

8/11/91 JGD D

11/11/91

SANDIA REPORT

SAND90-0294 • UC-704
Unlimited Release
Printed November 1991

An Experimental/Analytical Comparison of Strains in Encapsulated Assemblies

T. R. Guess, S. N. Burchett

Prepared by
Sandia National Laboratories
Albuquerque, New Mexico 87185 and Livermore, California 94550
for the United States Department of Energy
under Contract DE-AC04-76DP00789

Issued by Sandia National Laboratories, operated for the United States Department of Energy by Sandia Corporation.

NOTICE: This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government, any agency thereof or any of their contractors or subcontractors. The views and opinions expressed herein do not necessarily state or reflect those of the United States Government, any agency thereof or any of their contractors.

Printed in the United States of America. This report has been reproduced directly from the best available copy.

Available to DOE and DOE contractors from
Office of Scientific and Technical Information
PO Box 62
Oak Ridge, TN 37831

Prices available from (615) 576-8401, FTS 626-8401

Available to the public from
National Technical Information Service
US Department of Commerce
5285 Port Royal Rd
Springfield, VA 22161

NTIS price codes
Printed copy: A03
Microfiche copy: A01

SAND--90-0294

DE92 002838

SAND90-0294
Unlimited Release
Printed November 1991

An Experimental/Analytical Comparison of Strains in Encapsulated Assemblies

T. R. Guess
Organic Materials Division
S. N. Burchett
Engineering and Structural Mechanics Division
Sandia National Laboratories
Albuquerque, New Mexico 87185

Abstract

A combined experimental and analytical study of strains developed in encapsulated assemblies during casting, curing and thermal excursions is described. The experimental setup, designed to measure in situ strains, consisted of thin, closed-end, Kovar* tubes that were instrumented with strain gages and thermocouples before being over-cast with a polymeric encapsulant. Four bisphenol A (three diethanolamine cured and one anhydride cured) epoxy-based materials and one urethane elastomeric material were studied. After cure of the encapsulant, tube strains were measured over the temperature range of -55°C to 90°C. The thermal excursion experiments were then numerically modeled using finite element analyses and the computed strains were compared to the experimental strains. The predicted strains were over estimated (conservative) when a linear, elastic, temperature-dependent material model was assumed for the encapsulant and the stress free temperature T_i was assumed to correspond to the cure temperature T_c of the encapsulant. Very good agreement was obtained with linear elastic calculations provided that the stress free temperature corresponded to the onset of the glassy-to-rubbery transition range of the encapsulant. Finally, excellent agreement was obtained in one of the materials (828/DEA) when a viscoelastic material model was utilized and a stress free temperature corresponding to the cure temperature was assumed.

* "Kovar" is a registered trademark of Carpenter Technology Corporation

MASTER

Contents

Introduction	9
Experimental Procedure	13
Finite Element Model	15
Encapsulating Materials	17
Results and Discussion	21
Validation of Test Procedure	21
Potting and Cure	21
Thermal Excursions	22
Kovar Encapsulated in 828/DEA	22
Kovar Encapsulated in 828/CTBN/DEA	29
Kovar Encapsulated in 828/CTBN/GMB/DEA	29
Kovar Encapsulated in SRIR	29
Kovar Encapsulated in Adiprene L-100/BD/TMP	29
Comments	33
Summary	35
References	37
Acknowledgments	39

Tables

1	Materials Studied and Governing Process Specifications	17
2	Temperature-Dependent Material Properties	20
3	Room Temperature Residual Strains in Kovar Tube Following Cure of Encapsulant	23

Figures

1	Analysis of Encapsulated Assemblies	10
2	Experimental Configuration	14
3	Finite Element Idealization of Experiment	15
4	Dynamic Shear Modulus Measured at 2 kHz	18
5	Free Expansion of Non-encapsulated Kovar Tube	22
6	828/DEA - Potting and Cure Strains	23
7	828/DEA - Axial Strain in Kovar - Elastic Analysis - Stress Free Temperature = $T_i = 71^\circ\text{C} = T_c$	24
8	828/DEA - Hoop Strain in Kovar - Elastic Analysis - Stress Free Temperature = $T_i = 71^\circ\text{C} = T_c$	24
9	828/DEA - Axial Strain in Kovar - Viscoelastic Analysis - Stress Free Temperature = $T_i = 71^\circ\text{C} = T_c$	26
10	828/DEA - Hoop Strain in Kovar - Viscoelastic Analysis - Stress Free Temperature = $T_i = 71^\circ\text{C} = T_c$	26
11	828/DEA - Axial Strain in Kovar - Elastic Analysis - Stress Free Temperature = $T_i = 55^\circ\text{C}$	27
12	828/DEA - Hoop Strain in Kovar - Elastic Analysis - Stress Free Temperature = $T_i = 55^\circ\text{C}$	27
13	828/CTBN/DEA - Axial Strain in Kovar - Elastic Analysis - Stress Free Temperature = $T_i = 50^\circ\text{C}$	28
14	828/CTBN/DEA - Hoop Strain in Kovar - Elastic Analysis - Stress Free Temperature = $T_i = 50^\circ\text{C}$	28
15	828/CTBN/GMB/DEA - Axial Strain in Kovar - Elastic Analysis - Stress Free Temperature = $T_i = 60^\circ\text{C}$	30
16	828/CTBN/GMB/DEA - Hoop Strain in Kovar - Elastic Analysis - Stress Free Temperature = $T_i = 60^\circ\text{C}$	30
17	SRIR - Axial Strain in Kovar - Elastic Analysis - Stress Free Temperature = $T_i = 20^\circ\text{C}$	31
18	SRIR - Hoop Strain in Kovar - Elastic Analysis - Stress Free Temperature = $T_i = 20^\circ\text{C}$	31
19	Adiprene - Axial Strain in Kovar - Elastic Analysis - Stress Free Temperature = $T_i = 140^\circ\text{C} = T_c$	32
20	Adiprene - Hoop Strain in Kovar - Elastic Analysis - Stress Free Temperature = $T_i = 140^\circ\text{C} = T_c$	32

Introduction

The design of weapons component assemblies is often critically dependent upon the appropriate use of polymeric encapsulating materials. The encapsulation provides sensitive electrical assemblies with protection against severe shock, vibration, and atmospheric environments. The materials also provide, in many instances, high voltage protection and isolation. Due to substantial differences in thermal expansion between electronic assemblies and the encapsulation materials, however, detrimental stresses and deformations can be developed in the encapsulation process and subsequent thermal excursions. To assist in design and evaluation, accurate predictions of the states of stress and strain in encapsulated assemblies are needed. Finite element computations can yield the desired results provided there are proven material models and accompanying thermomechanical material properties to input into the models.

The states of stress and strain in encapsulated assemblies have been numerically predicted for many years using finite element analyses. These calculations, however, were based on critical assumptions about stress-free conditions in the assembly and material behavior. When a polymeric material cures, complicated chemical reactions occur that produce substantial volume reductions (cure shrinkage) in the encapsulant. The first assumption was that the encapsulation material was stress free and geometrically sound after the cure, i.e., the response during the cure process was neglected. Secondly, to determine the response due to temperature excursions (thermal shrinkage), a stress free temperature T_i was assumed. The assumed stress free temperature was either the cure temperature T_c or the glass-transition temperature of the encapsulant T_g . Finally, the stresses in the assembly were normally calculated assuming linear, elastic, temperature dependent material response for the encapsulant; however, in some isolated instances nonlinear (elastic plastic) or viscoelastic material models were employed. It was believed that the stress state predicted with these assumptions were conservative (higher than actual). The predicted responses qualitatively agreed with observed behavior and when the analyses were used to compare various designs, valuable insights were obtained.

More rigorous analyses of encapsulated assemblies must account for both the stresses developed during cure and the thermal and environmental stresses developed after the material cures [1]. At the present time, the development of this rigorous capability is proceeding along parallel paths as schematically illustrated in Figure 1 [2]. Along one path research is focused on developing a methodology to predict curing response in polymeric encapsulants [3]. Before the problem of computing cure-shrinkage stresses in non-isothermally cured encapsulants can be solved, a reaction kinetics and heat transfer model ("thermal model") must first be developed and used to provide both the extent of reaction (conversion) and the temperature as functions of time and position. In the ab-

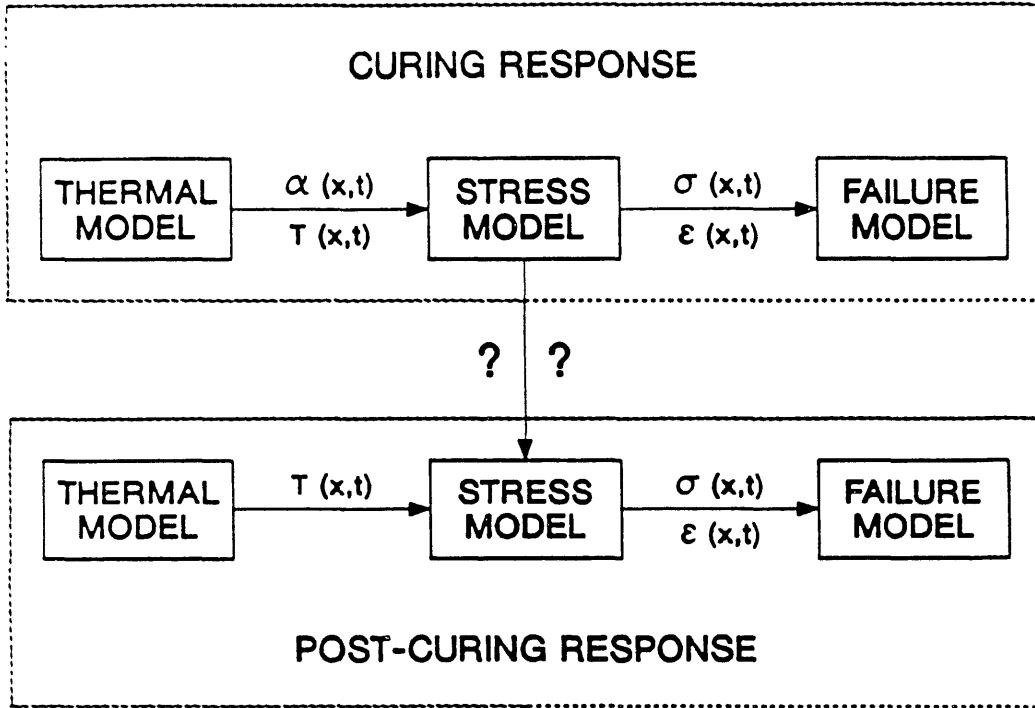


Figure 1. Analysis of Encapsulated Assemblies

sence of convection, the thermal model provides the initial conditions for a cure-shrinkage stress model. The development of the stress model involves four tasks: developing a material constitutive model, obtaining material constants, validating the material model, and deriving and implementing finite element solution procedures. Predicted stresses and strains from the cure-shrinkage stress model can then be used to examine possible failure modes during curing. The cure-shrinkage response will, at some point in time, provide the yet to be determined initial conditions for the post-cure analysis.

