CONF-9509201--1

SAND95-1658C

IMPREGNATING MAGNETIC COMPONENTS WITH MDA FREE EPOXY

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ABSTRACT

This paper describes the use of "Formula 456" an aliphatic amine cured epoxy for impregnating coils. Methylene dianiline (MDA) has been used for more than 20 years as the curing agent for various epoxy formulations throughout the Department of Energy. Sandia National Laboratories began the process of replacing MDA with other formulations because of regulations imposed by OSHA on the use of MDA.

INTRODUCTION

The magnetic components required to support the various Department of Energy (DOE) and Department of Defense programs include transformers, solenoid coils, and inductors. A rugged package is essential because of some military type applications where high voltage, size and severe environments are major considerations. For more than 20 years Sandia National Laboratories (SNL) has used a formula defined in SNL process specification 9927020 for encapsulating/impregnating these type of coils. The formula consists of an epoxy resin (Epon 828), a hardener methylene dianiline (MDA), a filler (normally mica), and some color paste. OSHA has regulated MDA because it is a suspect carcinogen. Formulation development for MDA-free epoxies has targeted not only the replacement of MDA, but also the improvement of other formulation properties and processability. Typically the elimination of OSHA regulated materials provides a rare opportunity to qualify new formulations in a range of demanding applications. It is important to take full advantage of that opportunity and the costly materials qualification effort associated with it.

SNL and the DOE production agencies have evaluated a number of MDA-free epoxy encapsulants which relied on either anhydride or other aromatic amine curing agents. The use of aliphatic amine curing agents was evaluated only more recently and has resulted in the definition of two promising alternative resins. Both rely on the same Bis A epoxies previously used with MDA, and both use the same replacement curing agents; a blend of Jeffamine D-230 (a flexible polyether diamine) and Ancamine 2049 (a cycloaliphatic diamine). Formula 456 is a rubber modified formulation which also includes a Dow Bis A epoxy containing 40% by weight of a phase-separated rubber modifier (Dow XU-71790). Formula 459 contains only a unmodified Bis A epoxy and adds a silicone based de-gassing aid. Both formulations are readily processed at temperature below those required for the MDA cured formulations. They possess lower viscosities and also de-gas very effectively. Electrical and mechanical properties are typically comparable to or better than those of similar MDA cured materials.

THE PROCESS

A power transformer identified as 378012, and a pulse transformer identified as 378128 were selected as the baseline design components used to evaluate this MDA free encapsulant. MIL-SPEC MAGNETICS, INC. undertook the task of evaluating Formula 456 on the two different transformer designs. Two lots of fifty parts each were used during the evaluation. SNL specification SS396751 was followed during encapsulation. Part number 378012 was of special interest because of the intricate winding and insulation interleaving pattern of its design. The challenge was to ensure that the encapsulant totally reaches the spaces between each layer of Kraft paper and between the winding wire spaces.

378012 POWER TRANSFORMER APPLICATION

The transformer is used in a flyback converter to charge a capacitor to 4.2KV (see Figure 1).

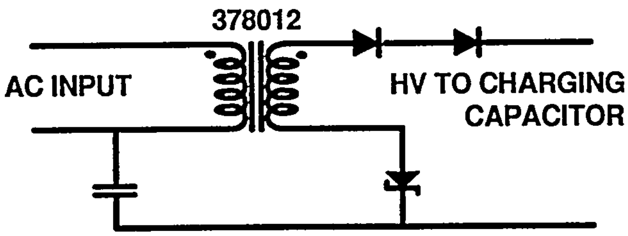

Figure 1

378012 POWER TRANSFORMER DESIGN

The design for the 378012 power transformer is described to provide an understanding of the difficulty of impregnating this type of coil.

DESIGN AND PROCESSES: The Primary winding consist of 26 turns of 29 gage, polyester-insulated copper magnet wire closely wound and centered within the flanges of a nylon bobbin. The secondary winding consists of 1114 turns of 40 gage, heavy polyester-insulated copper magnet wire closely wound and centered within the bobbin flanges. The primary is placed in a single layer and separated from the secondary using Kraft paper insulation (KPI). The secondary is divided into 21 layers, with KPI separating each layer. Several layers of KPI are placed over the last layer. The finish of the secondary winding is internally spliced to a Teflon-insulated, flexible-stranded high voltage lead wire.

CORE / COIL SUBASSEMBLY: The coil is assembled into a matched set of 2616 ferrite pot. The core halves are bonded to each other, using a thixotropic epoxy adhesive, and to a pre-molded diallyl phthalate (DAP) contact assembly using the sealing compound polysulfide. The start and finish of the primary, and the start of the secondary are stripped, cleaned, and soldered to their appropriate pins on the DAP contact assembly. The core halves are painted with a Phenolic micro balloon filled polysulfide (Ref. 1) for the purpose of reducing stresses caused by differences in coefficient of thermal expansion between the ferrite core and the epoxy encapsulating system (Ref. 2). The center cavity of the core is also filled with the polysulfide resin to exclude epoxy from the cavity. The assembly is vacuum baked and plasma cleaned prior to the encapsulation process to promote adhesion of the epoxy to the DAP contact assembly.

