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Model Validation Studies of 459 Epoxy

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Abstract

Neutron generator production is an important part of Sandia National Laboratories contribution to national security. Part of our responsibility in department 14172 is to develop and perform experiments that aid in the development of computer models used to test new neutron generator designs. Accurate models require good agreement between calculated results and experimental data, and this set of tests is another step in the process of providing experimental data to validate model calculations. In the past, verification studies have been performed on mold releases, material properties, and cure schedules. This set of tests is specifically designed to measure cure strains that develop as 459/GMB and 459/ALOX cures. The experimental variable was an altered cure schedule.

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Summary

Material and finite element models are being developed to analyze and predict the success of new neutron generator designs. This set of tests uses the Kovar tube test method to validate calculated cure strains that develop as formula 459 epoxy cures.

- Past experiments have utilized this test method to evaluate materials, mold releases and cure schedules.
- This set of experiments tested a cure cycle with a longer ramp time.
- The data collected from the Kovar tube experiments will be used to improve the accuracy of material and finite element models.

Model Validation Studies of 459 Epoxy

Introduction

Neutron Generator research and production are an important aspect of Sandia National Laboratories nuclear stewardship program. As new designs are produced new ways must be found to test the safety and reliability of the new design in a more cost-effective manner than building and testing a model. Material and finite element models can be used as cost-effective alternatives for testing new neutron generator designs. However, before models can be confidently used, their accuracy must be verified. This set of experiments is another step in a series of tests designed to bring calculated results and experimental data into agreement.

Context

The process of developing a model for predicting neutron generator reliability has been going on for some time. Six different encapsulants have been characterized and evaluated for their applicability. Formula 459 encapsulant with glass microballoon (GMB) or aluminum oxide (ALOX) fillers has been approved for certain neutron generator applications and different mold releases have been tested for their efficiency. Several different cure schedules have been tested in an effort to reduce the production time necessary to make neutron generators. Other cure schedules have been tested in an effort to minimize the mechanical strain placed on the Neutron Generator Tube during the cure cycle.

Methods and Materials

This set of tests made use of thin walled kovar tubes as test specimens (Figure 1.) The lightly sand blasted kovar tubes were instrumented with two thermocouples and four biaxial strain gages laid approximately ninety degrees from one another. Both ends of the tube are sealed and an initial calibration is done to record the thermal strain that the tubes and the gages will encounter. The kovar tubes are then placed in aluminum molds and the encapsulant is mixed and poured into the molds according to the process described in table 3. Data is recorded as the encapsulant cures. Two different tubes are used per test to verify that the data recorded is accurate.

The encapsulant material for the first set of tests was formula 459 epoxy with a glass micro-balloon filler (GMB). In the second set of tests formula 459 epoxy was filled with Aluminum Oxide (ALOX). The mix ratios are presented in Table 1. The variable for this set of experiments was an altered cure schedule. Cure schedule #4 (Table 2) contains a longer ramp time up to 93°C than either cure schedules #1, or #2.

Conclusions

The cure strain experiments performed this summer successfully provided accurate data as shown in Figures 2-7. This data has been turned over to the modeler and, although this project is not yet completed, model accuracy has been improved.

References

1) ***"Formula 459 Encapsulants – Exotherm and Curing strain experiments"***, Sandia National Laboratories internal memo to Distribution from Tommy Guess and Mark Stavig, November 30, 1999

2) Guess, T.R., and Stavig, M.E., ***"Curing Strains in Epoxy Encapsulants: Experimental Data for Model Validation Studies"***, Sandia National Laboratories SAND report, Albuquerque, NM, September 2000, in preparation.

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000.

Appendices

Table 1. Encapsulants

Encapsulant	Mix Ratio (parts by weight)
459/ALOX	100/300
459/GMB	100/35

Table 2. Cure Schedules

Baseline NG Cure	Cure #1	Cure #2
Pour in mold at 71C 2 hour hold at 23C 6 hour ramp to 55C 6 hour hold at 55C 6 hour ramp to 93C 16 hour hold at 93C 4.5 hour ramp to 23C	Pour in mold at 71C 1 hour hold at 23C 10 hour ramp to 93C 10 hour hold at 93C 1 hour ramp to 23C	Pour in mold at 54C 2.5 hours at 54C 2 hour ramp to 93C 2 hour hold at 93C 4 hour ramp to 23C
40.5 hours total	**22 hours total**	**10.5 hours total**

Cure #3	Cure #4
Pour in mold at 54C 2.5 hours at 54C 2 hour ramp to 93C 2 hour hold at 93C 4 hour ramp to 23C	Pour in mold at 54C 2.3 hours at 23C 8 hour ramp to 93C 3 hour hold at 93C 1 hour ramp to 23C
8.8 hours total	**14.3 hours total**

Table 3. Processing and Pouring of Formula 459 Encapsulants

Materials Preheat:

459A	54 C 2 hrs. min.
459B	54 C
ALOX	71 C
GMB	71 C

Hardware Preheat:

Vacuum chamber	60C
Hobart bucket/ plastic	71C
Molds	54 C for 2 hrs. min.