The second path of the development program involves studies to evaluate the current capability to predict stresses and strains in a fully cured material (post-cure response). Once this capability is established, the post-cure response of an encapsulated assembly due to thermal loading (from an appropriate thermal model) and mechanical loading can be confidently predicted. The predicted stresses and strains can then be used to determine possible service failure modes.

This report presents results from combined experimental/analytical research to evaluate the current capability for predicting post-cure stresses and strains for five encapsulating materials. The approach was (1) to perform experiments [4] that made direct in situ measurements of strains that developed in an embedded closed cylinder due to cooling from encapsulation temperatures and to subsequent thermal excursions, (2) to perform finite element stress analyses of the same experiments using published thermomechanical material properties for the encapsulants, and (3) to compare experimental strains to predicted strains from finite element calculations. The embedded closed cylinder was chosen for two reasons: first, the geometry is representative of many encapsulated assemblies,

and secondly, the cure shrinkage stresses are minimal. Good agreement would verify that the material constitutive model, the constitutive parameters, and the finite element modeling methodology are valid for calculating stress and strain states in encapsulated assemblies.

Experimental Procedure

One goal of the experimental phase of the study was to measure strains developed in encapsulated parts that are actually used in electronic assemblies of interest such as alumina ceramic vacuum tubes. Rather than initially attempt to instrument ceramic tubes, model specimens that readily lend themselves to experimental and analytical evaluation were designed and studied. The model specimens consisted of thin-wall tubes as shown in Figure 2. Kovar tubes were used in order to achieve greater strain sensitivity than could be obtained with ceramic tubes. Kovar was chosen because it has about the same value of coefficient of thermal expansion (CTE) as alumina ceramic. However, a CTE match is not required to validate the models and the tube could have been one of any number of materials.

The Kovar tubes were instrumented with Micro-Measurements CEA-06-125WT-350 biaxial strain gages at the three locations shown in Figure 2: 0°, 90° and 180° on the inner diameter (ID). The gages were oriented to measure strain in the axial and hoop directions of the thin-wall tubes. The tubes were also instrumented with a Micro-Measurement strain gage temperature sensor and a T-type thermocouple near the 270° ID position. Strain gage and thermocouple leads passed through the center of the top end cap. The wire opening was sealed with RTV silicone to prevent the encapsulant from filling the tube interior during the potting step. A threaded bolt through the bottom end cap was used to position the tube in a mold-released aluminum mold as illustrated in Figure 2. The mold-release was required to prevent constraint of the encapsulant by adhesion to sides of the aluminum mold. After the assembly was cured and cooled to room temperature, the aluminum mold was removed.

Three types of experiments were conducted on each tube. Strains and temperatures were monitored (1) during free thermal expansion of a non-encapsulated tube over the temperature range of -60°C to 121°C, (2) during the potting and cure cycle, and (3) after removal from aluminum mold, during thermal cycling of the encapsulated tube between -60°C and 90°C. All parts were in thermal equilibrium prior to each strain reading.

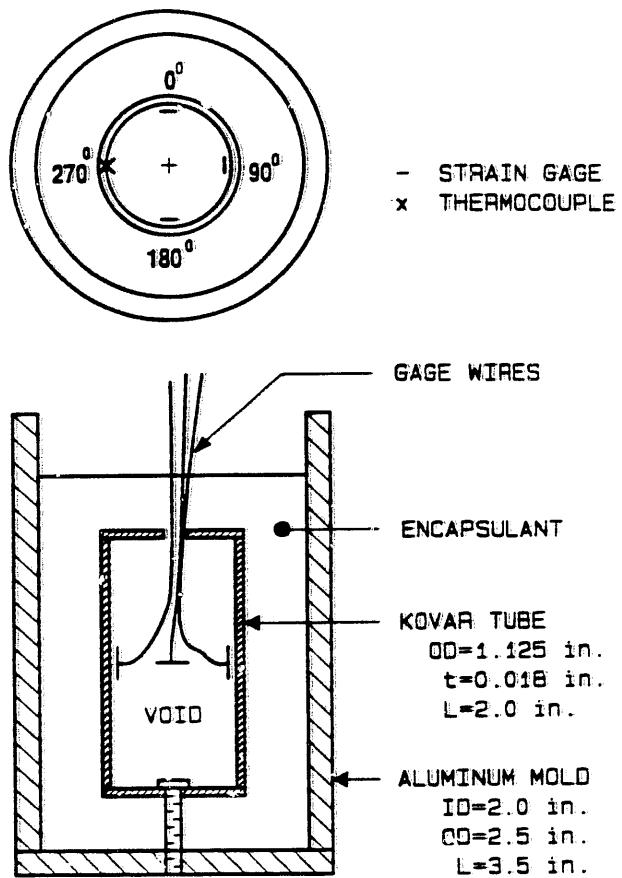


Figure 2. Experimental Configuration

Finite Element Model

Two-dimensional finite element analyses were used to predict the strains (and stresses) that developed in the fully cured encapsulated experimental assembly as the temperature of the assembly was cooled from the cure temperature. The two-dimensional geometry and finite element idealization used in this study (Kovar tube OD=1.125 in) is shown in Figure 3. This axisymmetric model takes advantage of a plane of symmetry but does not include the aluminum mold as it was assumed that the encapsulant did not adhere to the mold-released surface of the mold. To perform these calculations one would ideally like to assume the assembly was stress free at the cure temperature of the encapsulant and predict the stresses developed due to both cure shrinkage and thermal shrinkage. However, as stated earlier, the methodology to predict cure shrinkage effects is not complete but is the subject of active research. Therefore, various stress free temperatures were examined in this study. Originally, the stress free temperature was assumed to be the cure temperature T_c of the encapsulant. In subsequent calculations, the assumed stress free temperature was varied to obtain better correlation with experimental results. Thermoelastic calculations were performed using the finite element computer program JAC2D [5], and thermo-viscoelastic calculations were obtained with the finite element computer program SANTOS [6].

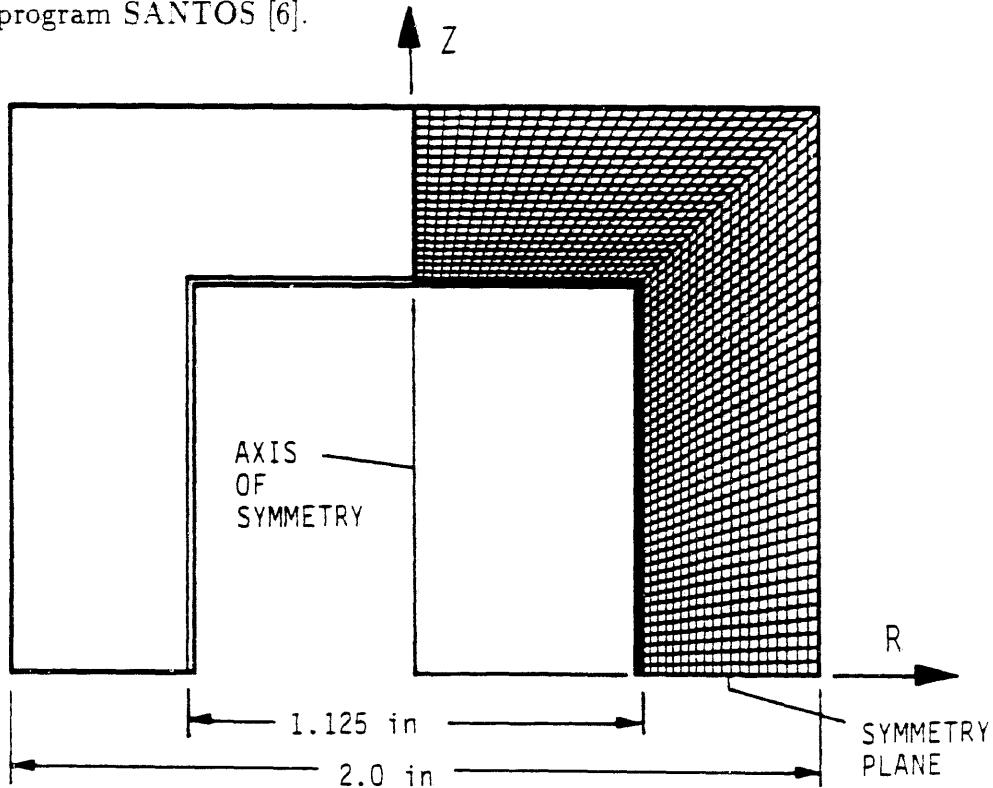


Figure 3. Finite Element Idealization of Experiment

Encapsulating Materials

The five encapsulants utilized in this study are listed in Table 1 along with process specification numbers and the cure temperatures used in the experiments. The encapsulants were 1) 828/DEA, an unfilled epoxy system, 2) 828/CTBN/DEA, a rubber toughened epoxy resin, 3) 828/CTBN/GMB/DEA, a rubber toughened epoxy filled with glass microballoons, 4) SRIR, an unfilled semi-rigid epoxy system, and 5) Adiprene L-100/BD/TMP, a polyurethane. A wide range of thermal and mechanical properties are represented by these encapsulating materials. The material descriptors above have the following meanings. The 828 designation refers to Shell's Epon 828 epoxy resin which is a formulation of diglycidyl ether of bisphenol A, DGEBA. DEA (diethanolamine) is a liquid curing agent that can be processed to have cure exotherms less than 90°C. CTBN is a carboxyl-terminated butadiene acrylonitrile rubber used to toughen epoxies. Glass microballoons (GMB) is a filler material that is used to reduce the weight and the coefficient of thermal expansion (CTE) of an encapsulating material without sacrificing other physical properties. Allied Signal's SRIR material is designated as a semi-rigid inspection resin. Because this two-part epoxy system is transparent, it can be used for inspection of encapsulated objects. And finally, Adiprene L-100 is Uniroyal's polyurethane that was cured with BD (1,4-butanediol) and TMP (trimethylolpropane) for 7 hours at 140°C.

