (Fig. 11). Note that a post-cure of 2 hours at 120 °C was required even though the recommended post-cure time is 10 minutes at 120 °C. Note that there is very little shift in Tg when the samples are properly post-cured. Fully cured samples are required for our viscoelastic experiments that use time-temperature superposition to predict long-term behavior.

The viscoelastic characterization consisted of a dynamic test that varied temperature and frequency and a step strain test (Stress Relaxation test) that was conducted at several temperatures. The strain was kept constant at 0.1 % strain. Results form other of viscoelastic tests are reported in reference 4.



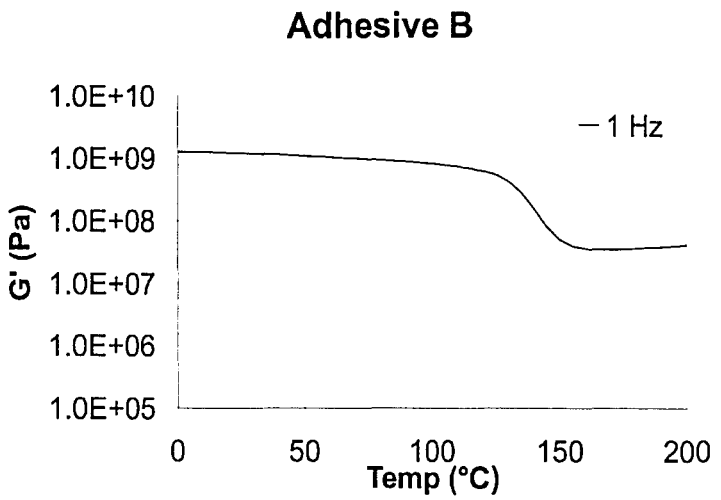



**Figure 12.** Dynamic modulus measurements reveal differences between the two adhesives.

Dynamic mechanical tests are important for determining the glass transition temperature and rubbery plateau modulus. The glass transition temperature influences the kinetics of the viscoelastic response and the difference between the glassy modulus and the rubbery plateau modulus determines the strength of the response. See Figure 12. These results indicate that Adhesive B (the model UV-cured adhesive) has a higher glass transition temperature and higher plateau modulus than Adhesive A (heat cured). Therefore Adhesive B is more crosslinked and should exhibit greater dimensional stability.

Stress relaxation experiments for the two adhesives show that relatively little relaxation takes place scale at short times at temperatures below 100 C. See Figure 13. Since the Adhesive B has a higher glass transition temperature, it relaxes more slowly and to a lesser extent than the Adhesive B. Both adhesives relax significantly at higher

temperatures. Utilizing the time-temperature superposition principle one can derive projected long-term behavior from experiments at higher temperatures. See Figure 14.

The stress relaxtion behavior of both adhesives was fitted to a stretch exponential [5]:

$$G(t) = G_r + (G_u - G_r)e^{-(t/\tau)^{(1-n)}} \qquad (1)$$

Where G(t) is the stress relaxation modulus in shear; $G_r$ is the relaxed modulus, $G_u$ is the unrelaxed modulus; t is time in seconds; t(tau) is the characteristic relaxation time; and, n is the coupling parameter.

