Strain measurement and numerical analysis of an epoxy adhesive subjected to thermal loads

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This study was undertaken to improve understanding of the mechanics of residual stresses in electronic assemblies when subjected to thermal cycles. Strain-gauge measurements of stress and finite element analysis were performed to determine the residual strain level in an epoxy adhesive. Discrepancies in the stress value between experimental methods and those predicted from the finite element model highlight the necessity of incorporating temperature-dependent material properties into the model. Furthermore, the effects of curing conditions on the epoxy layer were also quantified in terms of strain levels.

Introduction

Within the electronics community, finite element analysis is often used to predict stress as an assembly is subjected to thermal cycling [1–4]. Since the assembly is fabricated from numerous materials having disparate properties, calculation of thermally induced stress is dependent upon input of the individual material parameters which exhibit temperature-dependent properties (e.g., thermal coefficient of expansion, strength, and modulus of elasticity). For polymeric materials, the coefficient of thermal expansion (CTE) below the glass transition temperature (T_g) can be greater than an order of magnitude lower than the CTE above T_g. The incorporation of relevant temperature-dependent variables into a finite element model should successfully predict the thermally induced stress in an electronic assembly.

Determination of the temperature-dependent properties of individual materials is relatively straightforward. Thermal analysis techniques (such as thermomechanical analysis, TMA, dynamic mechanical analysis, DMA, and differential scanning calorimetry, DSC), coupled with mechanical testing of tensile strength and modulus of elasticity, provide adequate data suitable for input into the finite element model. From this input, residual stress and/or thermally induced stress is calculated and used to predict product performance [5]. One problem that arises in residual stress/strain calculations is the selection of a zero-stress temperature. Since the assembly is often fabricated via adhesive bonding, the zero-stress temperature of the cured adhesive must be determined. However, as the adhesive is generally a thermoset material which undergoes a phase transition from a liquid, paste, or

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Table 1 Manufacturer's (3M) specification for an acrylic-modified, epoxy film adhesive.

Composition	
Epoxy resin (DGEBA)	60–100%
Acrylic polymer (trade secret)	5–10%
Epoxy resin (2-propenenitrile/1,3-butadiene, carboxy-terminated polymer with bisphenol A and epichlorohydrin)	<5%
Amide curative (cyanoguanidine)	<5%
Amorphous silica	1–5%
1,1′-(4-methyl-m-phenylene)bis (3,3-dimethylurea)	0.5–1.5%
2,6-di-tert-butyl-p-cresol	0.1–1.0%
Nominal thickness	3, 5, 10 mils
Overlap shear adhesion	
Cold rolled steel/cold rolled steel	3100 psi
FR-4/FR-4	2500 psi

film to a cured, cross-linked material, determination of the zero-stress temperature is a nontrivial task. Since residual strain (ϵ_r) at a given temperature is the product of the CTE difference ($\Delta\alpha$) and the difference between the zero-stress temperature and the current temperature (i.e., $\epsilon_r = \Delta\alpha \cdot \Delta T$), residual strain at the electronic assembly operating temperature is extremely sensitive to the proper assignment of the zero-stress temperature. Inaccurate selection of the zero-stress temperature can result in erroneous stress predictions which either underestimate actual stress levels, leading to reliability degradation, or overestimate actual stress levels, leading to product overdesign.

From a design perspective, it is desirable to couple the theoretically predicted stress to the experimentally determined stress so that stress levels in future designs can be accurately predicted. To accomplish this, temperature-dependent strain measurements were made on suitable test vehicles.

Experimental section

Materials

Polished glass-ceramic substrates (IBM proprietary composition) were used as received.

Plates of heat-treated Invar39 (39% Ni; <1% Mn + Si + C; ~60% Fe) were purchased from Lake Machine Corporation[1] and polished to a 1-μm finish using diamond paste.

An acrylic-modified epoxy resin film adhesive designated as AHS 207, furnished by 3M,[2] was used to bond the glass-ceramic to the Invar** substrates. Physical property data, as supplied by the manufacturer, are listed in **Table 1**. The epoxy film was provided in various thicknesses on a silicone-coated Kraft paper release liner.