Reference [7] data for the dynamic shear modulus and $\tan \delta$ of the 828/DEA material are plotted as a function of temperature in Figure 4. The data were measured at a frequency of 2 kHz using a Rheometrics Mechanical Spectrometer RDS-2 instrument. Of particular interest in this plot is the zone in which the shear modulus changes drastically

Table 1. Materials Studied and Governing Process Specifications

	Process Specification	Cure Temperature (C)
828/DEA	9927019	71
828/CTBN/DEA	SS331645 (note 1)	71
828/CTBN/GMB/DEA	9927092 (note 2)	93
SRIR	9927014	71
Adiprene L-100/BD/TMP	(note 3)	140

Note 1:GE Process Specification for 828/CTBN/DEA

Note 2:Cured at higher temperature than specified in 9927092

Note 3:Processed per instructions on GE Drawings 3021918-200 for MC2993

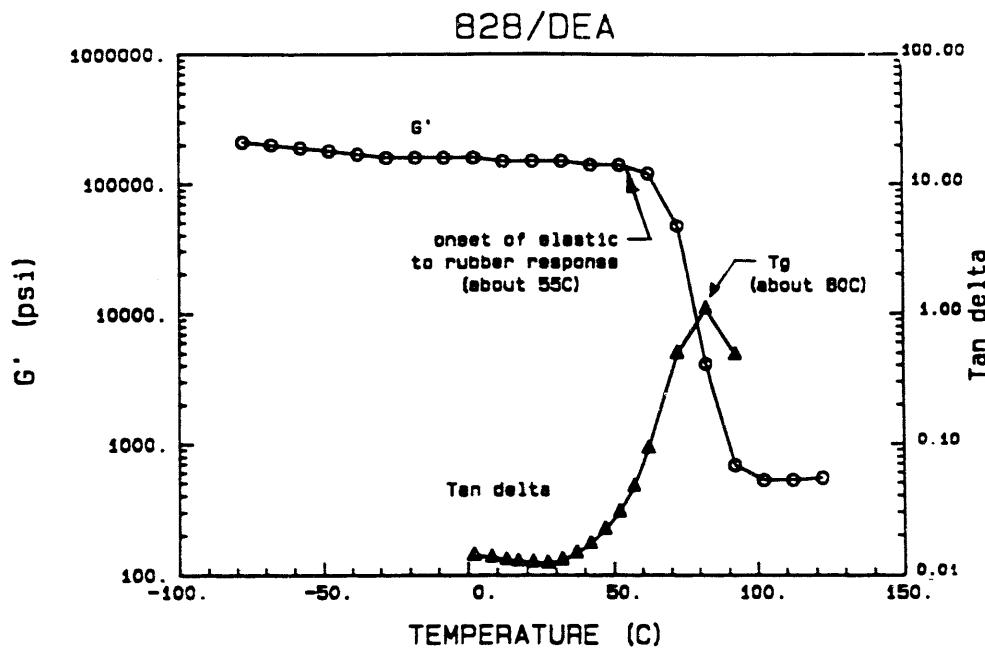


Figure 4. Dynamic Shear Modulus Measured at 2 kHz

as the temperature increases. This is a transition zone in which the material changes from a glassy to a rubbery solid. Various types of tests and methods of data interpretation are used to define the glass transition temperature T_g from the range of temperatures in the transition zone. Thus, the value assumed for T_g is dependent on several factors such as test method, rate of heating, frequency, etc, and is not a unique value. For example, T_g can be defined from Figure 4 as the temperature at which the $\tan \delta$ curve peaks.

The cure temperature is normally assumed to be the stress free temperature in an elastic analysis to determine the stresses developed in an encapsulated assembly as the temperature is cooled from the cure temperature. One could, however, define the stress free temperature in other ways, such as the glass transition temperature or the onset of the transition zone from the glassy state to the rubbery state.

Table 2 lists the elastic and thermal expansion properties of the five encapsulants used in the finite element analyses. The elastic properties for 828/DEA, 828/CTBN/DEA, and 828/CTBN/GMB/DEA were obtained from Adolf et al [7]. The elastic properties for SRIR were taken from the Casting Resins Property Chart [8] and those for Adiprene L100/BD/TMP from Holten [9]. The thermal expansion data for the 828/DEA, 828/CTBN/DEA, 828/CTBN/GMB/DEA, and SRIR were generated by Adolf and Chil-dress [10]. For the Table 2 data, thermal strain is defined as the free thermal expansion due to the coefficient of thermal expansion of the material, with -55°C arbitrarily set as zero strain. In the finite element analysis codes, only differences in thermal strain between two temperatures is considered, not the absolute value. Therefore, any temperature can be selected as the "zero strain" temperature as the strains at other temperatures are

adjusted appropriately.

The elastic material model and properties presented do not account for time dependent response. However, polymeric materials do exhibit time dependent response. Krieg and Cessac [11] approximated the response of 828/DEA as a thermorheologically simple linear viscoelastic material and used a nine term Prony series to define the shear relaxation modulus. In SANTOS, a linear viscoelastic material model implemented by Costin and Stone [12] is used. Using data from Krieg and Cessac, the material constants for 828/DEA were determined. The time-temperature shift function is given by a common WLF equation [13]

$$\log_{10}\alpha = \frac{C_1(T - T_0)}{C_2 + T_0 - T} \quad (1)$$

where

$$C_1 = 9.85 \quad (2)$$

$$C_2 = 60.0 \quad (3)$$

and

$$T_0 = 50.0^\circ C. \quad (4)$$

The shear modulus is given by:

$$G(t) = G^\infty + \sum_{i=1}^3 G_i e^{t/\beta_i} \quad (5)$$

where

$$G_1 = 50350 \text{ psi} \quad (6)$$

$$G_2 = 64309 \text{ psi} \quad (7)$$

and

$$G_3 = 53277 \text{ psi}. \quad (8)$$

The shear relaxation constants are given by:

$$\beta_1 = 6.342 \quad (9)$$

$$\beta_2 = 0.41 \quad (10)$$

and

$$\beta_3 = 0.0195. \quad (11)$$

The bulk response is assumed to be elastic with bulk modulus of

$$K = 740000 \text{ psi}. \quad (12)$$

Table 2. Temperature-Dependent Material Properties

	Temperature (C)	Modulus (psi)	Poisson's Ratio	Thermal Strain
828/DEA	-55.	517400.	0.383	0.0
	-30.	447700.	0.399	0.00113
	20.	421500.	0.405	0.00384
	50.	395000.	0.411	0.00576
	60.	352000.	0.423	0.00650
	71.	138400.	0.470	0.00762
	93.	2070.	0.499	0.01120
828/CTBN/DEA	-55.	454800.	0.385	0.0
	-30.	392600.	0.402	0.00160
	20.	339700.	0.415	0.00480
	50.	282400.	0.432	0.00725
	60.	243900.	0.441	0.00808
	71.	143500.	0.464	0.00900
	93.	21600.	0.499	0.01300
828/CTBN/GMB/DEA	-55.	491600.	0.347	0.0
	-30.	438600.	0.355	0.00084
	20.	409100.	0.363	0.00275
	50.	384100.	0.372	0.00405
	60.	363900.	0.380	0.00453
	71.	333300.	0.389	0.00522
	93.	38660.	0.487	0.00726
SRIR	-55.	550000.	0.350	0.0
	0.	325000.	0.400	0.003315
	20.	238200.	0.450	0.004835
	40.	15000.	0.490	0.007435
Adiprene	-55.	16000.	0.400	0.0
	-50.	2500.	0.425	0.000972
	-40.	470.	0.450	0.002915
	-23.	470.	0.480	0.006160
	4.	400.	0.490	0.011540
	38.	440.	0.490	0.018070
	140.	750.	0.490	0.037900

Results and Discussion

Validation of Test Procedure

In the first set of experiments, the apparent strain as a function of temperature was monitored for non-encapsulated Kovar tubes. This apparent strain ϵ_{a1} is the sum of 1) the strain ϵ_{Kovar} in the Kovar due to its coefficient of thermal expansion (CTE), and 2) the temperature-dependent apparent strain output of the strain gage ϵ_g . Experimental values of ϵ_{a1} were approximately identical in both the axial and hoop directions for the Kovar tubes. At each test temperature the manufacturer's value of ϵ_g was algebraically subtracted from the measured value of ϵ_{a1} (average of the three strain gages) to yield measured value of ϵ_{Kovar} , the free expansion/contraction strain in the Kovar due to its CTE.