ENCAPSULATION

The following covers the procedure for the encapsulation and impregnation of magnetic devices using Formula 456.

COIL DRYING:

a. The coils shall be dried in a vacuum oven at a temperature of 110 ±12°C. They shall be held at this temperature at a maximum pressure of 25mm Hg absolute for a minimum of 4 hours, after which the vacuum shall be broken with dry nitrogen.

b. The units shall then be cooled to and stabilized at 54 ±6°C and maintained at this temperature until ready for encapsulation. They shall be stabilized at this temperature either under dry nitrogen atmosphere, or in a vacuum oven at a maximum pressure of 25mm Hg absolute after which the vacuum shall be broken with dry nitrogen.

DRYING OF FILLER: The mica filler shall be dried by placing the material in a shallow pan, not over two inches in depth, and baked for four hours minimum in a forced convection oven at a temperature of 107 ±6°C.

FORMULATION: Unless otherwise noted, tolerance on weights of material shall be ±1%.

Material	Parts By Weight
Epoxy Resin	75
Mica Filler	65
Curing Agent	25
Color Paste	1.00 +/- .25

MIXING: The preferred batch size is 166 grams combined weight. The maximum recommended batch size is 700 grams. Materials must be homogenous.

a. Combine the epoxy resin, filler and color paste. Mix until material is uniform throughout.

b. Heat or cool the mixture to 54 ±3°C and add the curing agent which shall be at room temperature (18 to 29°C). Mix until material is uniform throughout.

c. Immediately after mixing, evacuate at a pressure of 0.5 to 3.0 mm Hg absolute and a temperature of 74 ±6°C for a minimum of 1 minute and a maximum of 3 minutes after the initial foam rise collapses. If time to collapse is not visible then 2 minutes minimum and 4 minutes maximum.

POURING UNDER VACUUM:

a. Place the preheated molds with components in a vacuum chamber and evacuate at a pressure of 0.5 to 3mm Hg absolute.

b. The deaerated mixture temperature should be at 45°C to 60°C and should immediately be poured into the mold under vacuum. The pressure shall be decreased to 0.5 to 3.0 mm Hg for a minimum of three minutes after pouring the last unit. Break vacuum slowly, 3 minutes minimum to avoid voids.

CURE: The compound shall be cured in a pressure chamber at 80 PSI minimum for 4 hours +/-1 hour @ 25 +/-5°C, plus 6 hours +/-1 hour @ 60 +/-3°C, plus 12 hours +/-1 hour @ 93 +/-3°C. At the end of cure let parts cool in chamber for 30 minutes minimum.

MACHINING TO DIMENSION AND MARKING:
Each transformer is machined to the proper height following removal from the RTV mold. After machining, the part is identified and serialized with epoxy marking.

THERMAL CYCLE AND TEST: Each transformer is subjected to 5 four-hour thermal cycles prior to inspection and test which helps in relieving internal stresses. The temperature extremes for each cycle are -60°C to +93°C, and each cycle requires one hour exposure at each of these temperatures. Each transformer is visually inspected then electrically tested. The electrical tests consist of: DC resistance, inductance, capacitance, turns ratio, polarity, insulation resistance, and induced voltage (corona test).

ENVIRONMENTAL TEST: As a minimum, this transformer design is subjected to the following environments: 3500G mechanical shock; sinusoidal vibration - frequency range (Hz) 10-2000-10; random vibration - complex power spectral density from 5 Hz to 2000 Hz at .001 G^2/Hz to .4G^2 /Hz; steady state acceleration 100G for 10 seconds; temperature shock - 3 cycles -55°C to +100°C; and temperature cycle 110 cycles -60°C to +93°C. The design is tested to these environments during qualification.

The mica-filled epoxy resin system must provide a rugged system that is capable of surviving high levels of shock and vibration, while having stable characteristics over a 25-year period.

POLYSULFIDE COATING: The process of painting the ferrite core with a phenolic micro-balloon filled polysulfide resin was implemented to reduce the stresses that are caused by the difference in coefficient of thermal expansion between the ferrite core and the epoxy resin (Ref 1). The polysulfide system was selected for this application because it adheres well to the ferrite core material, and the epoxy resin bonds well to the cured polysulfide. The Phenolic micro-balloons, 3.3% fill by weight, provide a crushable medium to relieve compressive stresses.

RESULTS

The process specified in SS396751 (and specified above) was followed closely. No coloring paste was used. Initially the batch size of the combined formula was 165 grams. Subsequent batches were reduced to 82.5 grams when it was observed that the formula tended to thicken during the vacuum encapsulation process. In order to speed up the elapsed time between encapsulations, a motorized carousel was used inside the vacuum chamber to index each part directly under the nozzle feeding the encapsulant. The following charts list the material characteristics for Formula 456. Chart 1 lists unfilled material, glass micro balloon filler, alumina filler, and mica filler. Chart 2 lists Formula 456, Formula 459, and MDA material characteristic comparisons.

