INTERIM DEVELOPMENT REPORT #1

FOR

PRECISION VOLUME RESISTOR

SEPTEMBER 20, 1963

INTERNATIONAL RESISTANCE COMPANY
401 NORTH BROAD STREET
PHILADELPHIA 8, PENNSYLVANIA

FOR

NAVY DEPARTMENT BUREAU OF SHIPS

(CLASSIFICATION-UNCLASSIFIED)

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18 JUNE 1963 TO 31 AUGUST 1963

INTERNATIONAL RESISTANCE COMPANY
401 NORTH BROAD STREET
PHILADELPHIA 8, PENNSYLVANIA

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FOR

NAVY DEPARTMENT BUREAU OF SHIPS ELECTRONICS DIVISION

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ABSTRACT

An investigation of materials and fabrication techniques capable of leading to a precision, stable, volume resistor composed of inorganic materials has been initiated.

The high temperature deposition of carbon into a porous ceramic has been found to produce resistors with excellent properties. Because of the increase in temperature coefficient of resistance with range, this method is only useful for very low-range resistors ($<50\Omega$). Limited performance tests were completed on these resistors.

Differential thermal analysis and thermogravimetric analysis studies were made on materials and materials systems of possible use for resistor fabrication.

Preliminary work has been carried out on a Pd-alumina-glass resistor body composition which shows promise for the independent control of range and temperature coefficient. A method for obtaining a good electrical and mechanical termination to this composition has been developed.

PURPOSE

The purpose of this contract is to develop a precision, stable volumetric element resistor which has the following characteristics:

a. high reliability with respect to catastropic failure

b. compatible compositional system over the whole resistance range.

c. performance characteristics in the MIL-R-10509D class.

In addition, the resistor shall be produced by dispersing a conductor (or semiconductor) into an insulating matrix. Both conductor and insulator shall be selected only from inorganic materials such as carbon, palladium, nickel, carbides, borides, glasses and oxide ceramics. The effects of materials and processing parameters on temperature coefficient and resistance value shall be investigated. The resistor shall be equal to or smaller than style RC20 (MIL-R-11D) in size and cover the range 100 to 100,000 ohms.

INTRODUCTION

During the period covered by this quarterly report, work has been carried out on the deposition of carbon into a porous ceramic body, thermogravimetric and differential thermal analyses of conductive materials, the dispersion of palladium in a ceramic body, and termination methods. The results of these investigations are covered in detail in the ensuing sections of this report.

Performance data on resistors prepared by the deposition of carbon into a porous ceramic has been collected and summarized in the appendix. No further work is planned on this process since acceptable resistor characteristics were obtained only below 50 ohms which is outside the range limitations of the contract.

A list of the personnel working on this project is included in the appendix.

DETAILED FACTUAL DATA

I. Deposited Carbon In A Porous Ceramic Body

This approach to a stable volume resistor is based on the deposition of carbon at ≈2200 °F from a nitrogen gas stream containing methane. Deposited carbon film type resistors are manufactured by this general method. In order to prepare a volume resistor, a porous ceramic rod is made first and then the carbon is deposited in the pores of the ceramic.(*) For a volume resistor, the approach has the disadvantage that higher resistance ranges cannot be made by cutting a helix as is done with film type carbon resistors. This means that range can only be controlled either by reducing the pore size or by changing the deposition rate and therefore the film thickness. The latter approach was chosen for this investigation.

* Patent application filed.

The porous ceramic body is prepared as follows:

1. Composition

Al_2O_3	-	Norton alumina of ~2 micron particle size
S86	-	A propreitary IRC glass frit
M_gCO_3	-	Reagent grade
Veegum	-	A commercially available binder

2. Mixing Operation:

The composition is mixed in a one gallon ball mill with porcelain balls for 20 hours.

3. Drying and Mulling Operations:

The wet mixture after ball milling is pan dried at 110 °C and plasticized in a muller by gradually adding distilled water.

4. Extrusion:

The plasticized material is extruded into rods with a diameter of .073".

5. Calcination:

The extruded rods are dried at room temperature, broken into 3" sections and fired at 1050 °C for 10 hours in a Harper conveyor furnace.

The fired rods are cut into 0.150" length slugs prior to carbon deposition. The carbon deposition apparatus is shown in Figure 1 and is essentially the same as equipment used for the production of carbon film resistors. The process is carried out as follows:

1. A batch of slugs is placed in a rotating silica flask which is heated to 2200°F (1204°C).

2. Nitrogen is introduced into the flask to flush out the air.

3. When the temperature of the furnace again reaches 2200°F, methane (CH_4) is passed into the chamber with a flow rate between 10 ml/min and 100 ml/min for 1 1/2 hours.

4. During the pyrolysis of methane, carbon is deposited into the porous ceramic.

5. The slugs are electroless plated with copper.

6. In order to remove the copper from the cylindrical surfaces of the slugs, they are passed through a centerless grinder.

7. The copper plated ends of the slugs are tinned and terminated with nail-head leads.

8. The subassembly is molded in a phenolic molding material to conform to style RC-07 (MIL-R-11D).

In this approach, resistance range is controlled by varying the flow of methane (see Figure 5) provided composition and deposition parameters are kept constant. Resistors manufactured by this process have been tested according to MIL-R-10509D specifications and the results are given in Table I.

II. Conductive Materials Study

The processes used to form volumetric resistors from inorganic materials involve high temperature firings which can lead to complicated chemical interactions between the conductor-insulator materials and with the atmosphere. In order to study these interactions, differential thermal analyses (D.T.A.) and thermogravimetric analyses (T.G.A.) were performed on the individual components and on the combined systems. The D.T.A. apparatus is used to determine at what temperatures endothermic and exothermic reactions occur. Such reactions can be chemical reactions between materials, oxidation, phase changes such as melting or recrystallization, or decomposition. The T.G.A. indicates at what temperatures a gain or loss of sample weight occurs. This can be related to decomposition of the sample or reaction with the atmosphere.

Materials studied include palladium, silver, tungsten, chromium, chromium silicide, and mixtures of these with ceramic materials.

