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AN INVESTIGATION OF SEALING
TECHNIQUES FOR ELECTROEXPLOSIVE
DEVICES

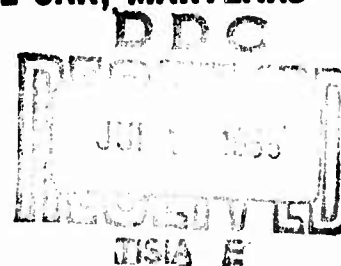
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NOLTR 65-41

AN INVESTIGATION OF SEALING TECHNIQUES
FOR ELECTROEXPLOSIVE DEVICES

By

Louis J. Montesi

ABSTRACT: An investigation of sealing techniques for electroexplosive devices has been conducted. Epoxy resins, solder amalgams, and high temperature solders were investigated. An epoxy resin gave a non-hermetic, but a water tight seal. Solder amalgams resulted in poor seals. Solders with melting points up to 600° F were successfully used to hermetically seal explosive components.

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EXPLOSION DYNAMICS DIVISION
EXPLOSIONS RESEARCH DEPARTMENT
U. S. NAVAL ORDNANCE LABORATORY
WHITE OAK, MARYLAND

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25 May 1965

AN INVESTIGATION OF SEALING TECHNIQUES FOR ELECTROEXPLOSIVE
DEVICES

This report describes the results of an investigation into moisture sealing techniques for electroexplosive devices. The investigation was performed under Task RUME-4E000/212-1/F008-08-11, Problem Assignment No. 016.

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By direction

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INTRODUCTION

1. Sealing of explosive devices against the ingress of moisture can be accomplished by various means. For example, by interference fits of hardware, by coating with varnishes or lacquers, or by potting with various plastics. More positive or hermetic seals can be obtained by soldering and welding. The latter methods usually involve the application of heat.

2. As our technology advances and the application of explosive components become more diversified, so do the sealing requirements for them. One such requirement is a seal which will remain hermetic over large temperature excursions. Another is an inexpensive seal which can be used for mass production of low cost "moisture proof" explosive components.

3. Methods to improve and extend the useful range of our present sealing techniques were therefore investigated. Developments in the field of plastics suggested that the utility of various epoxy compounds be investigated. The use of liquid or cold solder amalgams was also tried, and the feasibility of sealing with high temperature solders was investigated. Sealing with silver solders and welding are feasible, and items have been sealed by these methods. However, this investigation does not include a study of these techniques.

SEALING WITH EPOXY RESINS

4. Two epoxy compounds, Scotchcast Electrical Resin No.8* (Parts A and B) and Stycast Resin No. 2741** (with catalyst No. 15) were investigated. A preliminary test program was conducted using a 1 to 1 weight ratio of resin to the activator for these two compounds. Ten inert detonators, composed of a cup, a spacer, and a phenolic plug were assembled and the resin applied to the crimped area. They were then subjected to the following Navy environmental tests: (a) Vacuum-Steam-Pressure Test, Mil Std-305 and (b) Temperature and Humidity Cycling Test, Mil Std-304. Scotchcast Electrical Resin No. 8 demonstrated good sealing properties. Stycast Resin No. 2741 did not. Stycast Resin when cured, was porous and neither a hermetic seal nor a good water tight seal was obtained. Based on these results a more extensive test program using Scotchcast Electrical Resin No. 8 was devised.

* A product of Minnesota Mining and Manufacturing Company

** A product of Emerson and Cuming, Inc., Canton, Massachusetts

5. The program consisted first of testing the seal integrity of inert detonator units sealed with Scotchcast Electrical Resin No. 8, and then the sensitivity, output, and seal integrity of loaded detonators sealed with the same material.