Figure 5 is a plot of ϵ_{Kovar} in the axial direction of the Kovar tube as a function of temperature, with the data arbitrarily shown to have zero strain at 20°C. The solid curve is a quadratic least squares fit ($\epsilon_{Kovar} = -0.007011 \times T^2 + 6.1904 \times T - 160.0$) of the experimental data. A secant curve between -40°C and 100°C has a slope ($5.77 \times 10^{-6} \text{ in/in/}^\circ\text{C}$) that is in excellent agreement with literature values of Kovar CTE. These results indicate that the experimental and data reduction procedures are valid for measuring strains over the temperature range of interest.

Potting and Cure

Instrumented Kovar tubes with Kovar end caps were placed in mold-released aluminum molds and encapsulated. Apparent strains of the encapsulated tubes, ϵ_{a2} , were monitored during the potting and cure cycle. Thermal strains in the Kovar were obtained by subtracting ϵ_g from ϵ_{a2} . Representative results are shown in Figure 6 for the 828/DEA encapsulant over the 1.125" OD Kovar tube. The maximum oven temperature during cure was 71°C; however, during the exothermic reaction the resin temperature reached 90°C.

The solid line in Figure 6 is the free expansion of the Kovar from Figure 5. The strain gages were zeroed at 20°C at the start of the experiment. At the cure temperature both the axial and hoop strains in the 828/DEA encapsulated Kovar tube are equal to the strain in a non-encapsulated Kovar tube. Whenever the encapsulated tube response is the same as the non-encapsulated Kovar tube, there is no mechanically induced strain in the Kovar due to differences in CTE of the various materials. During cool-down from 71°C, the cure temperature, the experimental strains begin to deviate from the Kovar curve. This means that thermal stresses are beginning to develop in the encapsulant and

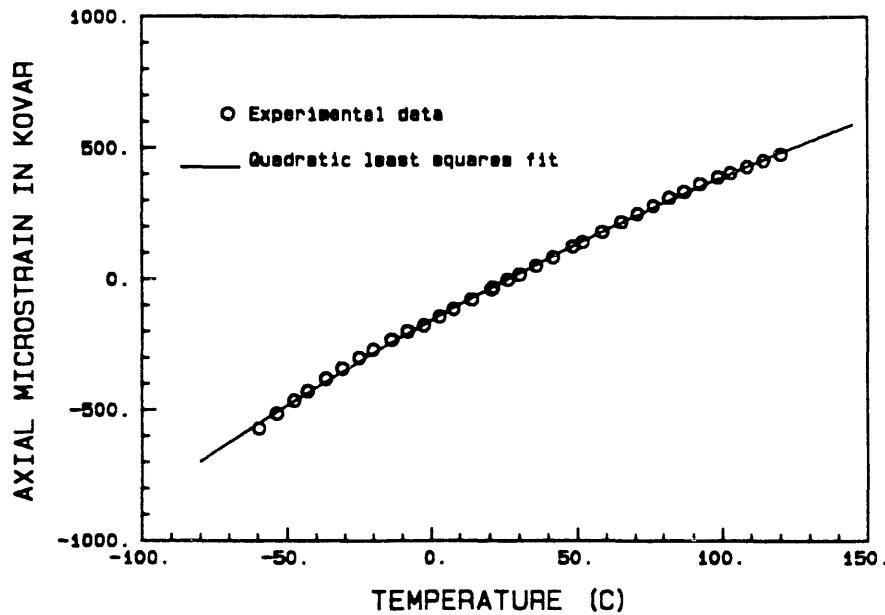


Figure 5. Free Expansion of Non-encapsulated Kovar Tube

in the Kovar tube due to the CTE mismatch between the two materials. The temperature at which the strains begin to build appears to be about equal to the temperature for the onset of the glassy-to-rubbery transition region for the potting material. The lowest temperature at which there is no mechanically induced strain in the Kovar was an important consideration for setting stress free temperatures in the finite element models of the experiments.

The mechanically induced residual compressive strain in the Kovar at room temperature following the cure cycle is equal to the difference in the curves for non-encapsulated (free expansion) and encapsulated tubes. A comparison of the experimental values of residual strains in the Kovar tubes following potting and cure of the five encapsulants is shown in Table 3. The fact that there are significant residual strains at room-temperature for DEA cured epoxies and none for the SRIR and Adiprene is a direct result of the differences among glass transition temperatures of the encapsulants.

Thermal Excursions

Kovar Encapsulated in 828/DEA

The Kovar tube potted in cured 828/DEA was thermal cycled between -60°C and 90°C. Apparent strains in the Kovar were corrected for ϵ_g , as previously described for the cure cool-down experiments, to yield a combined mechanical plus thermal strain.

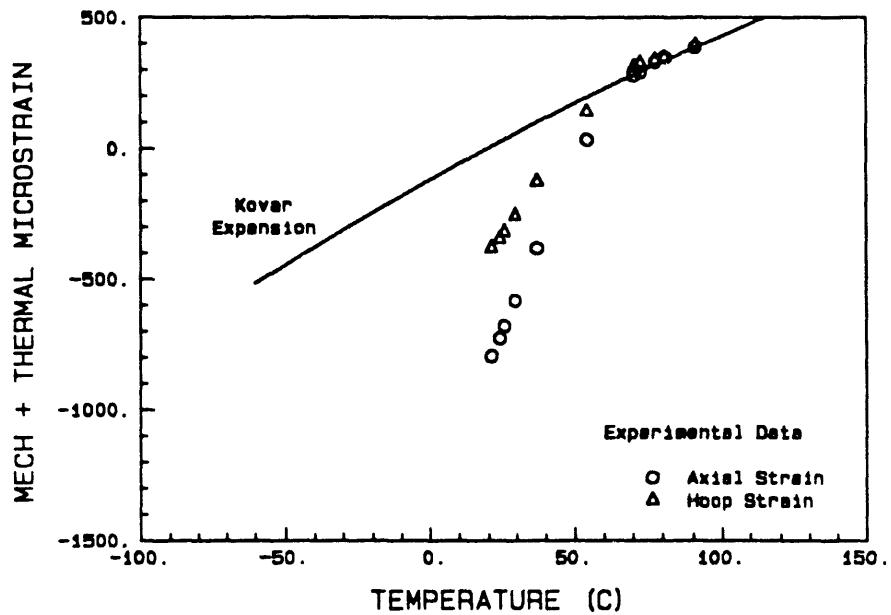


Figure 6. 828/DEA - Potting and Cure Strains

Table 3. Room Temperature Residual Strains in Kovar Tube Following Cure of Encapsulant

	Residual Axial Microstrain	Residual Hoop Microstrain
828/DEA	-765.	-380.
828/CTBN/DEA	-630.	-320.
828/CTBN/GMB/DEA	-540.	-230.
SRIR	0.	0.
Adiprene L-100/BD/TMP	0.	0.

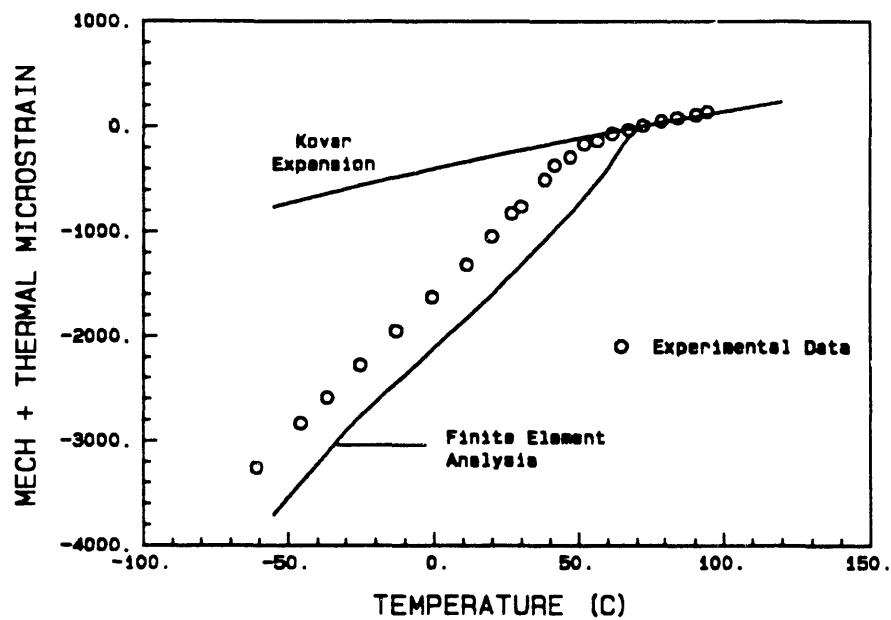


Figure 7. 828/DEA - Axial Strain in Kovar - Elastic Analysis - Stress Free
Temperature = $T_i = 71^\circ\text{C} = T_c$

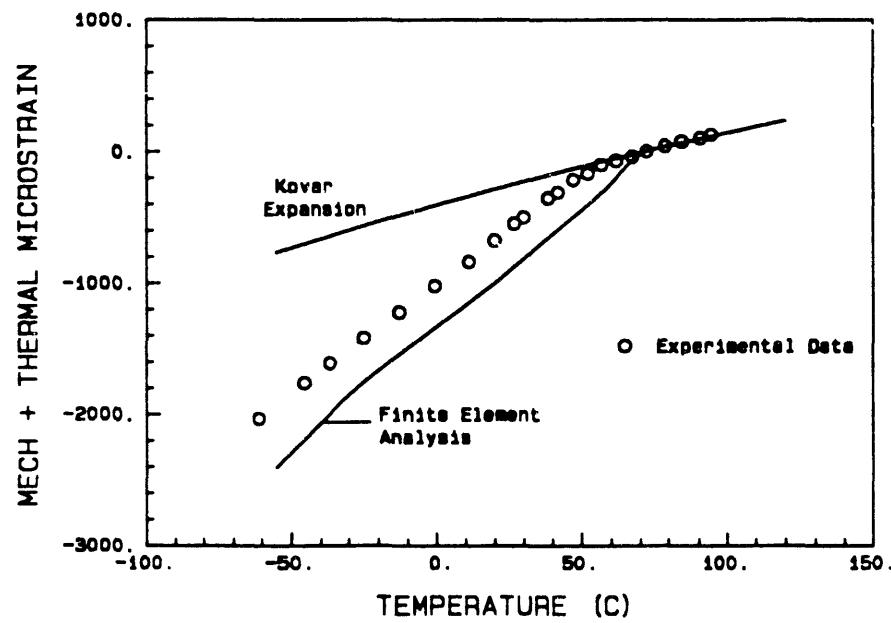


Figure 8. 828/DEA - Hoop Strain in Kovar - Elastic Analysis - Stress Free
Temperature = $T_i = 71^\circ\text{C} = T_c$

Elastic Analysis with T_c as Stress Free Temperature - Figures 7 and 8 show the axial and hoop strains, respectively, as functions of temperature, for a Kovar tube potted in 828/DEA. The experimental data (open circles) are set to zero at 71°C, the cure temperature. Included in these two figures are curves for the free expansion of a non-encapsulated Kovar tube and for finite element analyses of the experimental assembly. The material model incorporated in the analysis is a linear, elastic, temperature-dependent model using the properties listed in Table 2. The stress free temperature is assumed to equal the cure temperature of the 828/DEA. The slopes of computed and measured strain-temperature curves are in reasonable agreement. However, at -50°C the finite element analysis over estimates the compressive mechanical strains by about 25% in the axial direction and 40% in the hoop direction.