1. Palladium Black and Silver

Both D.T.A. and T.G.A. were made of palladium black, silver powder, 56% Pd-44% Ag prealloyed powders and co-precipitated mixtures of 56% Pd-44% Ag prepared by the Bishop Co. In every case a 10 °C/min heating rate was used. Different atmospheres such as O_2, He, and vacuum were used in the D.T.A., but air only, was used in the T.G.A.

Several thermo-grams are shown in Figures 6 to 14 and the results are presented in Tables II, III and IV.

From the D.T.A. and T.G.A. curves it can be concluded that pure palladium oxidizes to PdO between 300 and 650 °C in air and then the PdO decomposes to Pd between 850 and 910 °C. There is a clear cut temperature interval between 650 and 850 °C during which no change is observed. Pure silver resists oxidation and when mixed with palladium retards oxidation and accelerates the decomposition of palladium oxide. The oxidation and decomposition temperatures are different for the mixture, the co-precipitated, and the prealloyed 56/44, Pd/Ag samples. The order of oxidation is: mixture ~240 °C, co-precipitate ~300 °C and prealloyed ~320 °C. The order of decomposition is: mixture ~500 °C, co-precipitate ~540 °C and pre-alloyed ~650 °C.

The D.T.A. cooling curves indicate that a slight oxidation (estimated 5-10%) of Pd occurs on cooling through the temperature interval ~750-660 °C. A quantitative measure of this oxidation has not yet been obtained by T.G.A. due to instrumental difficulties.

Approximately 2% of PdO was calculated from the T.G.A. curves to be in the original palladium black powder.

2. Tungsten, Chromium and Chromium Silicide

Several other materials which might serve as the conductive phase in a volume resistor were studied by T.G.A.

Tungsten in air was found to oxidize completely to WO_3 below 600 °C. No decomposition of the WO_3 was observed up to 1000 °C, the maximum temperature of the equipment.

The reaction of chromium powder with air began at about 500 and continued up to 1000 °C. It was estimated that the weight gain up to 900 °C was about 2-3% by T.G.A. Evidently the initial oxide layer protects the chromium particles from further oxidation.

Chromium silicide was found to begin oxidizing at about 480 °C with a slowly increasing rate up to about 700 °C. Above 700 °C the oxidation rate increased rapidly.

3. Resistor Material Systems

A D.T.A. curve of a S314 glass (a proprietary borosilicate glass) and palladium black mixture showed an exothermic reaction at 360 °C due to oxidation of Pd and an endothermic reaction at 560 °C due to decomposition of PdO. An additional endotherm at 680 °C was related to the softening point of S314 glass.

The reactions between S314 glass, palladium black, Al_2O_3 and Veegum (a hydrated magnesium aluminum silicate used as a binder) were

found to be quite complicated. An endotherm at 100 °C was related to evaporation of water. The exotherm due to oxidation of Pd was found at 400 °C and the decomposition endotherm at 780 °C. Exothermic peaks at 820 °C and 900 °C are probably due to the decomposition of veegum and a reaction between S314 glass and alumina respectively.

III. Palladium Dispersed In A Ceramic Body

Palladium metal is known to have a positive temperature coefficient of resistance of about 3800 ppm/°C. Preliminary experiments have indicated that PdO has a temperature coefficient of about -780 ppm/°C. The results of the D.T.A. and T.G.A. study indicate that the ratio Pd/PdO can possibly be fixed by controlling the firing temperature. This in turn should make control over the temperature coefficient of resistance attainable. Since palladium metal is also commercially available in a very finely divided form (~0.1 particle diameter by electron microscope) it was decided to carry out a series of experiments utilizing Pd as the conductive phase in a ceramic matrix. Details of these experiments follow:

1. Formulation

Eight formulations were prepared with the following compositions:

Batch No.	Al_2O_3%	S86	S314%	Pd%	Veegum %
(1)	47.5	28.5		18.0	6.0
(2)	46.25	27.75		20.0	6.0
(3)	45.0	27.0		22.0	6.0
(4)	39.0		39.0	16.0	6.0
(5)	38.0		38.0	18.0	6.0
(6)	37.0		37.0	20.0	6.0
(7)	27.0		27.0	40.0	6.0
(8)*	38.0		38.0	18.0	6.0

* Same composition as (5), except extruded as a hollow cylinder for talon lead assembly.

2. Milling and Mulling Processes

A 200 gram batch of each formulation was ball milled for 20 hours in a half gallon mill with 500 grams of porcelin balls and 500 ml of distilled water. The batches were then pan dired and plasticized in a muller with distilled water.

3. Extrusion

The plasticized material was extruded under 2500 PSI into rods with a diameter of 0.073". The extrusion equipment is shown in Figure 2.

4. Firing Conditions

The green rods were fired in a BTU controlled atmosphere furnace (see Figure 3) using several different time-temperature-atmosphere combinations. Only the results on air firing with a 1/2 hour cycle are included in this report.

5. Strength

A modified Hunter spring tensile strength tester was used to determine the force necessary to break a one inch rod loaded at the center. The rods were found to break under a force of 2-4 lbs. (see Table V) This is considered adequate strength for resistor fabrication.

6. Results

Resistance and temperature coefficient of resistance (T.C.) are tabulated in Table VI and Figure 15.

In the past resistors prepared from palladium-ceramic systems formed by dry pressing were found to have T.C.'s as low as 100 ppm/°C. In this work resistor bodies were formed by extrusion in order to obtain bodies with greater uniformity of resistance. The extruded rods are denser than the dry-pressed bodies and this appears to decrease the oxidation of palladium in the interior of the body during firing. It has been found that in a body with a high concentration of Pd, the resistance through the center is much lower than the surface. A body with a low concentration of Pd is conductive only on the surface. These observations can be explained on the basis that the molar volume of PdO is 1.6 times as large as the molar volume of Pd. With a low percentage loading of Pd there are very few interconnected chains of Pd particles running through the resistor, therefore the resistance is very high. However, at the surface Pd particles have been oxidized to form larger PdO particles which causes more interconnected chains to form and therefore the resistance is decreased. In the case of a high loading of Pd, there are many interconnected chains of Pd running through the body so the resistance is low. Pd at the surface has oxidized to PdO which has a higher resistivity than Pd, and therefore the surface has a higher resistance relative to the core of the body.