Encapsulant formulation (parts by weight, pbw):

459 (unfilled)

459A Epon 826	75
459B[1]	25
KF865[2]	0.05

459/ALOX

459A Epon 826	75
459B[1]	25
KF865[2]	0.05
ALOX	300

459/GMB

459A Epon 826	75
459B[1]	25
KF865[2]	0.05
GMB D32	35

[1]459B is the hardener and contains of equal parts of Ancamine 2049 and Jeffamine D230.
[2]KF865 is a degassing agent.

Process 459, 459/GMB, or 459/ALOX as follows:

- **Weigh 459 parts A and B, KF865 degassing agent, and filler (if needed) into Hobart bucket, hand mix until wet, then mix on Hobart for 3 minutes.**
- **De-air for at least 3 minutes at 60C at 1-3 torr.**
- **Open oven door and pour encapsulant into heated molds.**
- **Close oven and start the curing with the selected cure schedule.**

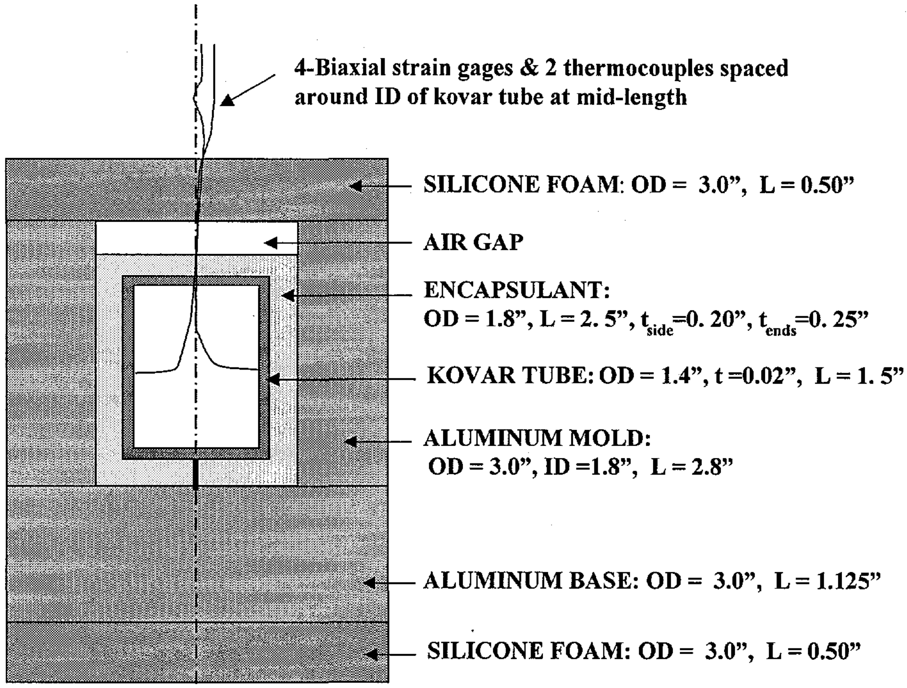

Figure #1: Schematic of kovar tube curing strain specimen for measuring strains and temperatures in kovar tube during encapsulant cure.