Viscoelastic Analysis with T_c as Stress Free Temperature - Strains computed with the previously described viscoelastic material model for 828/DEA are shown in Figures 9 and 10. These figures also include the experimental data and the free expansion strains of Kovar. Calculated viscoelastic responses are plotted for two rates of cooling. The assembly was first assumed to be uniformly cooled from the cure temperature to the cold temperature extreme in two hours and, secondly, in four hours. The calculated results are not significantly affected by the two rates of cooling assumed in these calculations. Near the stress free temperature, the viscoelastic calculations produce a nonlinear response that is similar to the nonlinear response observed in the experiment. The calculated Kovar strains are in excellent agreement with experimental data with a slightly better correlation for the hoop strains than for the axial strains.

Elastic Analysis with Onset of Glassy-to-Rubbery Transition Region as Stress Free Temperature - It is clear from Figures 7-10 that the Kovar tube encapsulated in 828/DEA begins to pick up mechanically induced strain when the temperature is lowered to about 55°C. In the shear modulus vs. temperature material tests by Adolf [7], the temperature at which the 828/DEA begins to transition from glassy to rubbery response also corresponds to about 55°C as shown in Figure 4. Finite element analyses with the linear, elastic, temperature-dependent material model for the 828/DEA potting material were performed for the condition of a 55°C stress free temperature. The results are shown in Figures 11 and 12. Excellent agreement is observed between the computed strains and the experimentally measured strains over the entire temperature range.

Summary - 828/DEA - In summary, three material models were used in finite element analyses to calculate strains in the 828/DEA encapsulated Kovar tube during thermal excursions. The results show that an elastic analysis yields excellent agreement with experimental data when the stress-free temperature is set equal to the temperature at which the Kovar tube begins to pick up mechanically induced strains in the tube experiments. This temperature corresponds to the onset of the glassy-to-rubbery transition temperature as indicated by dynamic shear modulus vs. temperature data. A viscoelastic

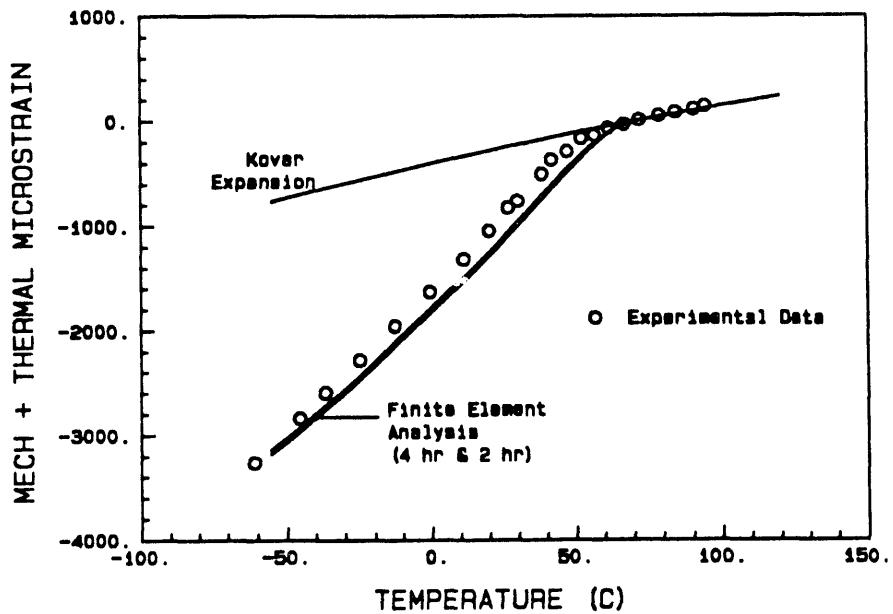


Figure 9. 828/DEA - Axial Strain in Kovar - Viscoelastic Analysis - Stress Free
Temperature = $T_i = 71^\circ\text{C} = T_c$

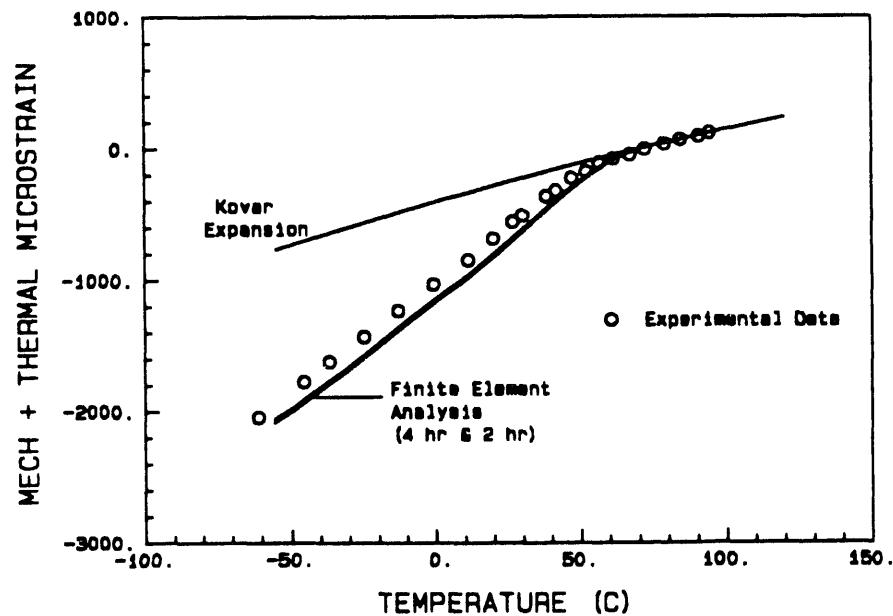


Figure 10. 828/DEA - Hoop Strain in Kovar - Viscoelastic Analysis - Stress Free
Temperature = $T_i = 71^\circ\text{C} = T_c$

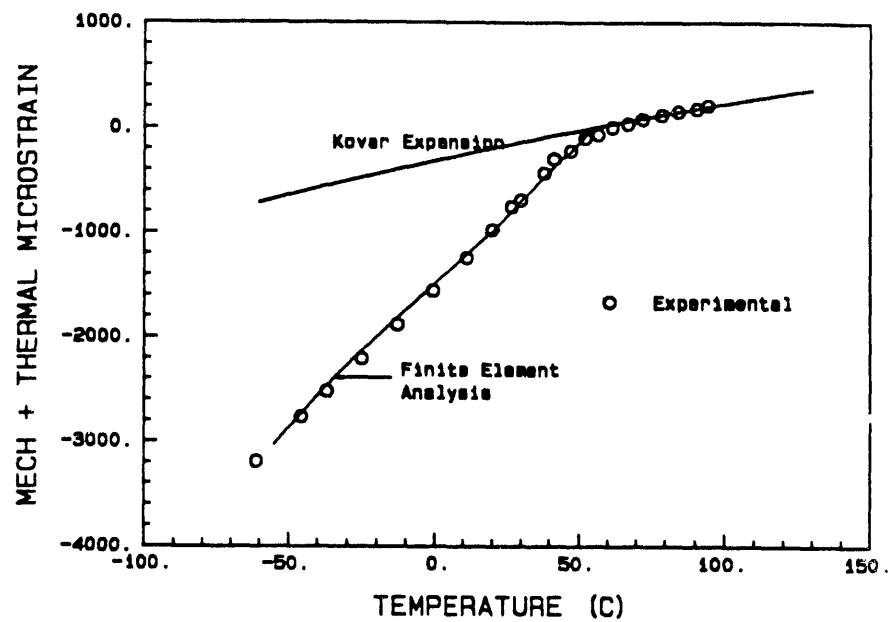


Figure 11. 828/DEA - Axial Strain in Kovar - Elastic Analysis - Stress Free Temperature = $T_i = 55^\circ\text{C}$