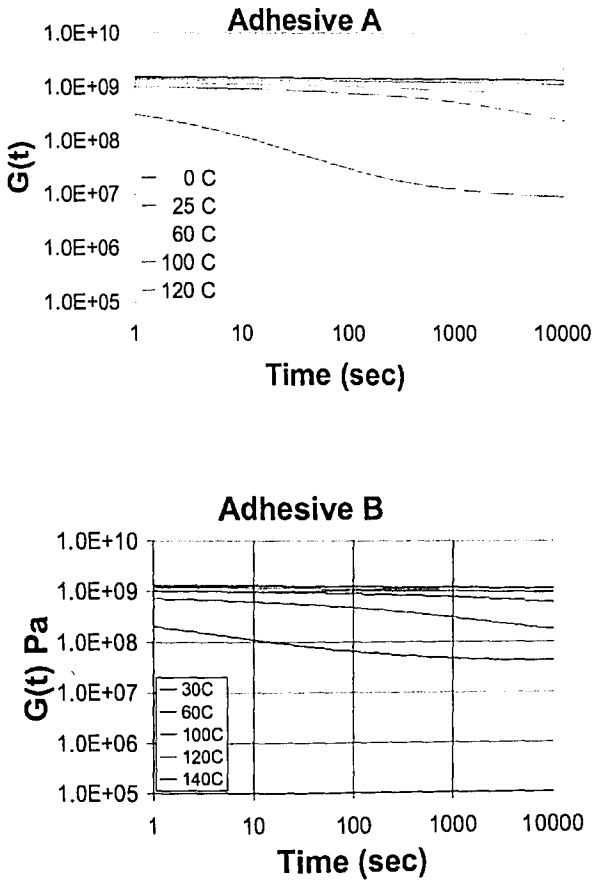
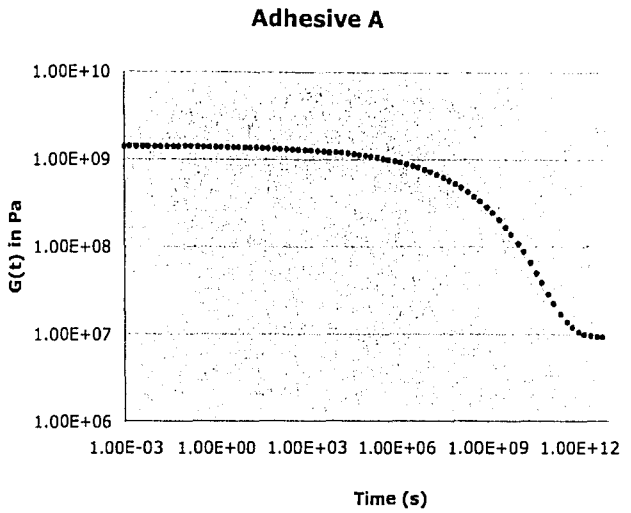



**Figure 13.** Stress relaxation tests also reveal differences between the two adhesives.



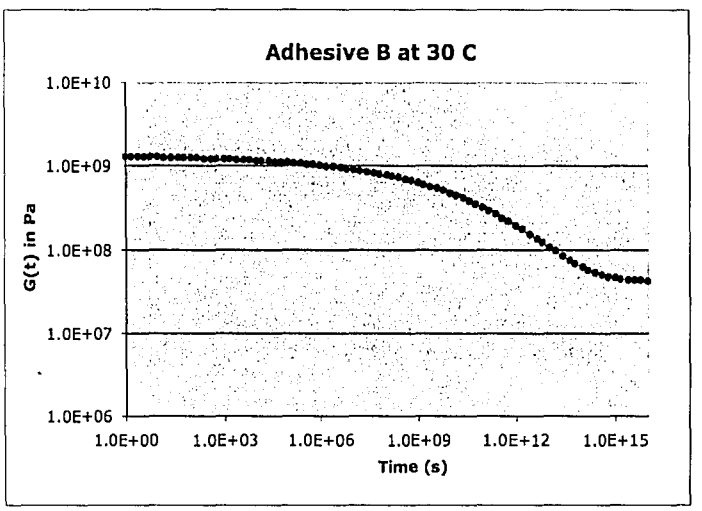




**Figure 14.** Time-Temperature Superposition plots of stress relaxation behavior. These plots contain shifted stress relaxation data (lines). The dots are data points from fitting the data to a stretched exponential [G(t) =Gr+(Gu-Gr)exp-(t/tau)^(1-n)].

Fitting the stress relaxation data of Adhesive A resulted in a $G_u$ = 1.41 GPa, $G_r$ = 9.12 MPa, relaxation time constant (tau) = 9.5E7 sec, and the coupling parameter n= 0.79. Fitting the stress relaxation data of Adhesive B resulted in a $G_u$ = 1.33 GPa, $G_r$ = 41.7

MPa, relaxation time constant (tau) = 5.0E9 sec, and the coupling parameter n= 0.84. The curve fits appear acceptable when plotting on a log scale. Note that Adhesive B has a longer time constant and lower strength of the relaxation ($G_u - G_r$). This should result in a more dimensionally stable adhesive.

Although the fit of the relaxation data to a single stretched exponential looks good on a logarithmic modulus scale, if one plots the modulus versus time data for several temperatures on a linear modulus scale then the parameters must be adjusted to fit data at each time scale. See Figure 15. The affect of temperature on the time constant is well-known and predictable. Therefore, the shorter time constants at higher temperatures is expected. See Table 1. The affect of temperature on the coupling parameter n is not so well understood [7]. The change in the coupling parameter as a function of temperature means that the shape of the relaxation time spectrum changes with temperature, i.e. our materials are not rheologically simple. This will be an issue when trying to predict the performance of the test vehicle quantitatively.