Specimen #17	459/ALOX with Cure #3
Aluminum Mold	Sand Blasted Mold

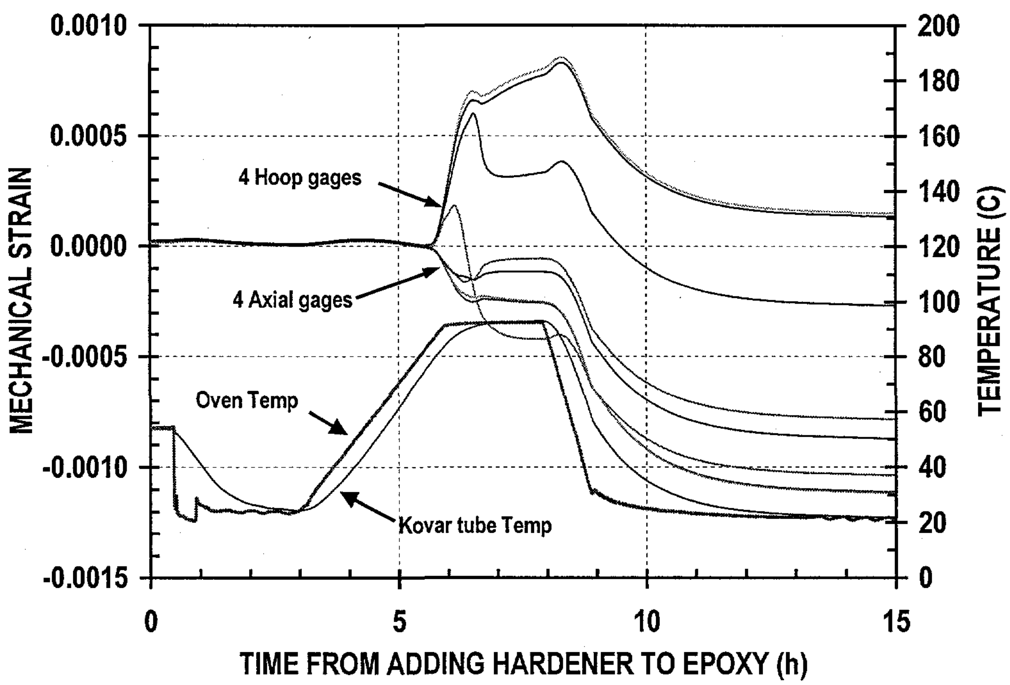

Figure #2: Strain and temperature responses for kovar tube curing strain Specimen #17.

Specimen #28	459/ALOX Cure #4
Aluminum Mold	Sand Blasted Mold

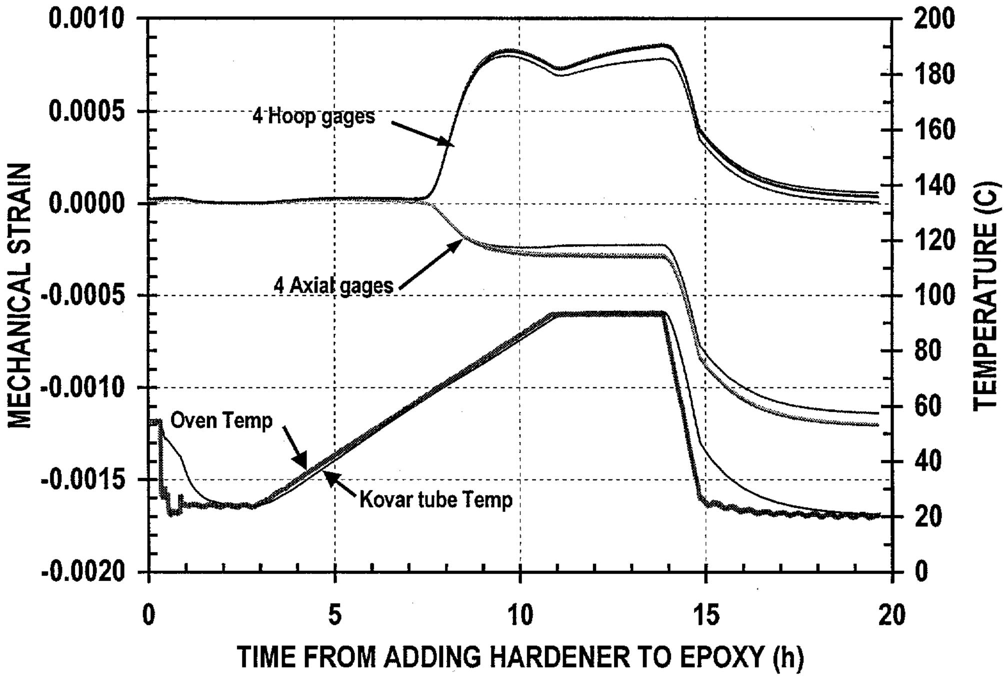

Figure #3: Strain and temperature responses for kovar tube curing strain Specimen #28.