6. The inert detonator units contained silica gel, and were subjected to the following tests:

- | | |
|-------------------------|--|
| a. Mil Std-300 | Jolt |
| b. Mil Std-301 | Jumble |
| c. Mil Std-304 | Temperature and Humidity
Cycling |
| d. Mil Std-353 | Transportation Vibration |
| e. TN-1441-VIA | Aircraft Vibration |
| f. Water Immersion Test | Units were placed in water
and pressurized to 250 and
440 psi for 24 hours |

The seal integrity was checked before and after each test by the following methods:

- a. In a vacuum of 25 inches of mercury with "leak tec"
- b. In a vacuum of 25 inches of mercury under water

Samples of the units were opened after each test, and the silica gel examined for presence of moisture. The leak frequency of the inert-silica gel loaded units after the various environmental tests is shown in Table 1. After Mil Std-304, these units showed a leak frequency of 6 in 25. No leakers were observed after the other environmental tests, except 1 leaker in 18 after water immersion at 250 psi.

7. Additional seal integrity data, as well as some sensitivity and output data, was obtained when a third group of detonators, loaded as shown in Figure 1, were sealed with Scotchcast resin, and subjected to the standard Navy environmental tests. The seal integrity of this group was satisfactory after the environmental tests** including Mil Std-304 for the prescribed 4 week period, and for an 8 week period. These test results are

* A product of American Gas and Chemical, Inc., N.Y., N.Y.

** The units after Jumble (Mil Std-301) Test need only be safe to handle and dispose.

given in Table 2. It appears that the method of application and the care taken to avoid air bubbles from forming in the seal will determine how good a seal is obtained. In addition to the seal integrity data, sensitivity and output data were also obtained on these units and are shown in Table 3. The sensitivity of these units after the various tests, as measured by the 50% initiation point from a Bruceton type test^{1*}, range from 15,500 to 19,000 ergs. The difference observed in the 0.50 probability of firing level is not believed to be significant. It can probably be attributed in part or wholly to the small sample size Bruceton tests conducted^{2,3}. The output measured by dent impressions in steel blocks (see Figure 2) show no significant changes after environmental testing. The average output dent values were 13.8 to 16.3 mils.

SEALING WITH COLD SOLDER AMALGAMS

8. Several references to successful bonding with various gallium based amalgams^{4,5,6} prompted an investigation of this method as a possible sealing technique. Several attempts were made to seal copper, brass, gilding metal, aluminum, and steel to kovar with various gallium based amalgams.

9. Various chemical formulations of gallium amalgams were tried without much success. The formulations tried are given below:

Composition	Percent by Weight		
	Mix No. 1	Mix No. 2	Mix No. 3
Gallium	32%	41%	40%
Copper	44%	-	35%
Tin	-	59%	25%
Gold	24%	-	-

10. A summary of the procedure used to seal the various units is given below:

a. Two gram batches of amalgam were prepared by weighing out the proper proportions of the constituents. The materials were then prepared as follows:

Mix No. 1 and Mix No. 2

The ingredients involved were placed into a glass beaker

* References are given on Page 7.

and heated on a hot plate. The metal ingredients were worked into the liquid gallium with a teflon stirring rod to start the reaction. Occasional stirring continued until the reaction was completed. The resulting amalgam was a granular substance, silvery in nature.

Mix No. 3

The gallium and tin materials were placed into a glass beaker and heated on a hot plate. The tin flakes were worked into the gallium liquid with a teflon stirring rod. Once the reaction was started, the copper powder was added. The copper powder was worked into the other substance. The resulting amalgam was again a granular silvery substance.

b. The amalgams were hand packed around the kovar plug-cup interface and pressed firmly into place. This work was very time consuming and tedious.

11. The seals obtained using the above procedure were poor. In most cases, the sealed units failed to pass the leak test.* In addition, the amalgam reacted chemically with the aluminum cups and caused what appeared to be stress cracks in the cups. The bond between the kovar plug-amalgam-cup interfaces were extremely poor and the seals could be broken by hand pressure. Finally, the cost of the amalgams would be extremely high. Gallium costs approximately \$4.00 a gram, and a two-gram batch of material would seal only approximately 10 units. As a result, further work with the gallium amalgams was not attempted.