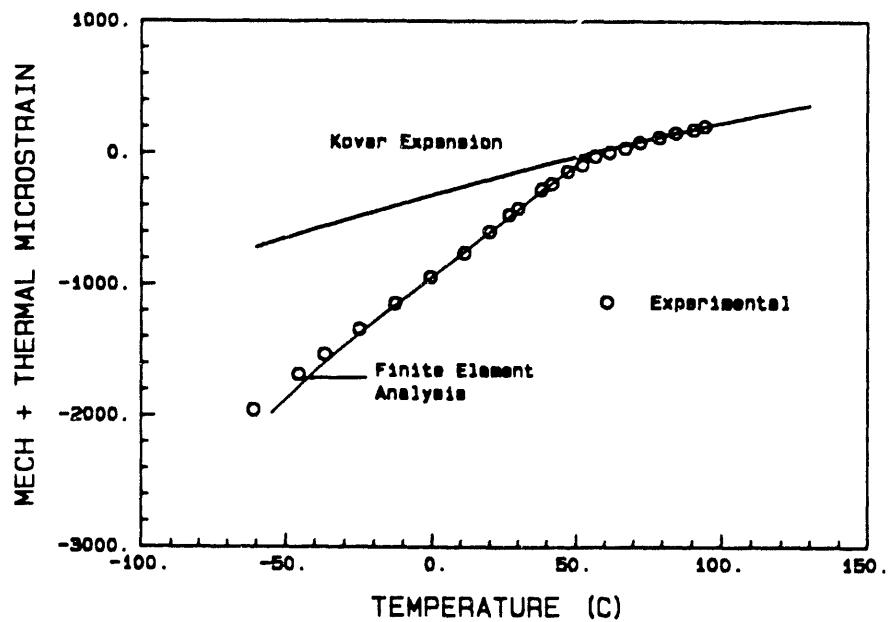


Figure 12. 828/DEA - Hoop Strain in Kovar - Elastic Analysis - Stress Free Temperature = $T_i = 55^\circ\text{C}$

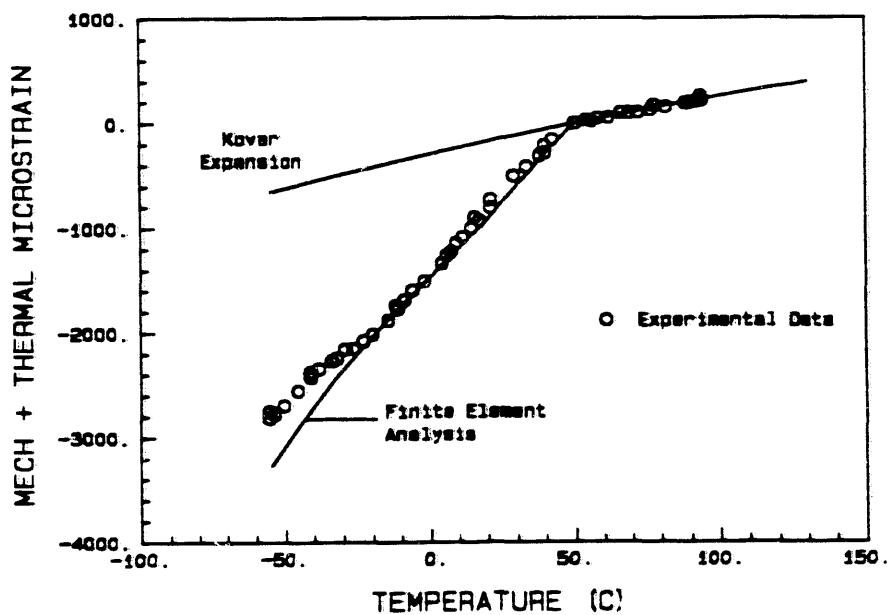


Figure 13. 828/CTBN/DEA - Axial Strain in Kovar - Elastic Analysis - Stress Free Temperature = $T_i = 50^\circ\text{C}$

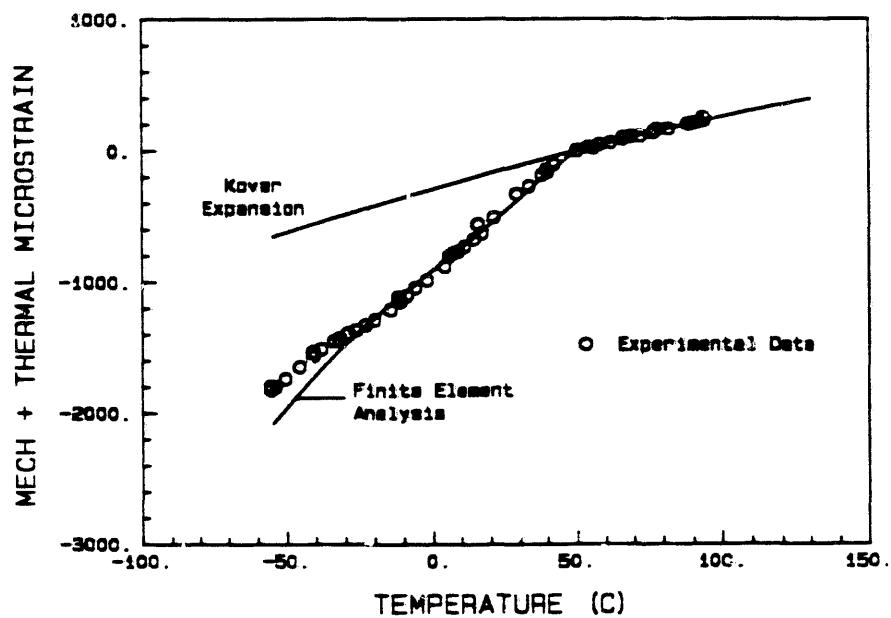


Figure 14. 828/CTBN/DEA - Hoop Strain in Kovar - Elastic Analysis - Stress Free Temperature = $T_i = 50^\circ\text{C}$

model with the cure temperature, T_c , as the stress free temperature also yielded excellent agreement with experiment. In addition this model reproduced the nonlinear behavior near the stress free temperature. Because viscoelastic properties are not presently available for the other four encapsulants, only elastic material properties with a stress free temperature taken from the experimental data were used in finite element analyses of Kovar tubes potted in the other four encapsulants.

Kovar Encapsulated in 828/CTBN/DEA

Comparisons of elastic analyses with experiment are shown in Figures 13 and 14 for the 828/CTBN/DEA potting. Based on the experimental data, the stress free temperature is taken to be 50°C. The finite element predictions are in good agreement with experimental data at all temperatures down to -30°C, below which the analysis overestimates the strain. The shape of the experimental curve at the low temperatures suggest that there may be a secondary glass transition in 828/CTBN/DEA near -30°C.

Kovar Encapsulated in 828/CTBN/GMB/DEA

Figures 15 and 16 show the experimental and analytical results for Kovar potted in 828/CTBN/GMB/DEA assuming a stress free temperature of 60°C for the finite element computations. The agreement is not quite as good as for the two previous encapsulants. The slope of the analytical curve is greater than the experimental curve. Thus, for the elastic analysis with a 60°C stress free temperature, the finite element analysis underestimates the Kovar strain at temperatures above 20°C and overestimates them below that temperature.

Kovar Encapsulated in SRIR

As noted in the material properties section, the most recent documented data for SRIR is from a 20 year old chart which listed elastic data at four temperatures. An elastic analysis based on these properties and a stress free temperature of 20°C are in good agreement with the experimental data over the entire temperature range as can be seen in Figures 17 and 18. The slopes of the computed curves are greater than the experimental curves. Therefore, at the low temperatures the finite element results are conservative, i.e., they overestimate the compressive stresses in the Kovar. Note again that there are no residual strains in the encapsulated tube at room temperature which is near the SRIR glass transition temperature.

Kovar Encapsulated in Adiprene L-100/BD/TMP

The comparison of the experimentally measured and computed strains for the urethane elastomer which was cured at 140°C and thermal cycled down to -80°C are shown in Figures 19 and 20. For the finite element calculations of this experiment, elastic,

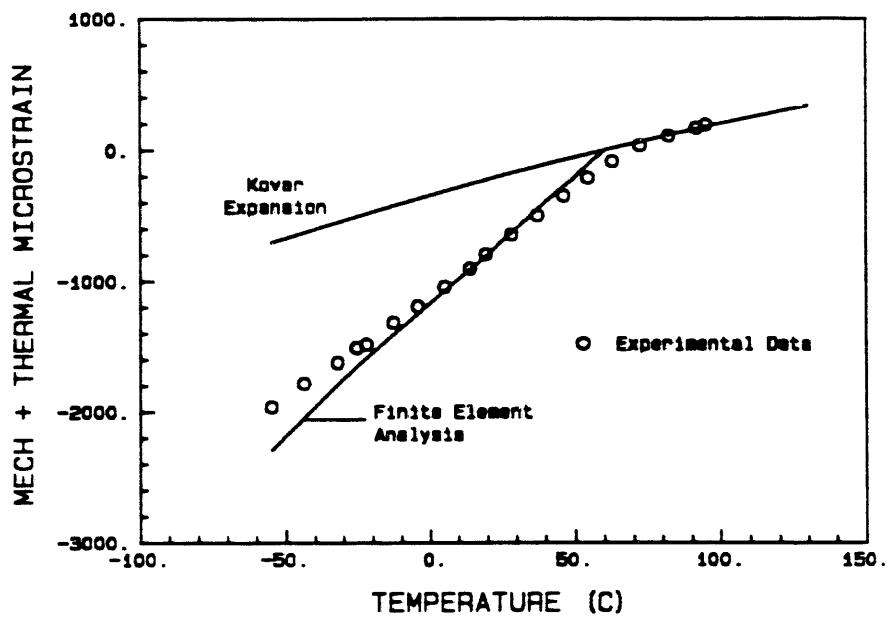


Figure 15. 828/CTBN/GMB/DEA - Axial Strain in Kovar - Elastic Analysis - Stress Free Temperature = $T_i = 60^\circ\text{C}$