This concept can also be used to explain the relative variations of resistance with firing temperature and atmosphere. For instance, the sample with a high percentage of Pd (batch 7 Table VI) has a resistance which is much lower when fired at 900 °C in nitrogen (< 1Ω) than when it is fired at 770 °C in air (300Ω). Resistors with a low percentage of Pd (batches 1 to 6) have a resistance which is much higher when fired at 900 °C in air than at 770 °C in air because PdO is subject to decomposition at the higher temperature and conducting chains are broken.

If the above explanation is correct, better control of the oxidation of Pd throughout the body is necessary to achieve the proper Pd/PdO balance required for low temperature coefficients of resistance. This work is continuing. Performance data on these Pd-ceramic resistors is not yet available.

IV. The Termination Problem

In order to evaluate the electrical characteristics of a resistor body, a good electrical and mechanical termination must be made to it. Several termination methods have been investigated for use with the Pd-ceramic body.

1. Organic Contact Paint

Resistor bodies from batch 8, which was extruded as a hollow cylinder, were bonded to talon leads with an organic resin loaded with carbon. This technique decreased the active length of the resistor and also produced an unstable electrical contact. No further work is planned on this termination method.

2. Electroless Nickel

The resistor bodies were potted in an organic resin in such a way that the ends were exposed to a commercially available electroless nickel plating solution. After sufficient nickel had plated onto the ends, the bodies were removed from the plating material and soldered with 95/1.5/3.5 Pb/Ag/Sn solder to nail-head leads. It was found that this method produced a mechanically weak termination.

3. Electroless Copper Plating

A satisfactory termination was finally achieved with an electroless copper plating solution manufactured by Enthone Inc., New Haven, Conn. using a procedure similar to the one described above. The assembled units had sufficient strength to withstand the molding operation. These units are now undergoing electrical testing.

CONCLUSIONS

Work during this period has shown that volume resistors prepared by the deposition of carbon into a porous ceramic body have excellent properties at low resistance values. Because the temperature coefficient of resistance increases rapidly with range, this resistor system is unsuitable for high ranges. No further work will be carried out on this process.

The D.T.A. and T.G.A. study has shown that silver additions have a pronounced effect on the oxidation characteristics of palladium. Chromium silicide and chromium were found to be somewhat resistant to oxidation and might be useful as the conductor phase in an inorganic resistor system if they can be prepared as finely divided powders.

In the palladium-Al_2O_3 - glass system, a wide variation in T.C. and range has been found depending on firing conditions. A model has been formulated which relates these variations with the Pd/PdO ratio attained during firing. In order to control the Pd/PdO ratio, longer firing cycles and/or more porous ceramic bodies are probably required.

PROGRAM FOR THE SECOND QUARTER

1. Work will continue on resistor bodies based on the Pd/PdO system. Attempts will be made to achieve greater uniformity of oxide formation throughout the resistor body so that lower temperature coefficients or resistance are obtained. For a better understanding of this system, an investigation will be made of the resistivity and temperature coefficient of a PdO layer on Pd.

2. A study will be made of the properties of the alumina-glass matrix system as related to composition and firing conditions. It will then be possible to determine what effect the conductor phase has on the complete resistor body formulation.

3. Finely-divided, colloidal suspensions of conductive materials will be prepared as a technique to achieve more uniform dispersion of the conductive phase throughout the ceramic matrix. Colloidal dispersions of both nickel and palladium will be investigated.

PERSONNEL

LABOR HOURS CHARGED TO PROJECT 88-72

PRECISION VOLUME RESISTOR

INCEPTION THRU AUGUST 31, 1963

EMPLOYEE		TOTAL HRS.
K. Merz	Associate Director, Passive Component, Research and Development	31.0
C. Huang	Senior Research Chemist	59.5
W. Ebling	Project Manager (Volume Resistor Dev.)	21.0
H. Saba	Junior Research Technician	6.5
G. Vaccaro	Junior Research Ceramist	194.0
M. Burk	Senior Research Ceramist	75.0
W. Fishl	Senior Research Analyst	1.0
J. Blessing	Senior Research Technician	6.5
	TOTAL HOURS (ENGRG)	394.5