A strain gauge (ID number CEA-13-250UW-350, purchased from the Vishay Micromeasurements Group[3]) was used for all microstrain measurements. This strain gauge has an operating temperature range of −100°F to 350°F.

Sample preparation

To extend the shelf life of the adhesive film, the material was stored in sealed bags at −40°C. Prior to use, the film was removed from the freezer and permitted to equilibrate to room temperature within the bag to avoid moisture adsorption. Samples in the shape of dog bones were excised from the tensile sheet stock using an ASTM 638D Type IV metal die. Freestanding films of the adhesive were prepared by removing the Kraft paper liner, placing the samples on a flat sheet of Delrin** plastic, clamping the top and bottom edges to the Delrin, and curing the clamped samples at 120°C/60 min. To minimize cured-in stress, the samples were slowly cooled to ambient temperature from 120°C by gradually reducing power to the oven.

Strain measurements on the epoxy film adhesive were recorded for freestanding and constrained films bonded between Invar and ceramic substrates. The instrumentation consisted of a three-wire strain gauge sandwiched between two 2-mil films of the adhesive, cured at various conditions and measured using a quarter Wheatstone bridge. The third wire of the strain gauge compensated for resistance changes as a function of temperature. For the freestanding films, the release liner was removed, and the strain gauge was laid flat on a block of Delrin in order to prevent shrinkage-induced curling of the adhesive. No load other than the weight of the Delrin plate was used to keep the sample flat. For the constrained films, the adhesive was positioned between the Invar and ceramic plates with two large binder clips used to provide a nominal clamping force. The strain gauge was zeroed at room temperature. Samples of the freestanding films were cured at either 150°C/30 min or 110°C/90 min in a convection oven; samples of the constrained films were cured at 150°C/30 min. Strain was recorded at the completion of the cure cycle at the cure temperature and following cooling to ambient. Subsequent strain measurements were recorded as the samples were ramped at 5°C/min to 150°C.

Thermal analysis

Stress-free samples of the adhesive were prepared as described above. A rectangular section of the sample was secured in the extended measuring thermal analysis system

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[1] Lake Machine Corporation, 470 11th St., Palisades Park, NJ 07650.

[2] 3M Adhesives Division, 3M Center, St. Paul, MN 55144.

[3] Vishay Micromeasurements Group, Inc., 951 Wendell Blvd., Wendell, NC 27951.

(Perkin-Elmer TMA7) and the temperature ramped at 5°C/min. CTE measurements were made both below and above T_g. In order to determine whether or not the film expansion was isotropic, duplicate samples were prepared parallel and transverse to the coating direction. Since the CTE varied less than 2 ppm/°C between samples, the film adhesive was considered to be isotropic.

The glass transition temperature of the cured adhesive was determined via differential scanning calorimetry as well as thermomechanical analysis. Calorimetry was conducted using a Perkin-Elmer Pyris 1 differential scanning calorimeter (DSC) by ramping the temperature from 0 to 300°C at 20°C/min. The T_g was taken as the inflection point in the endothermic baseline shift. Thermomechanical analysis was conducted using a Perkin-Elmer TMA 7 by ramping the temperature at 5°C/min from 25 to 150°C. In this case, the T_g was calculated as the intersection temperature of the extrapolated low- and high-temperature expansion curves. Residual stress calculations are dependent on both T_g and CTE inputs, both of which may vary with the cure conditions. The glass transition temperature input into the model was calculated from TMA data collected at a heating rate of 5°C/min. DSC analysis was used solely to evaluate whether the T_g was dependent upon the curing conditions. Although accurate determination of the T_g is typically accomplished via extrapolation of the DSC heating rate to 0°C/min, as long as the heating/cooling rate is identical for each sample, the T_g obtained at 20°C/min may be used for comparative purposes. The absolute magnitude of the T_g may be higher than that obtained from extrapolation to a 0°C/min heating rate (the observed T_g decreases as the heating rate decreases because of supercooling of the rubbery phase), but this is inconsequential for the intent of this study. The effect of the cure cycle on T_g of the film adhesive is illustrated in **Table 2**. It is apparent that T_g is independent of the cure cycle, provided that the epoxy film is cured completely. T_g and CTE values obtained via TMA were input into the finite element model.