FORMULA 456 DATA BASE

PROPERTY		unfilled	GMB, phr 30	GMB, phr 35	alumina, phr 250	alumina, phr 300	mica, phr 60	mica, phr 70
Tg, °C (DMA at SNL/CA)		95						
(DSC at Pinellas)		110	103	104	103	100	111	106
Thermal cycle cracks (after 10 cycles of nut & bolt specimen)		0	0	0	0	0		0
Fracture toughness		1.74	0.97	0.90	2.66	2.78	1.86	1.90
CTE (ppm from -50 to 22/22 to 74°C)		69/80	39/44	37/42	29/37	25/33		
Density		1.12	0.72	0.70	2.27			
Dielectric strength, V/mil		631	419	443	549	567	593	604
Volume Resistivity, ohm-cm		1.67	7.67	3.68	1.48	3.88	2.16	0.97
Dielectric Constant	at 100Hz	3.96	2.79	2.72	5.63	5.83	6.39	5.99
	at 1 KHz	3.67	2.70	2.60	5.55	5.77	5.46	5.49
	at 1 MHz	3.58	2.60	2.48	5.32	5.55	4.06	4.15
Dissipation Factor	at 100 Hz	.009	.050	.057	.021	.020	.051	.052
	at 1 KHz	.009	.036	.014	.022	.019	.059	.062
	at 1 MHZ	.032	.021	.018	.019	.018	.041	.043
Pulse Dielectric Strength, KV/mil/Bkdn	at -54°C	11.69	2.69	2.38	4.58	4.27		4.89
	at 25°C	8.92	2.36	na	4.48	na	3.46	3.96
	at 71°C	9.53	2.25	1.83	4.01	3.86	3.98	4.06
Tensile strength, psi	maximum	8900		5128		10,100		7235
	at break	8550				10,100		
Tensile modulus, Ksi		360		424		2200		751
Elongation	at max load	9.6%		1.3%		1.1%		1.5%
	at break	--				--		
Butt tensile adhesive strength:								
ceramic substrate - initial				1513				
after 100 TCs				995				
Al 2024 substrate - initial						4174?		1186
after 100 TCs						5151?		818
Al 7075 substrate - initial								
after 100 TCs								
Lap shear adhesive strength, Al/Al (Al 2024)		2980						

FORMULATION 456, 459 AND MDA COMPARISON

	MDA Formulation toughened	MDA Formulation plain	Formula 456	Formula 459
Composition:	20 Shell Z 100 Epon 828-CTBN (X8 at 10 wt. %)	20 Shell Z 100 Epon 828	12.5 Jeffamine D-230 12.5 Ancamine 2049 50.0 Shell Epon 826 25.0 Dow XU-71790	12.5 Jeffamine D-230 12.5 Ancamine 2049 75.0 Shell Epon 826
Rubber modifier content	8.3 wt percent	none	10 wt percent	none
Appearance, unfilled	yellow, cloudy	yellow, clear	white, opaque	yellow, clear
Tg (DMA, storage mod.)	98°C		95°C	95-100°C
(DSC)			110°C	
NH/epoxy stoichiometry	1.11	1.00	1.16	1.01
CTE (-50°-22°/22°-50°C)				
unfilled			69/80 ppm	51/56 ppm
with 250-350 phr alumina		22/28 (250 phr)	25/33 ppm (300 phr)	19/25 (360 phr)
Thermal cycle (10) cracks				
unfilled	0	5	0	6
with 300 phr alumina	0		0	0
Tensile properties - unfilled:				
strength, maximum			8900 psi	11,490
at break			8550 psi	10,380
modulus			360 Ksi	487 Ksi
elongation, at max load			9.6%	5.0%
at break			--	7.3%
Tensile properties - with 250-350 phr alumina:		(250 phr)	(300 phr Alox)	(300 phr Alox)
strength, maximum		10,700	10,100 psi	10,840 psi
at break		--	10,100 psi	10,840 psi
modulus		2230 Ksi	2200 Ksi	2870 Ksi
elongation, at max load		0.5%	1.1%	0.6%
at break		--	--	0.6%
60°C viscosity data				
initial viscosity	555 cps		115 cps	67 cps
time to double	35 min.		17 min.	19 min.
time to 600 cps	6 min.		36 min.	42 min.

CONCLUSION

We consider and rate the Formula 456 encapsulant as an excellent method of encapsulating intricate parts similar to the P/N 378012. The fifty 378012 and the fifty 378128 parts were visually inspected and electrically tested. No voids were observed in any of the parts and no differences were noted in any of the electrical characteristics. The 378012 parts were machined to size on a lathe. The 378128 parts were machined to size on a milling machine. We did not have any delamination of the encapsulant from the contact assemblies.

SUGGESTIONS BY MIL-SPEC MAGNETICS

A two-part formula with the Mica incorporated into the epoxy resin would be desirable. We would prefer a longer pot-life of the mixed formula. This would result in more economical production.

REFERENCES

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ACKNOWLEDGMENTS

This work was supported by the United States Department of Energy under contract DE-AC04-94AL85000.

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