APPENDIX

Figure 1
B. T. U. Controlled Atmosphere Furnace

Figure 2
Extruder

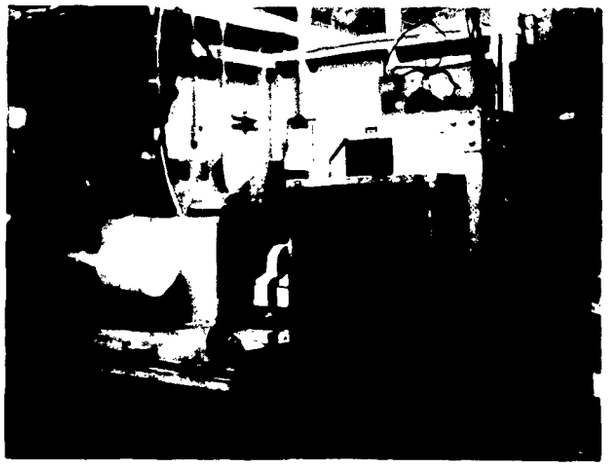


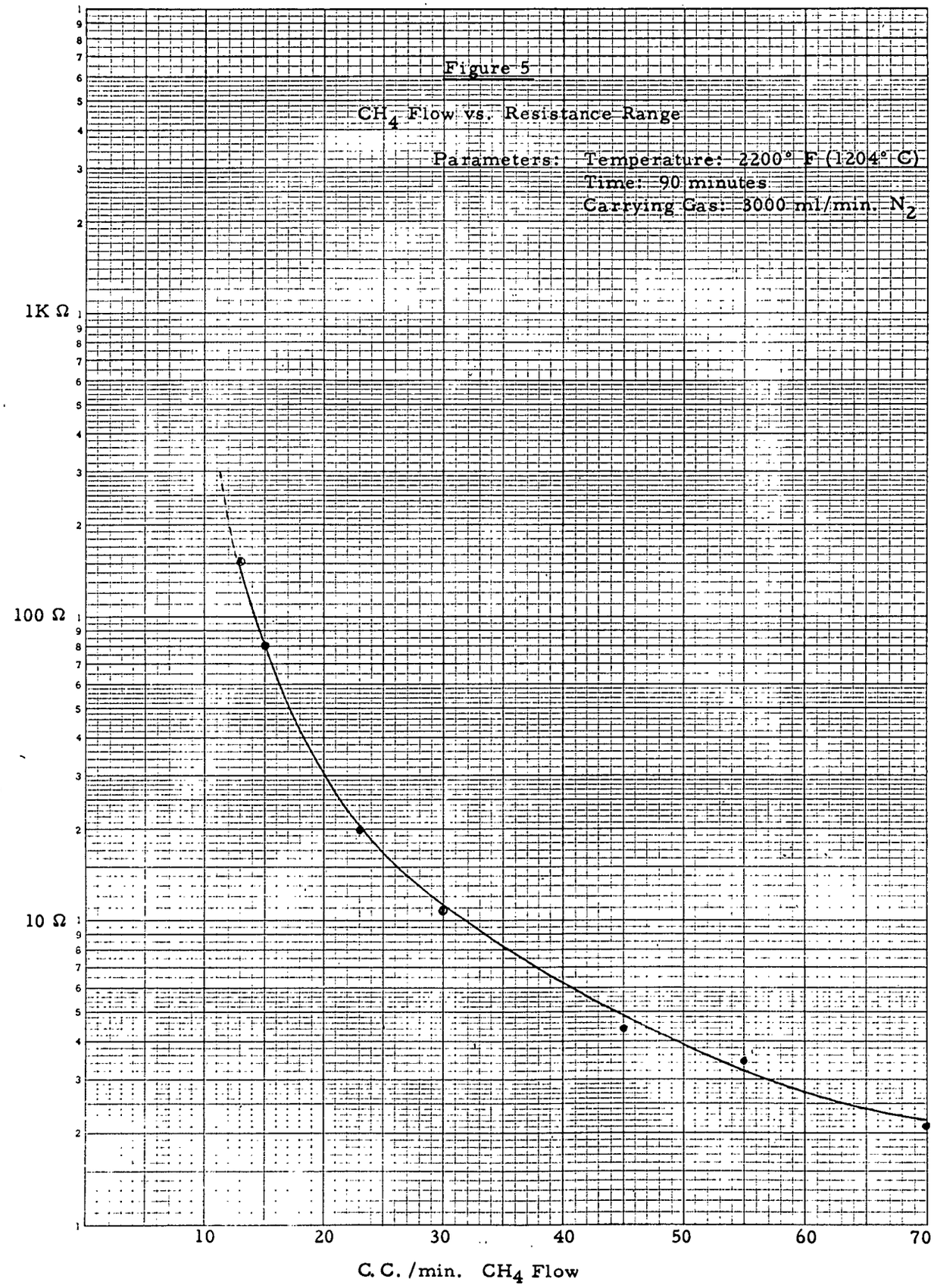
Figure 3
Carbon Deposition Chamber & Apparatus

Figure 4
Centerless Grinding Machine

Figure 5

CH_4 Flow vs. Resistance Range

Parameters: Temperature: 2200° F (1204° C)
Time: 90 minutes
Carrying Gas: 3000 ml/min. N_2



