FILLERS AND POTTING COMPOUNDS

H. W. Lichte

DEVELOPMENT DIVISION

OCTOBER - DECEMBER 1971

*Normal Process Development
Endeavor No. 201*

Mason & Hanger-Silas Mason Co., Inc.
Pantex Plant

P. O. BOX 647
AMARILLO, TEXAS 79105
806-335-1581

operated for the
ATOMIC ENERGY COMMISSION
under

U. S. GOVERNMENT Contract DA-11-173-AMC-487 (A)

FILLERS AND POTTING COMPOUNDS

H. W. Lichte

DEVELOPMENT DIVISION

DISCLAIMER

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, 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 or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

October - December 1971
Endeavor No. 201
Acct. No. 22-2-44-01-201

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

MASTER

DISCLAIMER

**Portions of this document may be illegible
in electronic image products. Images are
produced from the best available original
document.**

FILLERS AND POTTING COMPOUNDS

ABSTRACT

Epoxy/metal oxide formulations were studied to establish techniques to encapsulate various types of velocity gages and simulators for Manganin gages, all of which are to be used in shock hydrodynamics studies by others; and to produce large mock HE castings. Future efforts to characterize the raw materials are recommended.

INTRODUCTION

There is a requirement to assemble and encapsulate experimental designs of velocity gages and Manganin gage simulators. Simple molds, assembly techniques, and processes were developed to fabricate gages. The experimental gages consist of an aluminum oxide/epoxy form, about which is wrapped the sensing element wires—all of which are then encapsulated with aluminum oxide/epoxy resulting in a solid cylinder referred to either as an electromagnetic stress gage or a velocity gage(1). Manganin gage simulators were made in several configurations—solid right circular cylinders of aluminum oxide/epoxy, co-axial pin switches encapsulated in aluminum oxide/epoxy cylinders, and a ceramic "Toad" encapsulated in aluminum oxide/epoxy cylinders.

A design agency requirement(2) was met by modifying a known iron oxide/epoxy formulation toward future needs of casting mock HE parts.

DISCUSSION

ELECTROMAGNETIC STRESS AND VELOCITY GAGES

Experimental design gages were fabricated for testing. The gage functions are discussed in previous reports(1,3). Five different forms were made during this report period (Fig. 1). Form No. 1 is 1/8 x 1/8 x 2 inches. One wire is to be stretched over the 90° included angle wedge. Other variations pictured are Form No. 2 with one wire over each of the six flat ledges; Form No. 3 with a wire over each of the three 60° wedges and passing into the respective troughs; Form No. 4 with a wire over each of the three flat ledges; and Form No. 5 with one wire over the 30° included plane. Each wired form is encapsulated into a 1 3/4 x 2-inch long solid right circular cylinder.

The developed sequence of casting, subassembly and encapsulation begins with a pattern. A stainless steel pattern was machined for each gage form. The pattern was encapsulated in RTV 634^a and after removal from the cured silicone resulted in a female mold of the gage form. An aluminum oxide filler/epoxy

^aTrademark G. E. Silicone rubber potting compound

(1) see references

resin system (See Table I) was used to cast the gage form in the silicone mold. The 0.004-inch copper wire (0.0015-inch copper foil was substituted in several gages) was stretched over the gage forms and secured on the aft end. An aluminum pattern (1 3/4 x 3 inches long solid right circular cylinder) was machined and then encapsulated in RTV 634 and upon removal resulted in a female cylindrical mold. The subassembly of wire and gage form was positioned so that its forward end was a predetermined distance away from the cylindrical silicone mold bottom. The wired gage form was then encapsulated with aluminum oxide/epoxy resulting in a wired gage form embedded in a 1 3/4 x 2-inch solid cylinder. Density of the castings was verified to be 2.3 g/cc on all 30 gages fabricated.

Radiographs were evaluated to insure the position of the wires in the final assembly (Fig. 2).

MANGANIN GAGE SIMULATORS

Four types were cast with the aluminum oxide/epoxy formulations (Table I). The first was a 1-3/4 wide x 2-inch solid right circular cylinder. The second type was overcast upon an aluminum wafer 0.090-inch thick with a "Toad" (ceramic device) embedded at the interface. Fig. 3 is a photograph of the radiograph illustrating the 1 x 1 mm Toad. The third type was similar to No. 2 but the Toad had a single conductor lead (copper wire) plated to the rear (Fig. 4). The fourth type was embedded with two coaxial pin switches (Fig. 5). The twenty gages of these types(4) were ~ 2.3 g/cc density and were x-rayed for integrity.