Specimen #29	459/ALOX Cure #4
Aluminum Mold	Sand Blasted Mold

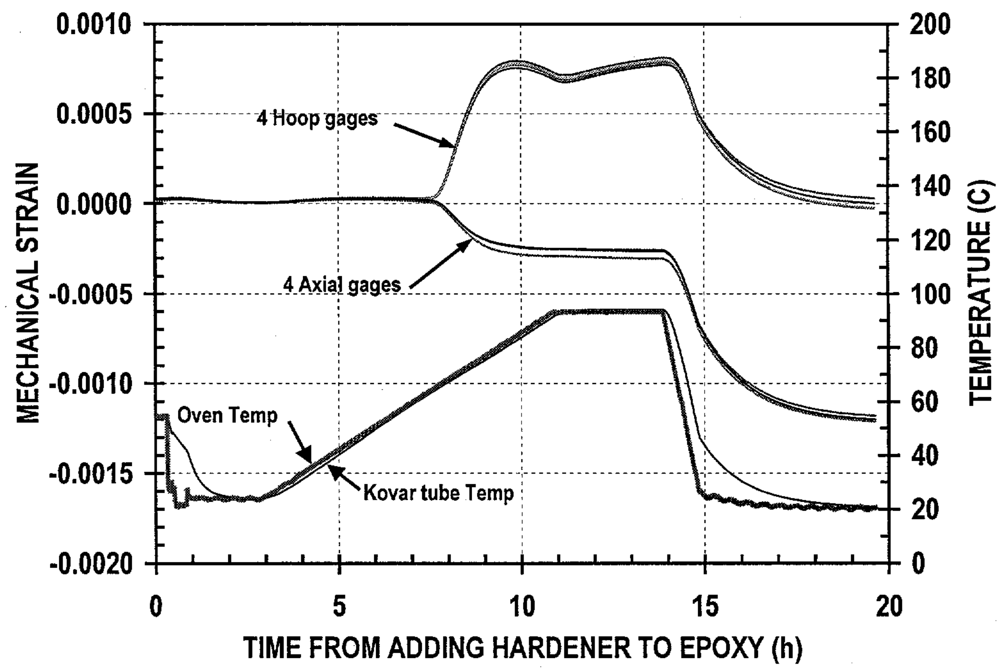

Figure #4: Strain and temperature responses for kovar tube curing strain Specimen #29.

Specimen #21	459/GMB with Cure #3
Aluminum Mold	Sand Blasted Mold

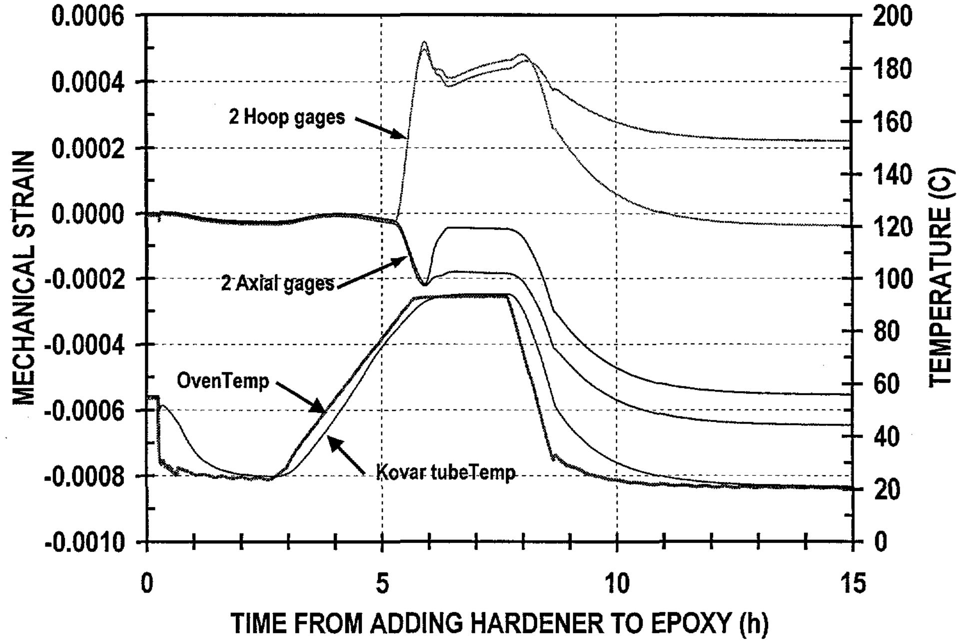

Figure #5: Strain and temperature responses for kovar tube curing strain Specimen #21.