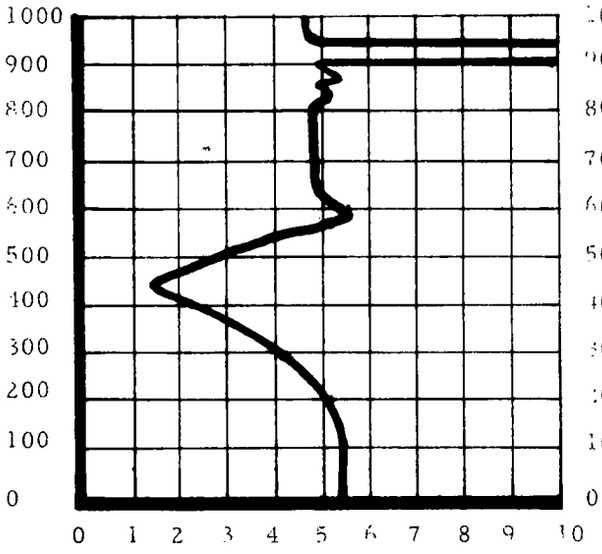


Figure 6. D. T. A. thermogram of Pd black in Oxygen

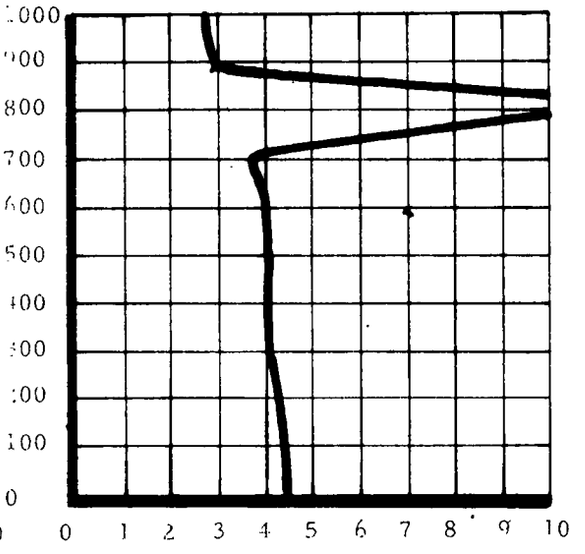


Figure 7. D. T. A. thermogram of PdO in Nitrogen

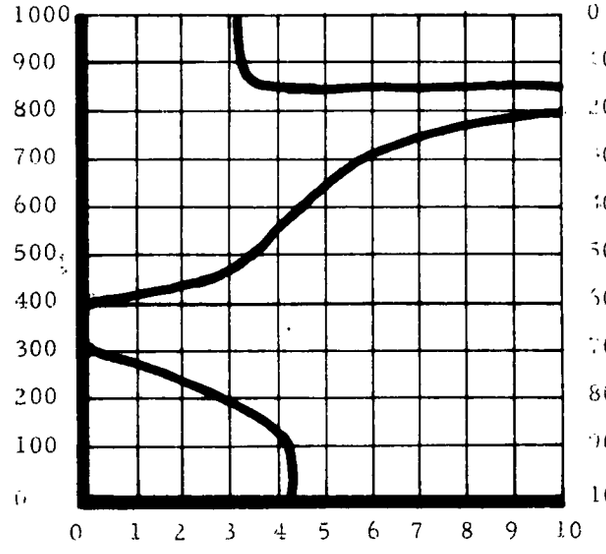


Figure 8. D. T. A. thermogram of 60% Pd-40% Ag mixture in air

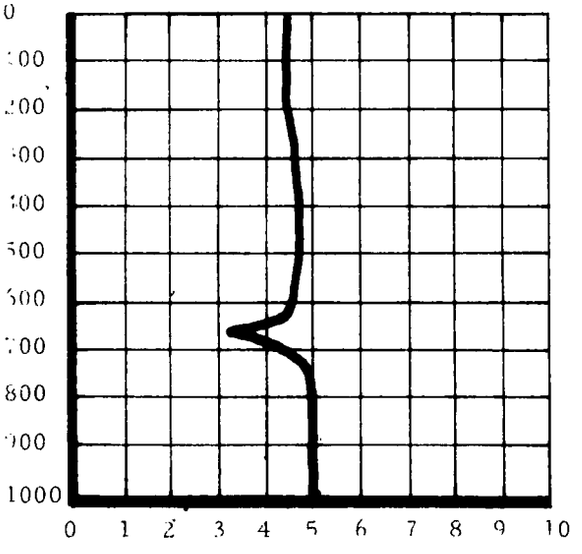


Figure 9. Cooling curve of Figure VIII

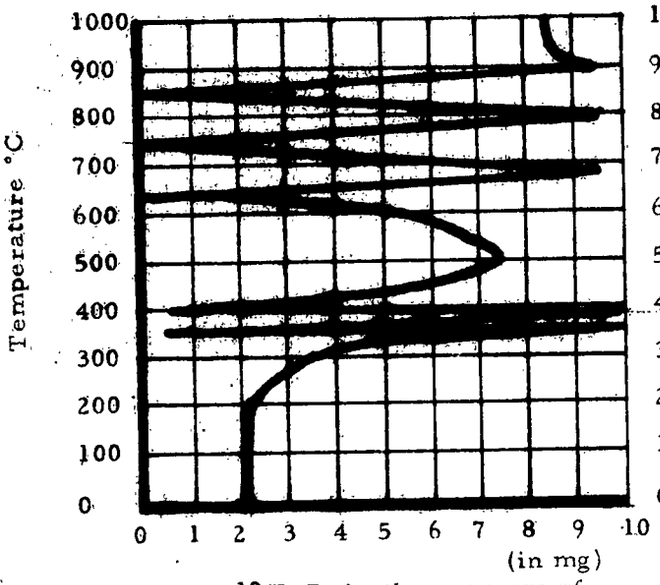


Figure 10. T. G. A. thermogram of 56% Pd-44% Ag mixture in air (330. 5 mg)

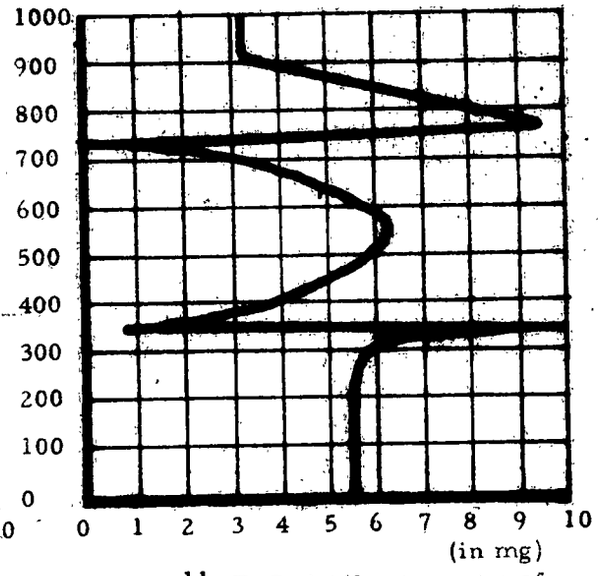


Figure 11. T. G. A. thermogram of 56% Pd-44% Ag Co-ppt in air (153. 5 mg)

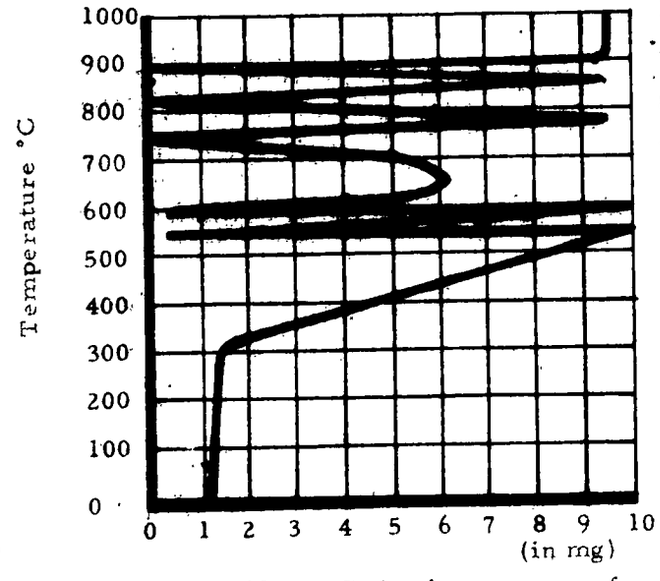


Figure 12. T. G. A. thermogram of 56% Pd-44% Ag alloy in air (429. 2 mg)

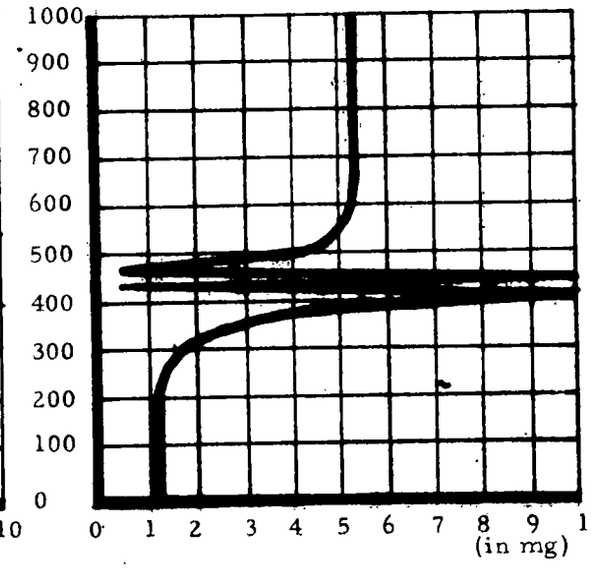


Figure 13. T. G. A. thermogram of W powder in air (24. 1 mg)