SEALING WITH HIGH TEMPERATURE SOLDERS

12. The temperature limitation of any particular explosive component is determined by the maximum temperature the component can experience and still function reliably either at the time of exposure, or at a later date. The newly developed high temperature resistant explosives can withstand temperatures of 500°F and higher without decomposing. Presently, many of our explosive components contain explosives which decompose at approximately 300°F and are sealed with solders whose melting points are in the same temperature range. Therefore, further hermetically sealed components containing the new high temperature resistance explosives will need a seal that can withstand temperatures greater than 500°F. One way of doing this is to hermetically seal with high temperature solders. Various alloys consisting of silver and lead, and antimony and tin are useful for high temperature requirements when the solder joint is exposed to temperatures above 450°F.

* The seal integrity of these units were checked in a vacuum of 25 inches of mercury with "leak tec".

It was unknown whether explosives subjected to these soldering temperatures would be affected during the soldering operation. As a result, the following two solders were selected for test purposes:

- a. A 95% tin, 5% antimony mixture having a melting point of approximately 450°F, and
- b. A 97% lead, 3% silver mixture with a melting point of approximately 600°F. Solder rings, 0.260 O.D. and 0.030 thick were used in the sealing process.

The tin-antimony alloy was selected because its melting point is approximately that of the decomposition point of the explosives being soldered. The lead-silver solder was selected because of its high melting point, 600°F.

14. The soldering was done with a Lepal, water cooled induction heater, Model No. T-2.5-G. A UNI-coil transformer disc as shown in Figure 3, was used in the process. A brass cylinder, 3.0 inches in diameter was used as a heat sink for the detonator being soldered.

15. Detonators, with temperature limitations of approximately 400° and 500°F were made. See Figure 4. They contained explosives having decomposition temperatures close to the melting point of the solders used. The decomposition temperatures of the explosives used in the test detonator are given below:

Explosive	Explosion Temperature	Remarks
Normal Lead Styphnate	500°F ⁷	Burns or detonates
Dextrinated Lead Azide	464-482°F ⁸	Decomposes
RDX	399°F ⁸	Melts

These explosives are not normally used in high temperature resistant detonators. However, it was felt that if detonators containing these explosives showed no detrimental effect from the soldering process, then detonators with the high temperature resistant explosives would be less susceptible to thermal deterioration.

16. The 400°F temperature detonator contained normal lead styphnate, dextrinated lead azide, and RDX, and was sealed with 450°F melting point solder. The 500°F temperature detonator contained normal lead styphnate, and dextrinated lead azide, and was sealed with 600°F melting point solder. After soldering,

the seal integrity of the detonators were checked in a vacuum of 25 inches of mercury with "leak tec". All detonators passed this test.

17. The detonators were then tested for firing sensitivity and output, the results are shown in Tables 4 and 5. The sensitivity after soldering had not changed significantly from the control sample. Values (50% firing) ranged from 19,500 to 20,500 ergs. The control sample value was 20,000 ergs.

18. Also, no significant changes from the control sample were noted in the output of the soldered units. The output of the 400°F detonator gave average dent impressions on a steel block of approximately 18 to 19 mils. The output of the 500°F detonator gave average dent values on an aluminum block of 30 to 31 mils. The aluminum block was used because these detonators did not contain the usual secondary explosive base charge and consequently were incapable of producing easily measured dents in the steel block.

CONCLUSIONS

On the basis of the work performed, the following conclusions are drawn:

1. Hermetic seals were obtained with high temperature solders having melting points up to 600°F.
2. Good water tight seals can be obtained with Scotchcast Electrical Resin No. 8. This material could be used in place of varnishes and lacquers.
3. Gallium based amalgams gave poor mechanical and chemical bonds.

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April 1955

Table 1
Leak Frequency Data of Silica Gel Detonator Units
Sealed with Scotchcast Resin No. 8

Environmental Test	Sample Size	Number of Leakers
Mil Std-353 (Transportation - vibration)	10	None
TN-1441-VIA (Aircraft - vibration)	10	None
Mil Std-300 (Jolt)	10	None
Mil Std-301 (Jumble)	10	None
Mil Std-304 (T&H) (4 wks)	25	6/25 Leaked
Water immersion test - 250 psi - 24 hours	18	1/18 Leaked
Water immersion test - 440 psi - 24 hours	17	None

Table 2
Leak Frequency Data of Explosively Loaded Detonators
Sealed with Scotchcast Resin No. 8