Specimen #26	459/GMB Cure #4
Aluminum Mold	Sand Blasted Mold

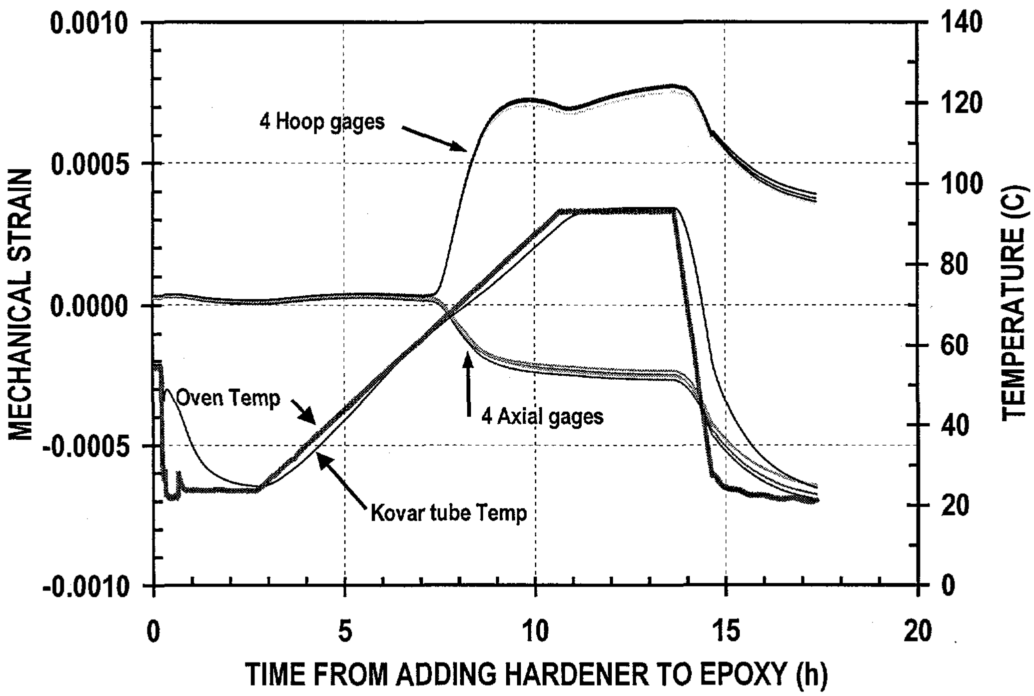

Figure #6: Strain and temperature responses for kovar tube curing strain Specimen #26.

Specimen #27	459/GMB with Cure #4
Aluminum Mold	Sand Blasted Mold

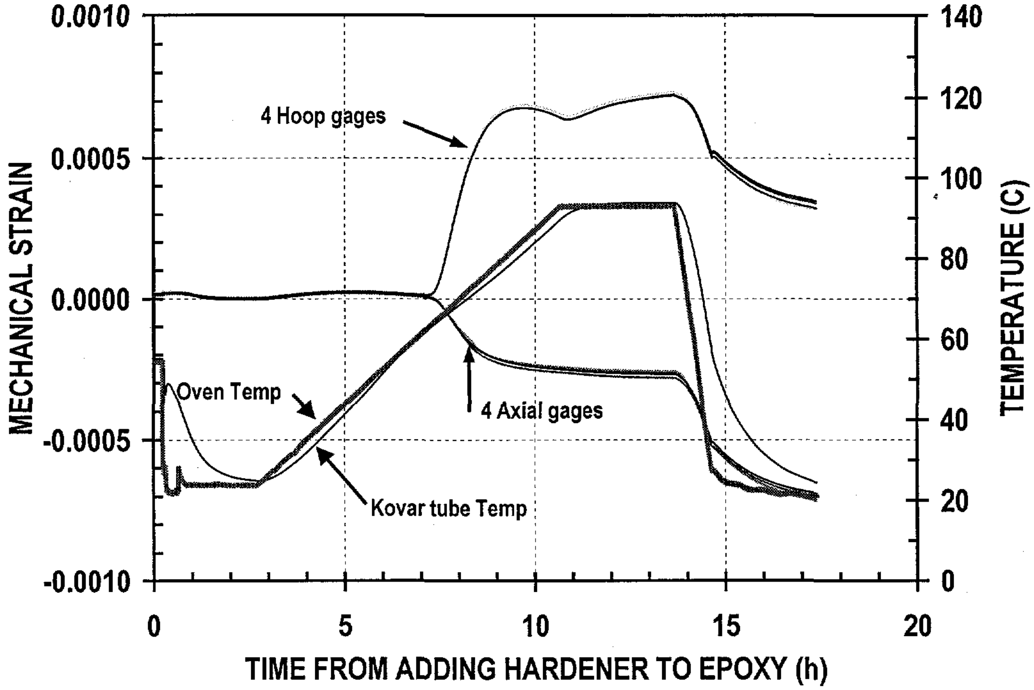

Figure #7: Strain and temperature responses for kovar tube curing strain Specimen #27.