Table 2 Effect of cure condition on the T_g of the epoxy film adhesive.

Cure temp. (°C)	*Cure time*	T_g (°C)
175	5 min	106.8
150	30 min	114.0
125	60 min	113.4
100	120 min	120.1
80	48 hr	112.7
		Mean = 113.4 ± 4.7°C

Table 3 Physical properties of the epoxy film adhesive.

Strain at break (%)	*Stress at break* (psi)	*Young's modulus* (psi)
4.949 ± 0.734	4601 ± 309	158,000 ± 13,000

Physical property analysis

Mechanical properties (Young's modulus and maximum stress/strain at break) of the film adhesive were determined via an Instron** 4505 Universal Testing Instrument equipped with a 50-newton (N) load cell by securing the tensile samples in the jaws of the instrument and driving the crosshead at 0.2 inches per minute (ipm). The results are summarized in **Table 3**. Young's modulus was obtained from the slope of the stress/strain curve.

Invar39 cylinders (0.5-in. diameter) were bonded to glass-ceramic substrates and the adhesive cured at 150°C/30 min. Bond strength was determined by securing the ceramic plate in a fixture in the lower jaws of an Instron 4505 equipped with a 1124-lb load cell and pulling the cylinder at 0.1 ipm. For those samples that exhibited cohesive failure in the epoxy film, the load exceeded 5000 psi. Typically, cohesive failure in the glass-ceramic was observed.

Overlap shear strength was determined by bonding 1-in. × 1-in. glass-ceramic plates (0.25 in. thick) to 2-in. × 2-in. Invar39 plates. A custom fixture was built to ensure that the load was evenly distributed across the leading edge of the ceramic as the assembly was pulled with a shearing force. At a load of approximately 3000 psi, an audible "crack" was accompanied by cohesive failure in the ceramic. From the peak stress value in Table 3, the maximum load-carrying capability of the adhesive is approximately 4600 psi. Thus, under shear loading, substrate failure occurs before the bond line is affected.

Finite element analysis

The objective of the finite element analysis (FEA) was to compare the predicted residual stress and strain levels in the epoxy material during cyclic heating and cooling from its stress-free temperature to those levels determined experimentally.

The sample to be analyzed and tested consisted of 50 mils of Invar and 50 mils of glass with 7 mils of epoxy applied along the perimeter of the sample. A schematic of the sample is shown in **Figure 1**. A quarter section of the above sample was modeled using the I-DEAS Master Series**, a finite element package.[4] The finite element model consisted of 31251 nodes with 5700 quadratic solid elements. The initial conditions considered were at a stress-free temperature of 110°C with symmetric boundary

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[4] Structural Dynamics Research Corporation, 2000 Eastman Dr., Milford, OH 45150.

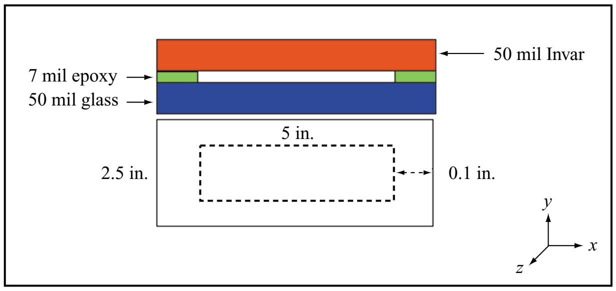

Figure 1

Schematic of the sample used for testing and analysis.