Environmental Test	Sample Size	Number of Leakers
Mil Std-353 (Transportation - vibration)	15*	None
TN-1441-VIA (Aircraft - vibration)	15*	None
Mil Std-300 (Jolt)	15*	None
Mil Std-301 (Jumble)	15*	3/15 Leaked
Mil Std-304 (T&H) (4 wks)	25	None
Mil Std-304 (T&H) (8 wks)	20	None
Water Immersion test - 440 psi - 24 hours	21	None

* These fifteen units were sequentially tested in above order

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Table 3

Sensitivity and Output Data of Detonators
Sealed with Scotchcast Resin No. 8

Sensitivity Data (Capacitor Discharge Firing, Potential 50 Volts)

Environmental Test	Sample Size	Capacitance for 50% Firing	Standard Deviation (log unit)	50% Firing Level (ergs)
Control	25	1.30	0.020	16,000
Sequential Tests # 1-4	15	1.34	0.025	16,500
Test # 5 (4 weeks)	25	1.52	0.098	19,000
Test # 5 (8 weeks)	20	1.40	0.023	17,500
Test # 6	21	1.25	0.060	15,500

Output Data

Environmental Test	Sample Size	Average Dent (mils)	Standard Deviation (mils)	Dent (mils)	
				Min.	Max.
Control	25	15.6	0.99	12.9	17.1
Sequential Tests # 1-4	15	14.4	1.89	9.1	16.3
Test # 5 (4 weeks)	25	15.7	0.87	12.5	16.9
Test # 5 (8 weeks)	20	13.8	0.88	12.2	15.9
Test # 6	21	16.3	0.96	13.0	18.1

Environmental Tests

1. Mil-Std-353, Transportation Vibration
2. TN-1441-VIA, Aircraft Vibration
3. Mil-Std-300, Jolt
4. Mil-Std-301, Jumble
5. Mil-Std-304, Temperature and Humidity Cycling
6. Water Pressure Test, 440 psi (24 hours)

Table 4

Sensitivity Data for the High Temperature Detonators

Solder	Sample Size	Mean Capacitance (mfd)	50% Firing Level (ergs)	Standard Deviation (log units)
Control Crimp only, no solder	60	1.62	20,000	0.036
Tin-Antimony 95/5	68	1.64	20,500	0.032
Lead-Silver 97/3	72	1.56	19,500	0.044

Table 5
Output Data of the High Temperature Detonators

Cup Material	Sealing Material	Sample Size	Average Dent (mils)	Standard Deviation (mils)	Dent Range	
					Minimum (mils)	Maximum (mils)
400°F Detonator (Output on Steel Block)						
Gilding Metal	Scotchcast Resin	15	19.0	0.735	17.7	20.6
Gilding Metal	Solder-Tin Antimony (95/5) M.P. - 450°F	36	19.2	0.791	17.0	20.6
Brass	Scotchcast Resin	15	18.0	0.802	16.1	19.6
Brass	Solder-Tin Antimony (95/5) M.P. - 450°F	37	18.0	0.840	16.2	20.1
500°F Detonator (Output on Aluminum Block)						
Gilding Metal	Scotchcast Resin	15	30.9	2.142	27.4	33.9
Gilding Metal	Solder-Lead Silver (97/3) M.P. - 600°F	33	31.3	1.425	27.1	33.3
Brass	Scotchcast Resin	15	30.5	3.193	24.6	34.1
Brass	Solder-Lead Silver (97/3) M.P. - 600°F	37	29.5	1.704	24.2	32.4