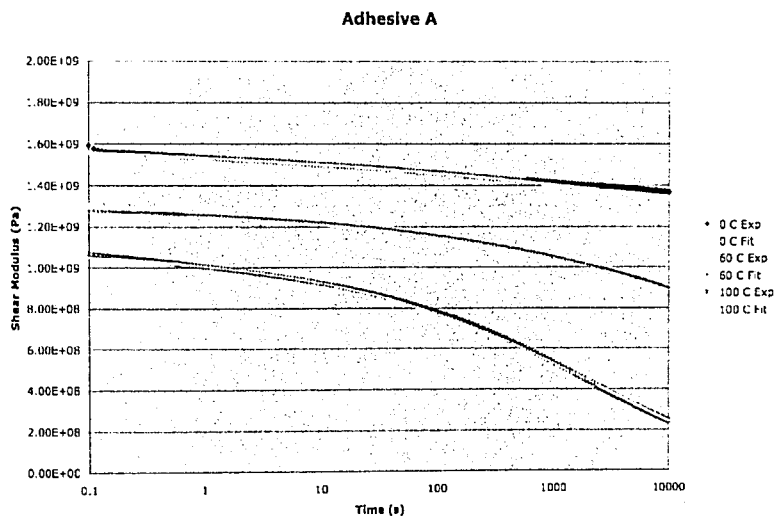



**Figure 15.** Stress relaxation behavior as a function of temperature.

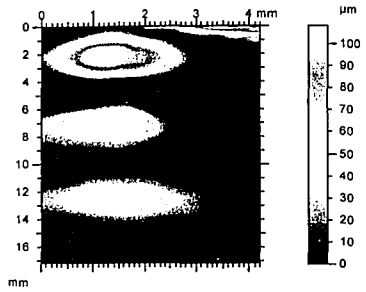
**Table 1:** Parameters used to fit viscoelastic data using a stretched exponential (Eqn 1).

| Temp. | $G_u$ in GPa | $G_r$ in MPa | Tau in sec. | n |
|---|---|---|---|---|
| 0 °C | 1.61 | 9.12 | $2.0 \times 10^{10}$ | 0.87 |
| 60 °C | 1.11 | 9.12 | $5.5 \times 10^{5}$ | 0.76 |
| 100 °C | 1.09 | 9.12 | $2.6 \times 10^{3}$ | 0.68 |

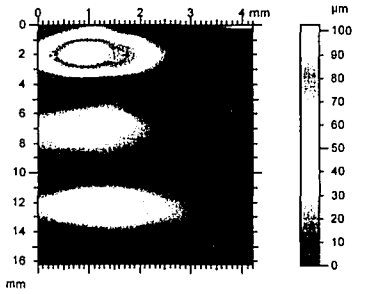
### 4.2 Cure Contraction

During the curing of an adhesive it undergoes a volume shrinkage or cure contraction. This shrinkage may move some optics out of alignment. Stress from shrinkage is an inherent property of the chemicals making up an adhesive. Chemical bond changes and molecular distances contribute to shrinkage [8, 9]. Bonds from the relatively distant molecules in a liquid state polymerize to form shorter bonds in the cured state. The molecular bonds of a polymer are typical shorter than the bonds of the monomer. Molecular bond lengths changes are independent of either fast or slow curing process.

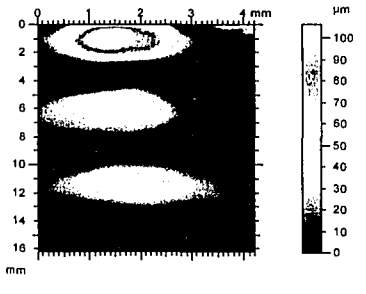
The changes in the volume of the tested adhesives were measured using an optical profilometer. This was done at various points; as dispensed and no-cure, post-UV cure and finally post-Thermal cure. Figure 16 shows these optical profilometer measurements for Adhesives B, C and D.




no-cure




post-UV cure




post-Thermal cure

**Figure 16.** Optical profilometer measurement of cure contraction for Adhesive C

During the curing of an adhesive a percentage of material is lost due to outgassing or the release of volatile substances. This results in a weight loss during curing and can be measured using a TGA. Weight loss during thermal cure of the adhesives weas measured using a TA Instrument 2980 TGA. Figure 17 shows the results for adhesive B, C and D.

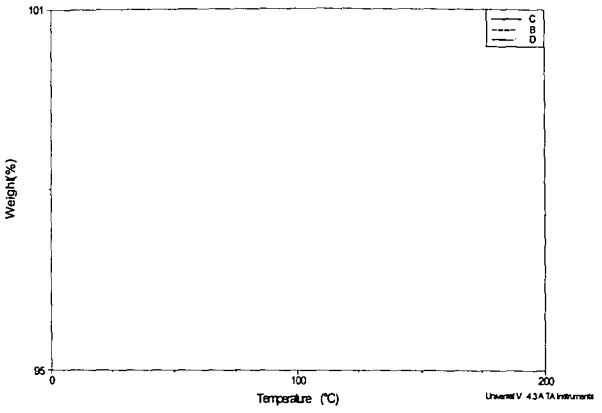



**Figure 17.** Weight loss as a function of temperature for Adhesive B, C and D

The results indicate a significant difference between the amounts of material given off during the cure for the three adhesives.