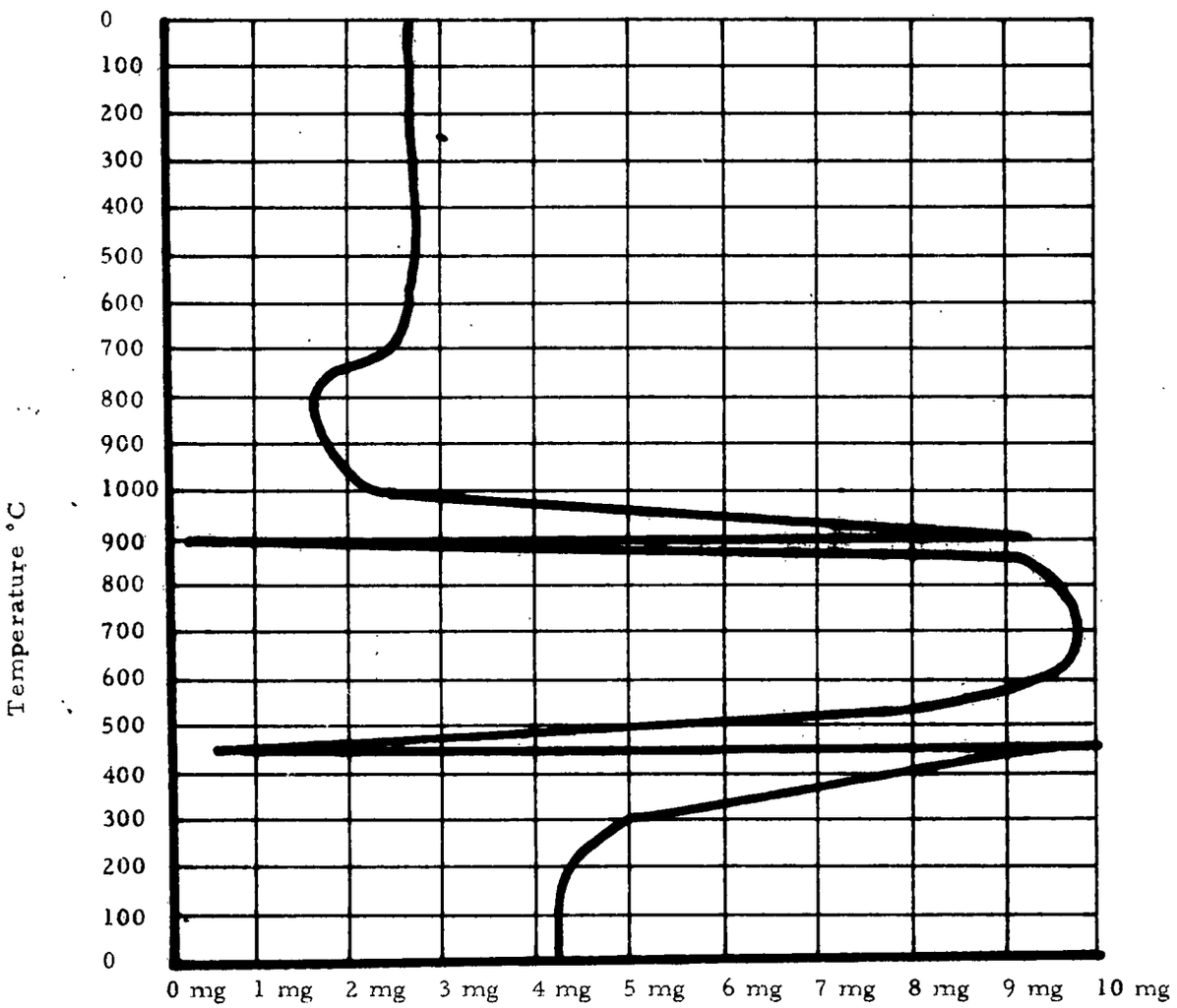


Figure 14. T. G. A. thermogram of Pd black in air, both heating and cooling curves (117. 3 mg)