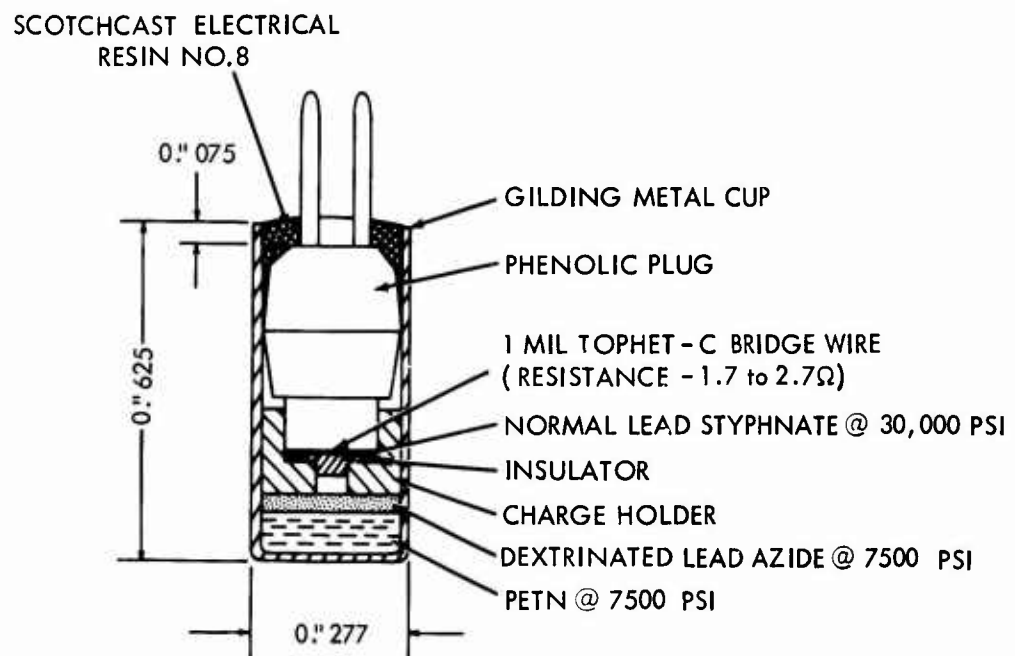


FIG. 1 TYPICAL DETONATOR SEALED WITH SCOTCHCAST RESIN

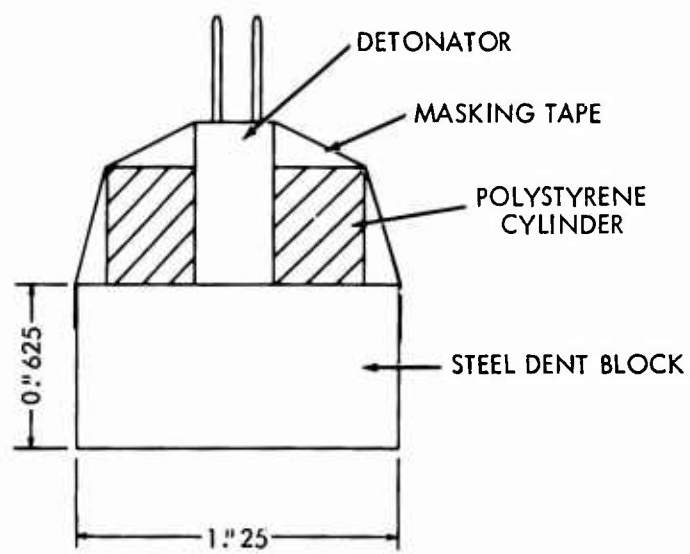


FIG. 2 STEEL DENT TEST ARRANGEMENT USED TO MEASURE OUTPUT

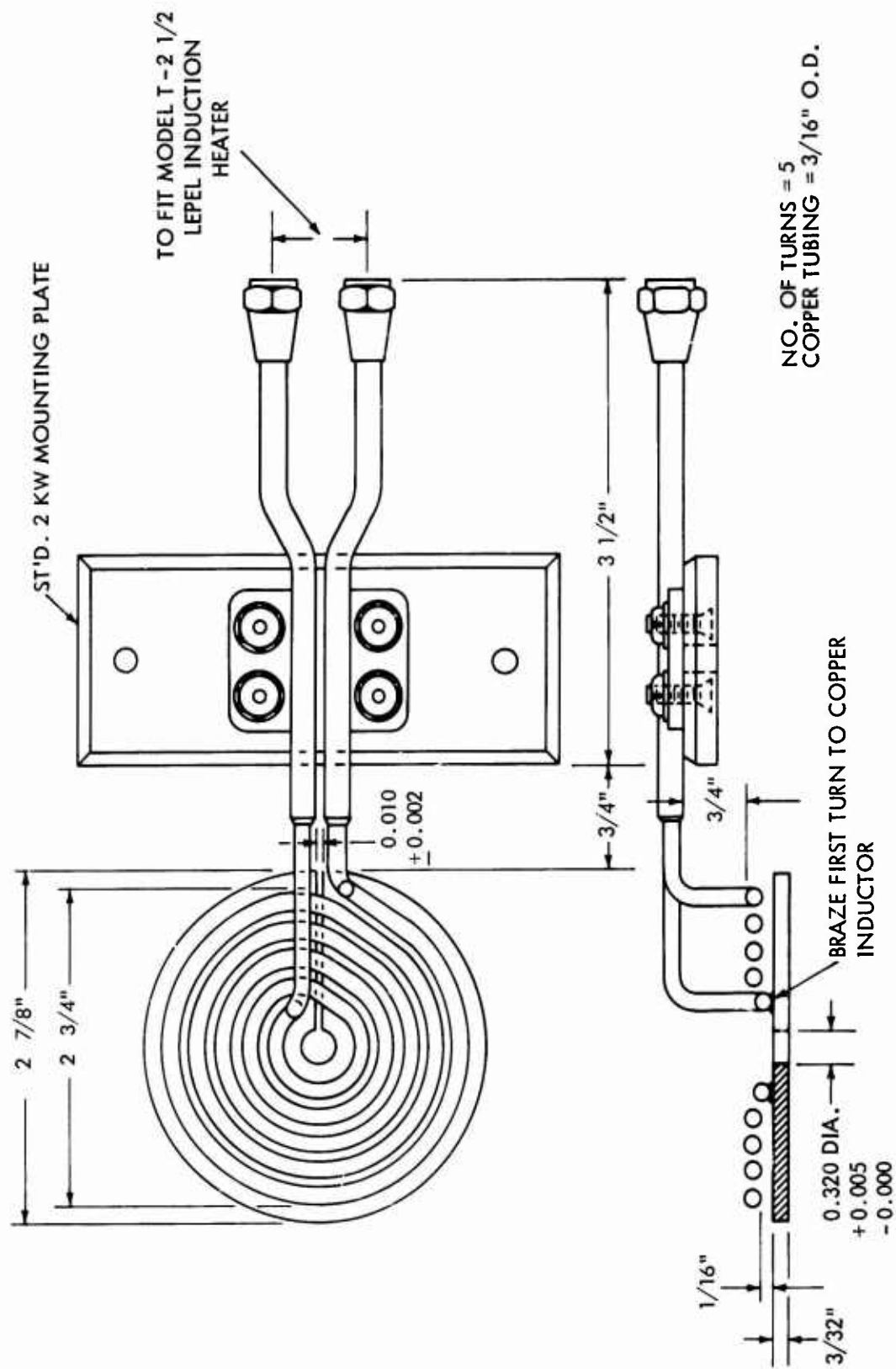


FIG. 3 UNJ - COIL TRANSFORMER DISC

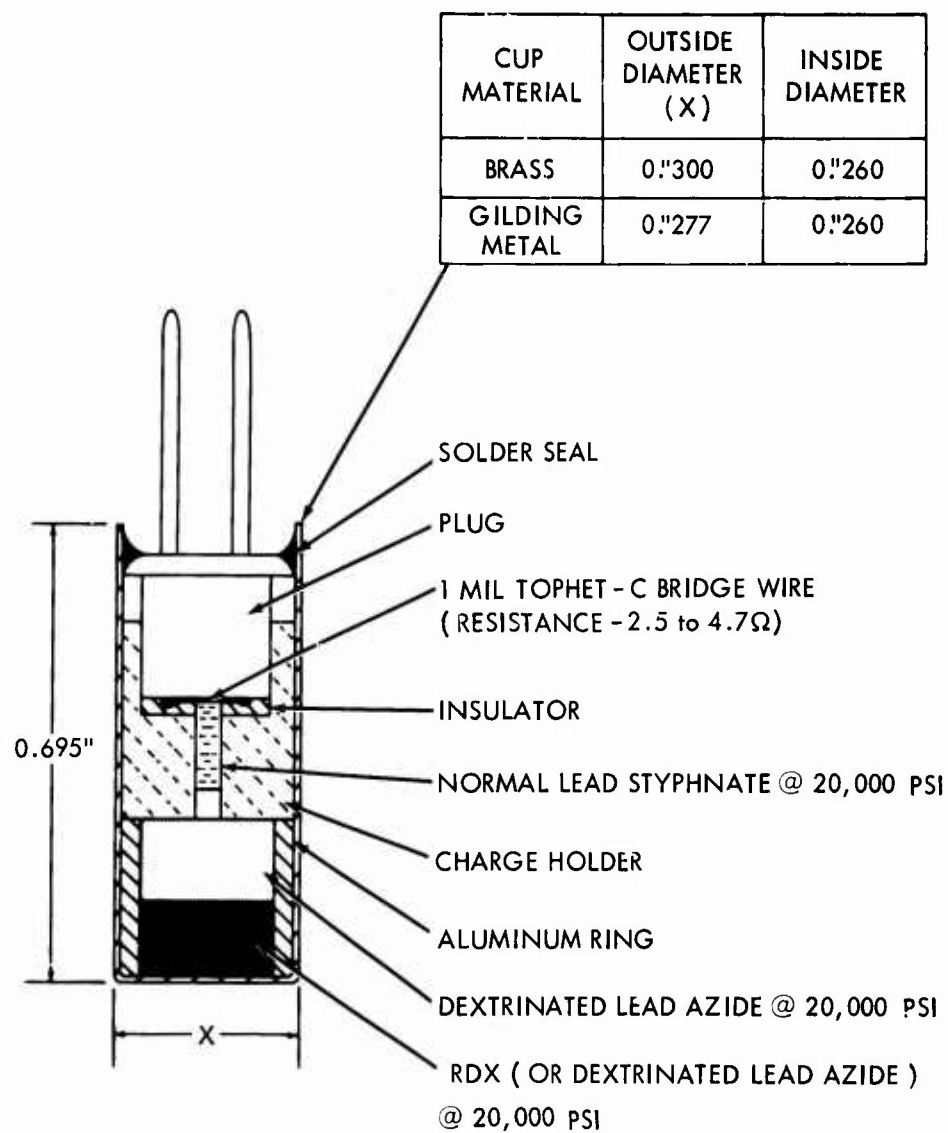


FIG. 4 TYPICAL HIGH TEMPERATURE
DETONATOR

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Epoxy resins, solder amalgams, and high temperature solders were investigated for use in hermetically sealing electro-explosive devices. An epoxy resin gave a non-hermetic, but a water tight seal, solder amalgams gave poor seals, and solders with melting points up to 600°F were successfully used to hermetically seal electroexplosive components.		