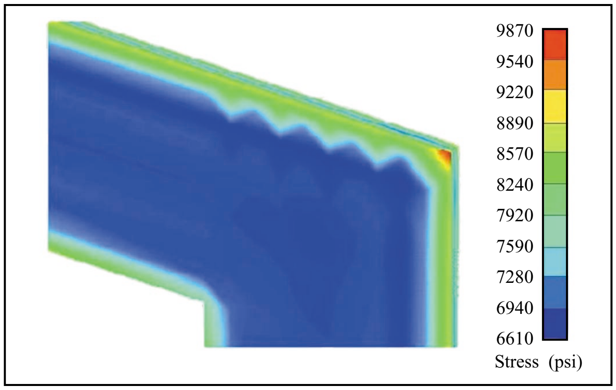

Figure 2

Von Mises stress distribution in a quarter section of the epoxy sample at 25.5°C.

Table 4 Material properties of sample for finite element analysis.

Material	*Young's modulus* (psi)	*Poisson's ratio*	*CTE* (ppm/°C)
Invar39	22×10^6	0.3	2.9
Glass	18×10^6	0.25	3
Epoxy	1.6×10^5	0.3	30

conditions applied at the *xz* and *yz* planes. The model was then thermally cycled by cooling to a steady-state temperature of 25.5°C, heating to 150°C and then cooling back to room temperature. The material properties used in the model are listed in **Table 4**.

Results and discussion

Von Mises stress analysis [6] contours indicate probable failure locations and their associated stress levels. On the basis of the steady-state cooling analysis, the maximum total stress (calculated using the Von Mises theory) seen in the epoxy material was 9870 psi. The maximum shear stress that the test vehicle could withstand was evaluated via mechanical testing of overlap shear specimens of Invar-to-glass bonded samples. In most cases, cohesive fracture at loads exceeding 3400 psi occurred in the glass prior to either interfacial failure along the bond line or cohesive failure within the epoxy. Therefore, 3400 psi represents a *minimum* value for the overlap shear strength. Note that the tensile strength of a freestanding film of the adhesive is 4600 psi, well above the overlap shear strength of the Invar-to-glass joint. These results indicate that the strength of the joint exceeds the maximum stress imparted to the bond via any mismatch in CTE. Since the Von Mises value (9870 psi) exceeds the shear strength of the epoxy (4600 psi), the probability of a thermally induced crack is high, as shown in the contour plot of **Figure 2**. In the figure, regions of maximum stress are indicated in red. However, in order to calculate residual stress at room temperature, it is necessary to calculate shear stress components. Shear stresses in the *xy*, *xz*, and *yz* planes were 180, 3640, and 3460 psi, respectively. The results of the steady-state heating indicated a shear stress of −3200 psi in the *yz* direction, resulting in a residual shear stress value of 260 psi at room temperature (**Figure 3**). The stress value of 260 psi was obtained by subtracting the heating-induced stress from the stress obtained upon cooling. Correlation of predicted stress with experimentally determined stress was attempted by means of quarter-bridge strain-gauge instrumentation. Strain in both freestanding and constrained films of the epoxy adhesive was measured as a function of temperature. The strain gauge was zeroed at room temperature prior to subjecting the samples to any thermal load. As the samples were cured at 150°C, strain was observed to increase because of cure-induced shrinkage of the film. Not surprisingly, strain in the freestanding film is several orders of magnitude greater than that in the constrained film. At the completion of the cure cycle, strain exhibits its maximum value. Upon cooling, strain decreases (**Figure 4**). Both the freestanding and constrained curves exhibit a pronounced break centered at 110°C, the glass transition temperature of the epoxy adhesive. Below T_g, strain varies linearly with temperature at a much greater rate of change than above T_g. This is attributed to the higher modulus of the film adhesive at temperatures below T_g. For both the constrained film and the freestanding film, the strain approaches zero at T_g, indicating that the stress-free temperature occurs at the glass transition. As the samples are cooled below T_g, the CTE mismatch between the substrates and cured adhesive results in the observed strain.