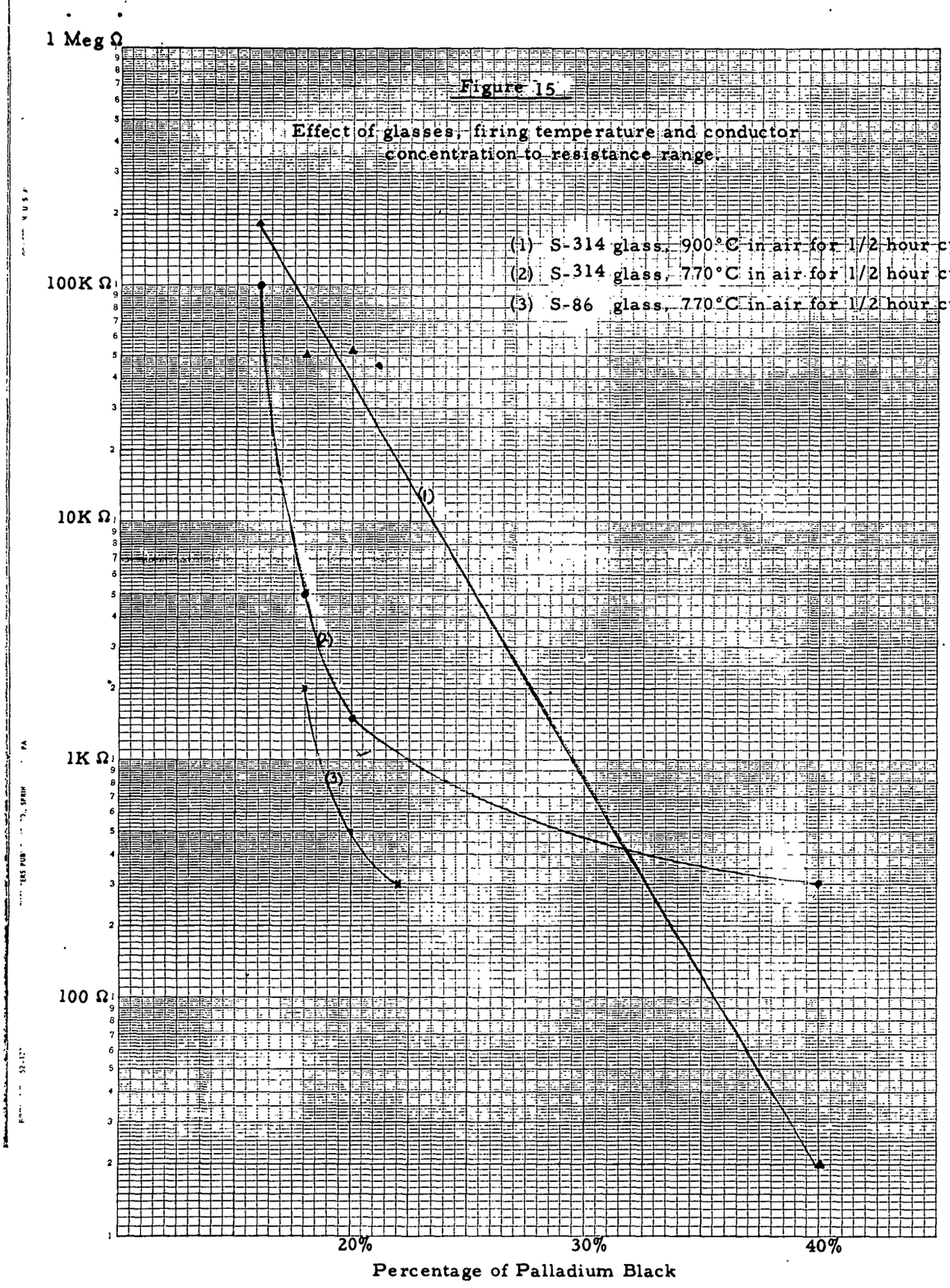


Figure 15

Effect of glasses, firing temperature and conductor concentration to resistance range.

(1) S-314 glass, 900°C in air for 1/2 hour cycle
(2) S-314 glass, 770°C in air for 1/2 hour cycle
(3) S-86 glass, 770°C in air for 1/2 hour cycle

PREPARED BY:

CHECKED BY:

APPROVED BY:

TITLE TABLE I . Performance On Deposited Carbon V

RESISTANCE & DATA Nº		RESISTANCE - TEMPERATURE CHARACTERISTIC P.P.m./°C -55°C	+105°C	VOLTAGE COEFFICIENT % PER VOLT	DIELECTRIC STRENGTH	INSULATION STRENGTH (MEGS.)	DATA Nº	LOW TEMP. OPERATION	TEMPERATURE CYCLE	MOISTURE RESIS LOAD	INSULATION RESISTANCE (MEGS.)	NO LOAD
ALLOWABLE PERCENT CHANGE												
2.1Ω	AVERAGE	+131	+47			ALL	08033	±.03	±.05	+.10	7×10^3	±.1
	SPAN	+158	+162			$>10^5$		±.02	±.07	±.17	MΩ	±.2
07993	FAIL	+87	-12			MΩ		-.09	-.10	±.03		±.0
4.5Ω	AVERAGE	+116	-108				08034	±.07	±.05	±.22	2×10^3	±.4
	SPAN	+161	-86					0.0	±.19	±.26	MΩ	±.5
07994	FAIL	+82	-125					-.20	-.04	±.08		±.1
11.3Ω	AVERAGE	+154	-134				08035	±.05	±.11	±.14	2×10^3	±.3
	SPAN	+162	-120					±.05	0.0	±.24	MΩ	±.8
07995	FAIL	+136	-136					-.14	±.18	±.16		±.0
20.0Ω	AVERAGE	+221	-157				08458	±.11	±.37	±.41	$<10^2$	±.1
	SPAN	+272	-133					±.15	±.77	±.56		±.4
08507	FAIL	+200	-171					-.30	±.22	±.15		±.1
72.05Ω	AVERAGE	+458	-348				08340	±.18	±.37	±.13	6×10^3	±.2
	SPAN	+532	-270					±.32	±.85	±.26	MΩ	±.3
08000	FAIL	+415	-412					-.06	-.05	-.05		±.0
120Ω	AVERAGE	+615	-448				08002	±.36	±.95	±1.18	2×10^4	±1.
	SPAN	+698	-370					±.65	±1.99	±3.76	MΩ	±3.
08002	FAIL	+563	-562					-.17	-.08	-1.59		±.0
	AVERAGE											
	SPAN											
	FAIL											
	AVERAGE											
	SPAN											
	FAIL											
	AVERAGE											
	SPAN											
	FAIL											
	AVERAGE											
	SPAN											
	FAIL											
	AVERAGE											
	SPAN											
	FAIL											