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2. Gallium Amalgams						
3. Epoxy resin						
4. High Temperature Solders						
5. Electroexplosive Components						

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14. **KEY WORDS:** Key words are technically meaningful terms or short phrases that characterize a report and may be used as index entries for cataloging the report. Key words must be selected so that no security classification is required. Identifiers, such as equipment model designation, trade name, military project code name, geographic location, may be used as key words but will be followed by an indication of technical context. The assignment of links, roles, and weights is optional.

Security Classification

Naval Ordnance Laboratory, White Oak, Md. (NOL technical report 65-41) AN INVESTIGATION OF SEALING TECHNIQUES FOR ELECTROEXPLOSIVE DEVICES, by Louis J. Mentesi. 25 May 1964. 12p. illus., charts, tables. BuWeps task RUME-45000/212-1/FO08-08-11. UNCLASSIFIED Epoxy resins, solder amalgams, and high temperature solders were investigated for use in hermetically sealing electroexplosive devices. An epoxy resin gave a non-hermetic, but a water tight seal, solder amalgams gave poor seals, and solders with melting points up to 600°F were successfully used to hermetically seal electroexplosive components.	1. Electro-explosive devices 2. Epoxies 3. Solders - High temperature I. Title II. Mentesi, Louis J. III. Project Abstract card is unclassified.
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