From the strain measurements, the maximum stress at the bond line can readily be calculated from $\sigma = \varepsilon E$, where strain is determined from Figure 4 and the modulus (E) from Table 3. The experimental maximum stress at 25.5°C in the constrained film is roughly 38 psi, well below the predicted stress level of 260 psi. This discrepancy can be accounted for by considering the calculation of residual stress in the model, as well as the manner in which the strain-gauge data were collected. The maximum residual stress was calculated by subtracting the heating-induced stress from that obtained upon cooling. Since the model utilized a single value for the modulus (the room-temperature modulus), the heating-induced stress was exaggerated, resulting in an elevated residual stress at 25.5°C. Strain-gauge measurements also contribute to the discrepancy between the model and the experimentally determined residual stress. Prior to curing the adhesive, the strain gauge was adjusted to zero. The assembly was subsequently placed in a convection oven set to the desired temperature for the duration of the cure cycle. Strain was observed to increase throughout the cure cycle. When the sample was cooled to room temperature, strain decreased to the values indicated in Figure 4. The change in strain values can be rationalized as follows. As the epoxy adhesive is heated, the viscosity initially decreases, then begins to increase rapidly as cross-linking ensues. Polymerization at the cure temperature results in film shrinkage corresponding to positive strain. Since the cure temperature is well above the T_g (150°C vs. 110°C), segmental motion in the polymerized film permits stress relaxation during cure. However, the strain gauge is responding to the film shrinkage and, consequently, recording positive strain. Upon cooling to room temperature, the mismatch in thermal expansion between the substrates and the adhesive translates into the observed strain. The finite element model calculates heating-induced stress on the basis of cured film properties and, consequently, predicts much higher stress values as the assembly is heated.

Conclusions

The foregoing results highlight the necessity of comparing experimentally determined stress/strain with that theoretically predicted from a finite element model to improve understanding of the mechanics of residual stresses in electronic assemblies. Although the analytical model correctly predicts the locus of highest stress, it overestimates the magnitude. In this specific instance, the model predicted higher stress levels than those experimentally determined. Although this situation is preferable to one in which the predicted stress is lower than that determined experimentally, erroneous stress predictions can lead either to reliability degradation or to product overdesign.

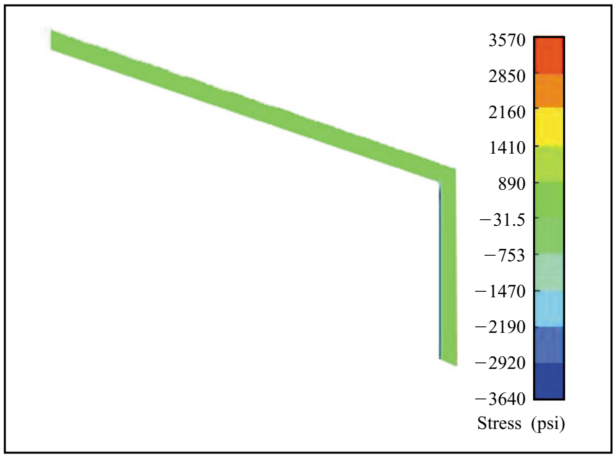

Figure 3

Shear stress in the *yz* plane of the epoxy sample at 25.5°C.

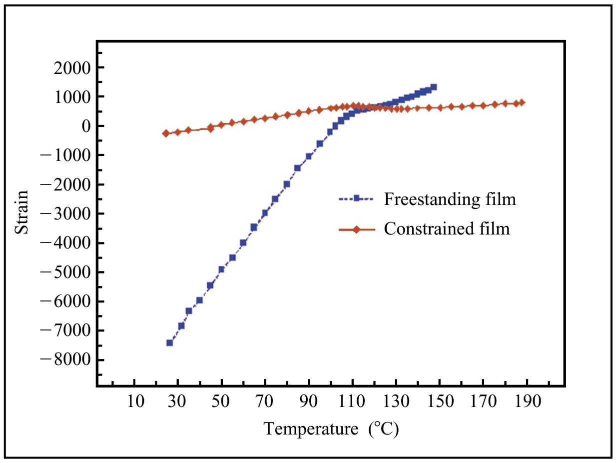

Figure 4

Strain of free and constrained 3M AHS-207 films of the cured epoxy as a function of temperature.

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