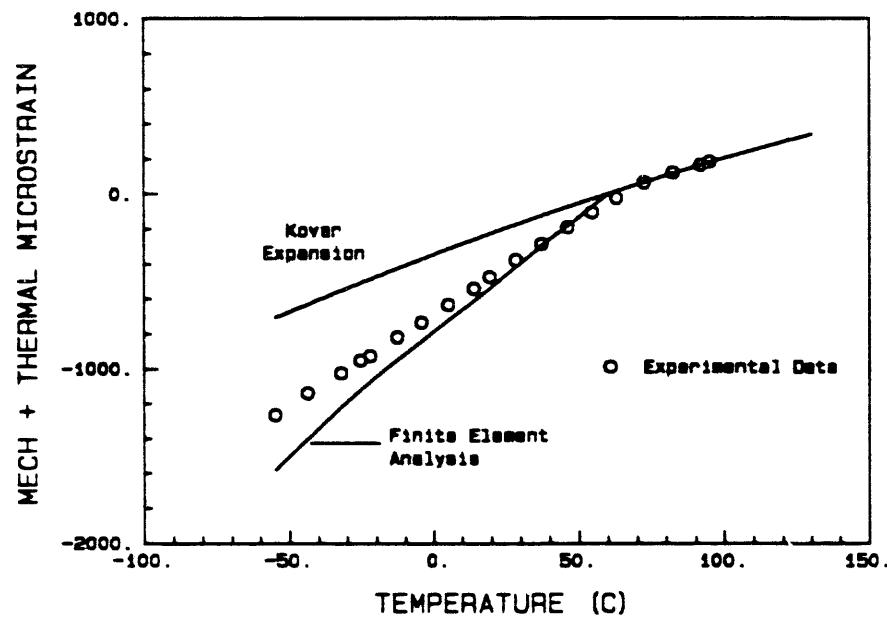


Figure 16. 828/CTBN/GMB/DEA - Hoop Strain in Kovar - Elastic Analysis - Stress Free Temperature = $T_i = 60^\circ\text{C}$

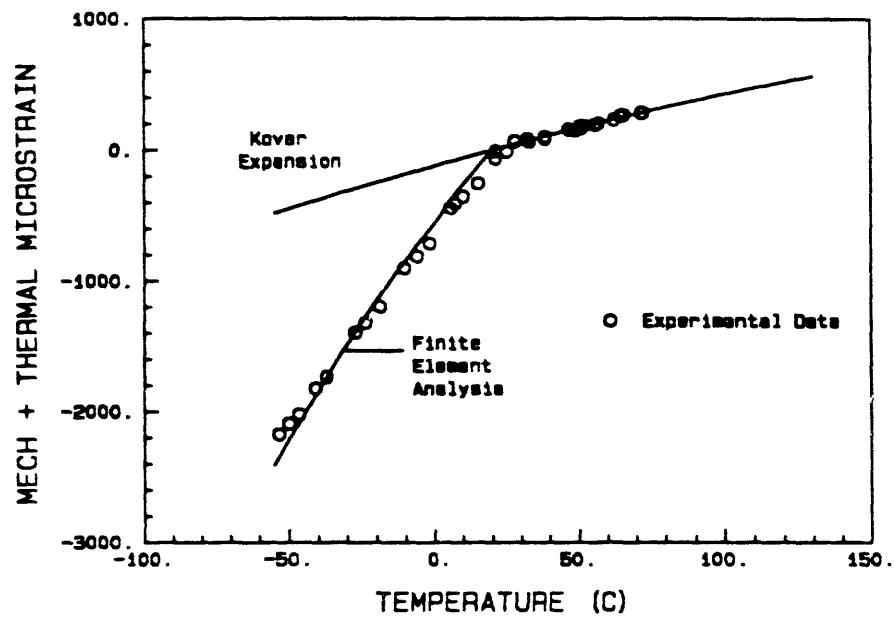


Figure 17. SRIR - Axial Strain in Kovar - Elastic Analysis - Stress Free Temperature = $T_i = 20^\circ\text{C}$

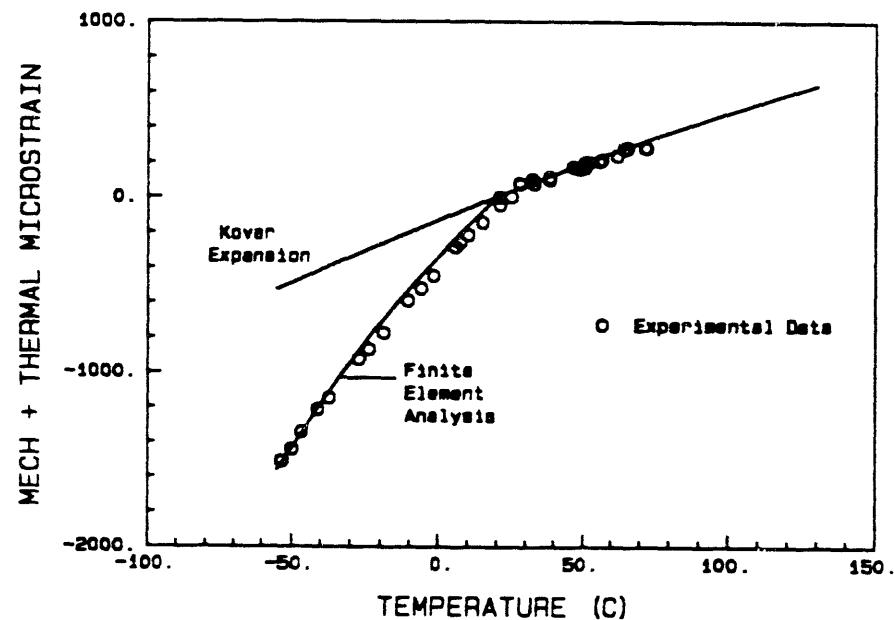


Figure 18. SRIR - Hoop Strain in Kovar - Elastic Analysis - Stress Free Temperature = $T_i = 20^\circ\text{C}$

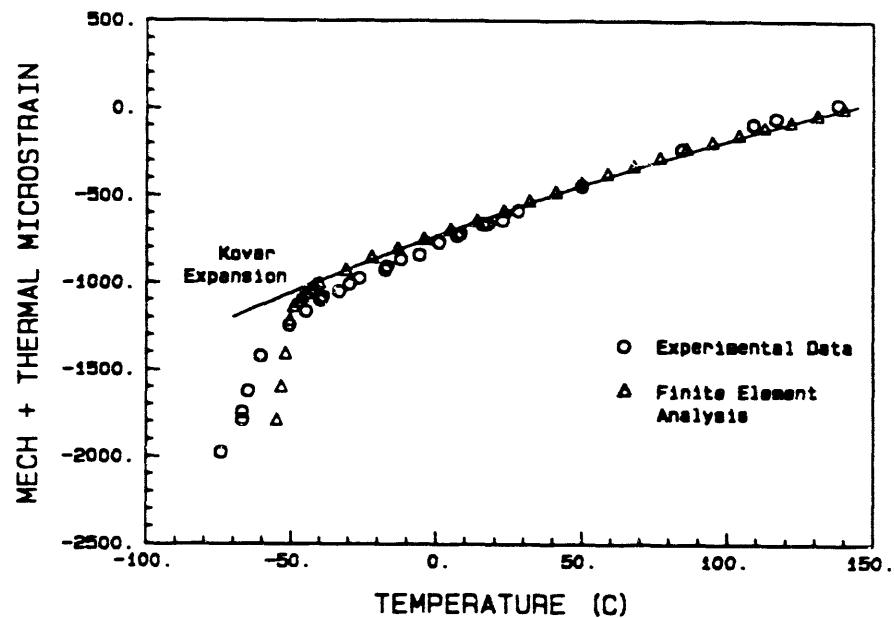


Figure 19. Adiprene - Axial Strain in Kovar - Elastic Analysis - Stress Free Temperature = $T_i = 140^\circ\text{C} = T_c$

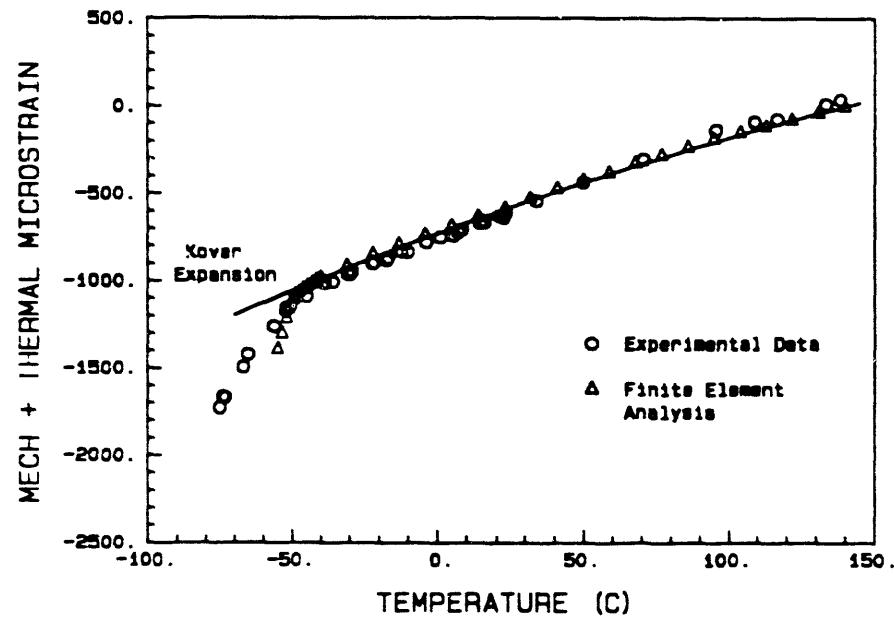


Figure 20. Adiprene - Hoop Strain in Kovar - Elastic Analysis - Stress Free Temperature = $T_i = 140^\circ\text{C} = T_c$

temperature-dependent properties were used while setting zero strain in the Kovar at the 140°C cure temperature. The computed strains agree with the free expansion strains of the Kovar down to a temperature of about -40°C, which is near the glass transition temperature of the material. Note that the experimental data deviate slightly from the Kovar free expansion curves, possibly due to experimental error. Once mechanical strains begin to build in the -40°C to -50°C range, the slopes of the experimental and analytical curves are significantly different with the computed strain overestimating those measured in the Kovar. However, at -55°C which is the low end of most temperature requirements, very little mechanical strain develops in the Kovar encapsulated in the urethane elastomer.