LARGE MOCK HE CASTINGS

Large filled epoxy castings can be formulated to a wide density range centered about standard HE densities. Initially the mock HE density requirement was 2.07 g/cc. Experience with anhydride cured systems^b indicated that changing from 1.8 to 2.07 g/cc would not change the processing techniques.

Calculations were made to predict the new iron oxide ratio to achieve the 2.07 g/cc keeping all the other ingredients in the original ratio (Table II) but substituting DMP-30(5) for BDMA (benzyl dimethylamine) and eliminating the glass microballoons. Small batches (1 kg) were cast for density and machinability tests to verify and adjust the formulation.

The calculated value (Table III) was generally 10% higher than required to meet the density requirements.

^b SLL P. O.'s 92-4353, 92-4884

Actual formulation for 2.07 g/cc castings

75 pbw Epon 828
25 pbw Epon 871
117 pbw DDSA
0.5 pbw DMP-30
333 pbw Fe₂O₃ (Note 333 vs 369.62g calculated from Table III)

A difference was expected due to using average specific gravities (from trade literature) of the materials for calculations and neglecting chemical reactions. The wet/dry weight method of measuring the casting density was utilized.

The small casting batches were mixed with standard lab mixers utilizing steam heated pots and vacuum bell jars. The large batches (~ 20 lbs each) will be mixed in the Groen Kettle(6) as with the previous work^b. The castings are cured in a large electric oven (27 cu ft). Each day a batch is mixed and poured, divided between four molds. After seven pouring days the net result is four large castings at 35 pounds each. The layers within a finished machined casting are not visually detectable, thus indicating a relatively homogeneous part.

A more recent requirement has been suggested by the Design Agency. This will be large castings at 2.7 g/cc density. Calculations indicate an ~ 70% filler loading using iron oxide (Fig. 6). If initial tests do not give promising results, it will be necessary to use higher density fillers such as lead or tungsten.

RAW MATERIAL CHARACTERIZATION

Reports(7,8) indicate changes in shock properties of the aluminum oxide/epoxy gages between lots. The processes used (Table I) could conceivably be the cause of the changes. For example, the aluminum oxide may vary in specific gravity from 3.65 to 3.80. The particle size requirements are that 96% minimum pass through a 325-mesh screen. The curing agent "Z" may vary from 20 to 40 poises (25 C). The resin may change epoxide equivalent 183 to 195 and viscosity from 100 to 160 poises—additionally, choices of six different suppliers could be made. It has been reported(9) the significant changes in oligomer concentration within and between lots of different manufacturers of the resin. The resin is basically diglycidyl ether of bisphenol A (DGEBA) plus oligomers. Manufacturer A had a lot at 73.9% DGEBA, manufacturer B's lots varied 68.4% to 76.6%, and manufacturer C's lots varied 74.7% to 79.7%. Variations in weighing and mixing the three ingredients would also have an effect on the strength properties. Annealing the castings would relieve stress concentrations at corners and near the wire interfaces. Future lots of these raw materials will be characterized or sorted and formulation processes improved. Further compatibility testing will be needed for specific mock applications (Table IV).

CONCLUSIONS

The investment casting technique using silicone molds provides an acceptable process for casting complicated shapes of aluminum-oxide/epoxy formulations. Specific data on the material lots must be known when the casting is to be used in shock hydrodynamic testing.

Large iron oxide/epoxy castings can be made in density ranges from 1.8 to 2.07 g/cc. Samples have been made up to 2.6 g/cc. Any higher densities should require other metal powders (tungsten, lead, barium sulfate).

Table I. Formulation (Process Spec 9927061)

100 pbw	Epoxy Resin - "Epi Rex 510" (Celanese Coating Co.) - Commercial grade of diglycidyl ether of bisphenol A without modifiers or diluents.
20 pbw	Curing Agent - "Z" (Shell Chemical Co.) - aromatic amine, eutectic mixture of methylenedianiline, m-phenylenediamine and phenyl glycidyl ether.
300 pbw	Filler - Aluminum oxide (ALCOA) - tabular alumina T-61 (-325 mesh) standard grade.