### 4.3 Test Vehicle Response

Results from thermal cycling are shown in Figure 18 and from thermal soak are shown in Figure 19.

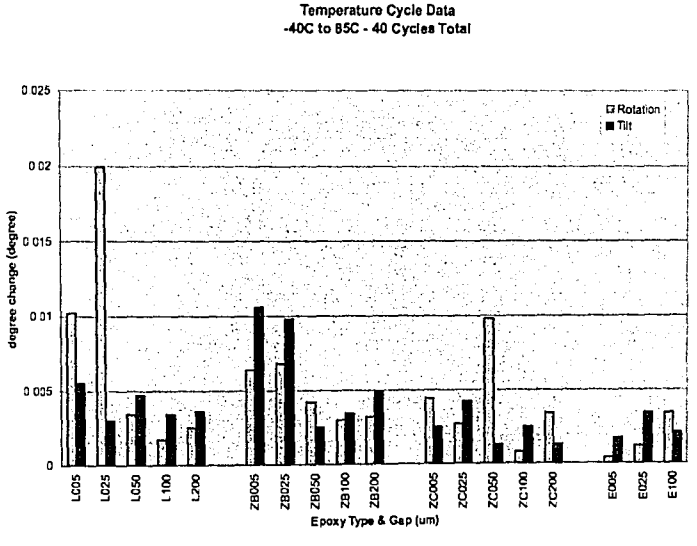



**Figure 18.** Thermally cycling affects both tilt and rotation.

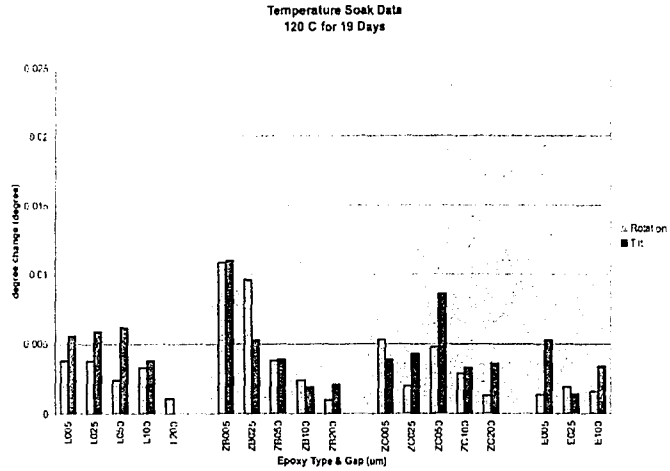



**Figure 19.** Thermal soaking affects both tilt and rotation.

These results show that Adhesive A (described as Exxx on these charts) is the most stable, although the UV cured adhesives are comparable at larger bondline thickness (100 and 200 microns). Although these results are similar to those obtained by Bournes and Thadani [10], Adhesive A (Epo-Tek 353 ND) is more stable in our studies. Several explanations can be given for the lack of performance of the UV-cured adhesives. Firstly, at small bondlines it may be difficult to get the light into the bondline and initiate the UV cure reaction. Secondly, the cure schedules used followed the manufacture recommendations and were different than that use determined to be optimal via the viscoelastic studies. Finally, a thinner bondline provides less bulk adhesive material for the distribution of internal stresses

## 5. SUMMARY AND DISCUSSION

Our results indicate that mechanical measurements can easily differentiate the behavior of optoelectronic adhesives, however, predicting the performance of these adhesives is a much more difficult task since they are not rheologically simple. A simple test vehicle was used to evaluate the dimensional stability of optoelectronic adhesives but clear trends on the effect of bond thickness and type of adhesive were not observed.

## 6. ACKNOWLEDGMENTS

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## 8. VITA

Thomas Robert Daugherty was born on March 25, 1964 in Allentown, PA to the proud parents of Francis William Daugherty and Margaret Herczog Daugherty.

He graduated from Catasauqua High School in 1982 and obtained college degrees from Lehigh Carbon Community College (Associates Degree in Liberal Arts), Kutztown University (Bachelors Degree in Physics), and Lehigh University (2 - Masters Degree in Physics and now a Masters Degree in Polymer Science and Engineering).

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