Comments

It is important to note that all experiments discussed in this paper were for Kovar tubes over-potted with encapsulants. The outside surfaces were not constrained during cure and thermal cycling because the aluminum mold was mold-released. For this condition, the volumetric shrinkage that occurs in polymers during gelation and cure does not contribute to the generation of mechanical strains in the Kovar. Volumetric shrinkage can be an important parameter when the resin is constrained during gelation and cure. An example of this condition is a thin tube filled with an encapsulant. When there is good adherence between the Kovar and the encapsulant, significant stresses and strains can develop in the tube and encapsulant during gelation and cure due primarily to volume shrinkage and the development of mechanical stiffness (shear modulus) during the gelation process. Experimental and modeling studies of thin tubes filled with 828/DEA have been reported elsewhere[3].

Summary

1. An experimental technique to measure in situ strains during potting, curing and thermal cycling of encapsulated assemblies has been developed and demonstrated.
2. The accuracy and validity of the experimental procedure were demonstrated by measuring the coefficient of thermal expansion of thin Kovar tubes. These results were in excellent agreement with literature values.
3. Kovar tubes were potted in five encapsulating resins (828/DEA, 828/CTBN/DEA, 828/CTBN/GMB/DEA, SRIR, and Adiprene L-100/BD/TMP) and monitored for strains during potting, cure, and thermal excursions. The epoxies with DEA produced residual compressive axial strains of $765 \mu\text{strain}$ in the Kovar at room temperature. This strain results because of the differential in coefficient of thermal expansion between the Kovar and the encapsulants and because the glass transition temperatures T_g of these potting materials are above room temperature. On the other hand, the SRIR and Adiprene do not produce room temperature residual strains in the Kovar because the glass transition temperatures are approximately equal to or below room temperature (25°C and -45°C , respectively).
4. Elastic and viscoelastic material properties were used in finite element models of the thermal excursion experiments of thin Kovar tubes encapsulated in 828/DEA. The elastic analysis yielded conservative results (predicted strains higher than measured strains) when the stress-free temperature was set equal to the cure temperature.

The elastic analysis yielded excellent agreement with experimental data over the entire temperature range provided the stress-free temperature was set equal to the temperature at which the Kovar tube began to pick up mechanically induced strains in the tube experiments. This temperature can be identified in material properties data, such as dynamic shear modulus vs temperature data by picking the temperature for the onset of the transition region from glassy response to the rubbery state. Since the dynamic shear modulus vs temperature curve is dependent on many factors such as test method, frequency, and rate of heating, it is difficult to obtain a unique value for the stress-free temperature. In these calculations the stress-free temperature in effect becomes a required material property value which must be obtained for an accurate analysis.

A viscoelastic analysis with T_g as the stress free temperature also yielded excellent agreement with experiment. In the viscoelastic analysis, however, it was not necessary to choose an "appropriate" stress-free temperature. The viscoelastic analysis reproduced the nonlinear behavior experimentally observed near the stress free temperature.

The experiments with 828/DEA suggest that it is not absolutely necessary to include

the viscoelastic behavior of epoxy potting materials to capture the principle features of the experimental results and to calculate strains (and stresses) in encapsulated components provided that the apparent stress-free temperature can be determined. However, a viscoelastic model is required to predict the nonlinear response that occurs near the stress free temperature or if stress-free temperature is unknown. Additionally, time dependent response such as the effect of long time storage can only be predicted with a viscoelastic model.

5. Viscoelastic properties were not available for the other four encapsulants. Therefore, finite element analyses of the experiments with these potting materials used elastic, temperature-dependent material properties along with the experimentally inferred stress-free temperature. In general there was good agreement between experimental and predicted strains in the Kovar over the entire temperature range. When the comparisons were not in exact agreement, the finite element results tended to be conservative, i.e., the strains in the Kovar were over estimated.

6. In the experiments described in this paper, the encapsulants were not constrained by the aluminum mold. Therefore, isothermal volume shrinkage of the resin during the gel process did not contribute to the build-up of Kovar strains. For hypothetical cases where potting materials are constrained, volume shrinkage can be important and the set of material properties presented and verified in this study may not be sufficient for an accurate finite element analysis.

7. The results of this study suggest that the thermomechanical, elastic properties listed in Table 2 accurately characterize the five encapsulants provided that the apparent stress-free temperature can be determined.

References

1. S. N. Burchett, 1521, "Encapsulant Materials Support - Stress Analysis Needs," memo to W. R. Leuenberger, 7484, Sandia National Laboratories, Albuquerque NM, March 23, 1988.
2. R. S. Chambers, R. R. Lagasse, and T. R. Guess, "A Cure Shrinkage Model for Analyzing the Stresses and Strains in Encapsulated Assemblies During Curing," SAND91-1829A, Sandia National Laboratories, Albuquerque, New Mexico, Submitted for presentation at the ASME/JSME Joint Conference on Electronic Packaging, April 9-12, 1992, San Jose, CA.
3. R. S. Chambers, 1523, T. R. Guess, 7472, and R. R. Lagasse, 1813, "Progress in the Development and Validation of a Material Model for Predicting Cure Shrinkage Effects in Thermoset Encapsulants," memo to Distribution, Sandia National Laboratories, Albuquerque NM, March 30, 1990.
4. T. R. Guess, 7472, "Proposed Experimental Studies Related to Neutron Generator Applications," memo to J. A. Sayre, 7472, Sandia National Laboratories, Albuquerque NM, January 12, 1988.
5. J. H. Biffle, "JAC - A Two-Dimensional Finite Element Computer Program for the Non-Linear Quasistatic Response of Solids with the Conjugate Gradient Method," SAND81-0998, Sandia National Laboratories, Albuquerque NM, April 1984.
6. C. M. Stone, "SANTOS A Two-Dimensional Finite Element Program for the Quasistatic, Large Deformation, Inelastic Response of Solids," SAND90-0543, Sandia National Laboratories, Albuquerque NM, in preparation.
7. D. Adolf, C. Childress, and D. Hannum, "Bulk and Shear Moduli of Epoxy Encapsulants," SAND89-0748, Sandia National Laboratories, Albuquerque NM, August 1989.
8. A. J. Quant and C. W. Hatcher, "Casting Resins Properties Chart," Sandia National Laboratories, Albuquerque NM, February, 1964.
9. D. I. Holton, "Evaluation of Alternate Cure Schedule for Adiprene L-100 Elastomer," General Electric Neutron Devices Department Technical Memorandum, No. 527, March 13, 1970.
10. D. Adolf and C. Childress, "Coefficients of Thermal Expansion of Common Encapsulants," SAND88-0777, Sandia National Laboratories, Albuquerque NM, July 1988.

11. R. D. Krieg, 1541, and G. L. Cessac, 2316, "Viscoelastic Properties of Epon 828/DEA," memo to Distribution, Sandia National Laboratories, Albuquerque NM, March 29, 1974.
12. L. S. Costin and C. M. Stone, "A Linear Viscoelastic Material Model for SANCHO," SAND85-1836, Sandia National Laboratories, Albuquerque NM, October, 1985.
13. M. L. Williams, R. F. Landel, and J. D. Ferry, "The Temperature Dependence of Relaxation Mechanisms in Amorphous Polymers and other Glass-Forming Liquids," J. Am. Chem. Soc. 77, 3701-3707, 1955.

Acknowledgments

G. D. Jones, 1812, and M. E. Stavig, 2472, were responsible for instrumenting kovar tubes with strain gages and thermocouples. They also participated in some of the thermal cycling tests. H. L. Anderson, Jr., 2472, encapsulated all units except those with the polyurethane elastomer which was potted by R. L. Myers, 2472. R. S. Chambers, 1514, and D. Adolf, 1813, reviewed and commented on the original manuscript.

Unlimited Release

Allied-Signal Aerospace Co. (3)	2400 W. E. Alzheimer
P. O. Box 419159	2410 T. J. Young
Kansas City, Mo 64141	2411 T. J. Young,actg.
Attn:	2413 G. L. Cessac
Jim Mahoney	2413 F. L. Gentry
Marc Shrank	2413 J. S. Arzigan
R S Sanders	2413 M. D. Lucas
General Electric Company (7)	2470 J. L. Ledman
P. O. Box 2908	2472 J. A. Sayre
Largo, FL 34292	2472 M. W. Donnelly
Attn:	2472 T. R. Guess (10)
E. L. Manson,D/100	2472 R. L. Myers
R. J. Antepenko,D/031	2472 K. B. Wischmann
T. Gillespie,D/048	2473 J. W. Munford
M. A. Merkel,D/049	2476 F. P Gerstle Jr
W. E. Swartz,Jr.,D/032	2484 R. C. Reuter Jr
B. J. Wells,D/033	2500 G. N. Beeler
1153 J. E. Martin	2560 J. T. Cutchin
1510 J. C. Cummings	2561 C. E. Spencer
1514 H. S. Morgan	2561 D. K. Morgan
1514 V. L. Bergmann	2561 W. E. Neuman
1514 S. N. Burchett (10)	2564 R. A. Damerow
1514 R. S. Chambers	2564 J. P. Brainard
1514 M. K. Neilsen	3141 S. A. Landenberger (5)
1514 C. M. Stone	3145 Document Processing for DOE/OSTI (8)
1514 G. W. Wellman	3151 G. C. Claycomb (3)
1540 J. R. Asay	8240 C. W. Robinson
1544 E. D. Reedy	8241 G. A. Benedetti
1544 G. D. Sjaardema	8242 M. R. Birnbaum
1550 C. W. Peterson	8243 M. L. Callabresi
1800 R. J. Eagan	8310 D. L. Linder
1810 D. W. Schaefer	8311 J. E. Costa
1812 C. L. Renschler	8311 R. E. Gott
1813 J. G. Curro	8314 M. W. Perra
1813 D. Adolf	8523-2 Central Technical Files
1813 P. B. Rand	
1813 R. R. Lagasse	
2361 T. J. Williams	
2364 J. F. Jones	

END

DATE
FILMED

12/31/91