Table II. Formulation (Process Spec 55230706)

75 pbw	Epoxy Resin - "Epon 828" (Shell Chemical Co.) commercial grade of diglycidyl ether of bisphenol A without modifiers or diluents
25 pbw	Epoxy Resin - "Epon 871" (Shell Chemical Co.) aliphatic polyepoxide resin
117 pbw	Curing Agent - "DD-SA" (Allied Chem. Corp) dodecenylsuccinic anhydride
0.5 pbw	Accelerator - "BDMA" (Interchemical Corp) benzyldimethylamine
22.8 pbw	Fillers, hollow glass spheres - "Microballoons" (Emerson & Cuming Inc.)
225 pbw	Fillers, iron oxide, Fe_2O_3 (98% pure) (Allied Chem. Corp)

Table III. Sample Calculations

$$\rho_T = \frac{W_T}{V_T}$$

$$W_T = w_1 + w_2 + w_3 + w_4 + w_5$$

w = weight,
v = volume, cc
ρ = density, g/cc

Subscript 1 = 828
2 = 871
3 = DDSA
4 = DMP-30
5 = Fe₂O₃
T = Total Mixture

$$V_T = v_1 + v_2 + v_3 + v_4 + v_5$$

$$= \frac{w_1}{\rho_1} + \frac{w_2}{\rho_2} + \frac{w_3}{\rho_3} + \frac{w_4}{\rho_4} + \frac{w_5}{\rho_5}$$

substituting:

1 = 75 g @ 1.163 g/cc
2 = 25 g @ .983 g/cc
3 = 117 g @ 1.001 g/cc
4 = 0.5 g @ .973 g/cc
5 = w₅ @ 4.843 g/cc

$$V_T = 207.317 + \frac{w_5}{4.843} = \frac{W_T}{\rho_T} \quad \text{assume } \rho_T = 2.07$$

$$W_T = 217.5 + w_5$$

$$w_5 = 369.62 \text{ g}$$

Table IV. Reactivity Tests *
(μliters gas & STP)

	<u>N₂</u>	<u>CO</u>	<u>NO</u>	<u>CO₂</u>	<u>N₂O</u>	<u>H₂O</u>
1. Sample No. 1 (Appendix B formulation)	4.06	—	.09	10.70	T	.225
2. EXT X	6.96	3.47	7.7	7.48	1.89	.17
3. 1 & 2 Above Combined	40.63	15.98	21.54	74.34	24.36	1.25
4. Sample No. 2 (Appendix A formulation)	12.81	—	T	3.98	—	2.24
5. 2 & 4 Above Combined	40.16	25.78	453.78	73.94	46.41	3.06
6. Sample No. 3 (Appendix C formulation)	5.27	—	—	6.07	—	.063
7. 2 & 6 Above Combined	49.79	29.02	73.56	81.92	32.17	.978

The following samples are entered for comparison

8. Adiprene L100/moca	9.74	—	—	70.93	—	.578
	6.63	—	—	31.68	—	.265
9. 2 & 8 Above Combined	1317.22	57.91	40.76	797.63	50.20	5.87
10. Adiprene L100/DMP30	8.47	—	—	235.15	—	.028
11. 2 & 10 Above Combined	1828.91	217.34	252.68	2040.06	492.36	1.26
12. Adiprene L167/moca	6.21	—	—	23.08	—	—
13. 2 & 12 Above Combined	1506.43	46.59	24.98	1025.91	34.97	5.86

* NOTE: Myers, L.C., "Chemical Reactivity Tests" @ 120 C 22 hours HE Atmosphere

← FWD End AFT →

Fig. 1. Gage Forms (Wire Not Shown)

Fig. 2. Radiograph Form No. 4

Fig. 3. Radiograph Embedded "Toad"

Fig. 4. Radiograph "Toad" With Wire

Fig. 5. Radiograph Co-Axial Pin Switches

Fig. 6. Iron Oxide Formulation Calculations

REFERENCES

1. R. S. Wilson, "Electromagnetic Stress and Velocity Gages," MHSMP-71-89.
2. Ltr., W. H. Bachelder/Ltr to D. L. Coder/PX, October 5, 1971.
3. Young, C., et al, "An Electromagnetic Stress Gage," Physics International Co.
4. Boettner, J. K., "Detonation Electric Effect" Qtr. Report, January-March 1972.
5. Rohm & Haas Co., 2,4,6-Tri(dimethylaminomethyl) phenol
6. Groen Mfg. Co. - 10 gallon, high vacuum, stainless steel, steam jacketed, power tilting kettle
7. C. E. Canada, "Hugoniot of an Aluminum Oxide Filled Epoxy @ -45 F, Room Temperature, and 165 F," April-June 1971
8. C. E. Canada, "Hugoniot Determination at -65 F," October - December 1971
9. F. N. Larson, "Gel Permeation Chromographic Analysis of Commercial Epoxy Resins," 6th International Seminar GPC, October 7, 1968, Miami, Florida