Performance On Deposited Carbon Volume Resistors

TYPE 8-

DATE -

INSULATION STRENGTH (MEGS.)	DATA Nº	LOW TEMP. OPERATION	TEMPERATURE CYCLE	MOISTURE RESISTANCE: LOAD	MOISTURE RESISTANCE: INSULATION RESISTANCE (MEGS.)	MOISTURE RESISTANCE: NO LOAD	MOISTURE RESISTANCE: INSULATION RESISTANCE (MEGS.)	SHORT TIME OVER LOAD	DATA Nº	70° LOAD LIFE HOURS: 48	200	500	700	DATA Nº	TERMINAL STRENGTH	EFFECT OF SOLDERING	DATA Nº	SHOCK TEST	HI-FREQUENCY VIBRATION	DATA Nº	SOLDERABILITY
ALL	08033	±.03	±.05	+.10	7×10^3	+.13	2.5×10^2		07983	±.06	±.02	±.06	±.11	07971	±.21	±.08					
>10^5		+.02	+.07	+.17	meg	+.21	meg			+.09	+.08	+.12	+.18		−.28	−.26					
meg		−.09	−.10	+.03		+.07				−.13	−.16	−.14	−.30		+.08	+.02					
	08034	±.07	±.05	+.19	7×10^2	+.40	5×10^3		07984	±.05	±.04	±.07	+.15	07972	±.11	±.09					
		0.0	+.17	+.16	meg	+.32	meg			+.12	+.15	+.18	+.25		−.18	−.38					
		−.20	−.04	+.08		+.18				−.09	−.05	−.06	+.04		+.02	+.10					
	08035	±.05	±.11	+.14	2×10^2	+.32	8×10^2		07985	−.12	±.13	±.20	+.31	07973	±.07	±.04					
		+.05	0.0	+.14	meg	+.25	meg			−.05	+.17	+.36	+.46		−.17	+.10					
		−.14	+.18	+.12		+.35				−.23	−.27	−.09	+.03		+.01	−.02					
	08458	±.11	+.37	+.41	$<10^2$	+.27	$<10^2$		08464	+.24	±.53	+.28	+.16								
		+.15	+.77	+.24	meg	+.47	meg			±.21	±.45	+.45	+.30								
		−.30	+.22	+.15		+.19				+.08	±.19	+.16	−.6								
	08110	±.18	± 27	± 3	6×10^3	+2.09	2.5×10^3		07990	±.12	±.20	±.33	±.38	07974	±.36	7.16					
		+.32	+.85	+ 3	meg	+3.88	meg			+.57	+1.10	+2.13	+2.64		+.57	+1.15					
		−.06	−.05	− 2		+.11				−.10	−.10	−.13	−.13		−.12	−.15					
	08512	±.36	±.95	±1.18	2×10^4	±1.42	1.8×10^4		07992	+.52	+.54	±.36	±.23	07980	+.37	±.62					
		+.65	+1.99	+3.76	meg	+3.32	meg			+.72	+.92	+.92	+.35		+.97	+1.19					
		−.17	−.08	−1.59		+0.01				+.09	+.26	−.44	−.62		+.08	0.0					

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TABLE II

D. T. A. data **

Material	Temp. Range	Peak Temp.	Reaction	Atmos.
Pd Black	200 - 650 °C	450 °C	Exothermic	O_2
	860 - 900 °C	490 °C	Endothermic	
Pd Black	200 - 650 °C	460 °C	Exothermic	H
	780 - 870 °C	830 °C	Endothermic	
Ag		No Reaction		O_2
*Pd - Ag	200 - 500 °C	350 °C	Exothermic	O_2
(mixture)	500 - 850 °C	680 °C	Endothermic	
*Pd - Ag	200 - 500 °C	300 °C	Exothermic	Air
(Co-ppt)	500 - 810 °C	800 °C	Endothermic	
*Pd - Ag	150 - 550 °C	400 °C	Exothermic	Air
(mixture)	550 - 820 °C	810 °C	Endothermic	

* -56% Pd - 44% Ag

** The exothermal reactions are due to the oxidation of Pd. ($2\ Pd + O_2 \longrightarrow 2\ Pd\ O$)

The endo thermal reactions are due to the decomposition of PdO
($2\ PdO \longrightarrow 2\ Pd + O_2$)

TABLE III

T.G.A. Data

Material	Temp. Range	Wt. Gain	Wt. Loss	Orig. Wt.	% Gain	% Loss
Pd Black	200 - 650°C	16.5mg		124.2mg	13.29%	
	850 - 910°C		18.5mg			13.15%
Ag Powder		No Change In Weight				
* Pd - Ag	320 - 650°C	25.1mg		429.20mg	5.85%	5.86%
Pre-Alloyed	650 - 870°C		26.6mg			5.86%
* Pd - Ag	300 - 540°C	10.6mg		153.5mg	6.91%	
(Co-ppt)	540 - 890°C		12.8mg			7.80%
* Pd - Ag	240 - 500°C	24.7mg		330.5mg	7.47%	
(mixture)	500 - 990°C		28.8mg			8.11%
Pd Black	200 - 650°C	16.3mg		122.4mg	13.32%	13.27%

TABLE IV

D.T.A. Cooling Curves

	Temp. Range	Peak Temp.	Reaction	Atmos.
*Pd - Ag (Pre-Alloyed)	650 - 500 °C	610 °C	Exotermic	Air
*Pd - Ag (Mixture)	750 - 660 °C	680 °C	Exothermic	Air

TABLE V

Physical Properties of the Rods fired at 770 °C in Air for 1/2 hour.

Batch No.	Green Diameter	Fired Diameter	Beam Strength
(1)	.0725"	.0725"	3 lb./in.
(2)	.0745"	.0730"	4 lb./in.
(3)	.0725"	.0715"	2 1/2 lb./in.

TABLE VI

RESISTANCE RANGE AND TEMPERATURE COEFFICIENT

Batch No.	Firing Temp.	Atmos.	Firing Time	Resistance Range	T.C. 25 - 105°C
(1)	770°C	Air	1/2 hr.	2K Ohms	-92 P.P.M/°C
(1)	900°C	Air	1/2 hr.	N.C.	-
(2)	770°C	Air	1/2 hr.	500 Ohms	-
(2)	900°C	Air	1/2 hr.	N.C.	-
(3)	770°C	Air	1/2 hr.	300 Ohms	-
(3)	900°C	Air	1/2 hr.	N.C.	-
(4)	770°C	Air	1/2 hr.	100K Ohms	-
(4)	900°C	Air	1/2 hr.	185K Ohms	-2265 P.P.M/°C
(5)	770°C	Air	1/2 hr.	5K Ohms	
(5)	900°C	Air	1/2 hr.	50K Ohms	-1803 P.P.M/°C
(6)	770°C	Air	1/2 hr.	1.5K Ohms	
(6)	900°C	Air	1/2 hr.	105K Ohms	-1696 P.P.M/°C
(7)	770°C	Air	1/2 hr.	300 Ohms	
(7)	900°C	Air	1/2 hr.	25 Ohms	+2898 P.P.M/°C
(8)	770°C	Air	1/2 hr.	42K Ohms	-422 P.P.M./°C
(8)	900°C	Air	1/2 hr.	93K Ohms	-1902 P.P.M/°C