NASA CR-111959

N71-35619

DEVELOPMENT OF A PLATINUM -THORIUM OXIDE ALLOY FOR RESISTOJET THRUSTER USE

FINAL REPORT

by

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PREPARED UNDER CONTRACT NO. NAS1–10433

for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

LANGLEY RESEARCH CENTER

JULY 1971

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SUMMARY

Testing of platinum-thoria alloys has established that under the conditions determined earlier for the preparation of these alloys, platinum with 0.6% thoria is the optimum composition from the point of view of manufacturing and usage of the alloy. Stress-rupture testing has shown a considerable increase in stress-rupture life for platinum-thoria alloys over the commercial platinum-rhodium alloys. Adding thoria to platinum at the 0.6% level has relatively little affect on the room temperature mechanical and electrical properties. The principal effect of the thoria is to provide a stable, elongated grain structure, thereby improving the long term, load carrying capacity at elevated temperatures. Tubing was fabricated without difficulty from the platinum 0.6% thoria alloys. Joining tests were carried out by electron-beam welding, dap welding, diffusion bonding, spot welding and high temperature brazing.

INTRODUCTION AND OBJECTIVES

This project relates to the development of materials of construction for a biowaste resistojet. This device uses a series of concentric tubes, heated by passing an electrical current through the tubing, to heat a propellent gas passing through the tubing. The gas may contain methane, CO_2, H_2, H_2O (steam), NH_3, N_2 and O_2 (though not concurrently). Testing at the Marquardt Company revealed that CO_2 passing through platinum-20% rhodium tubing at 1000-1500°C formed a rhodium carbonyl which vaporized and reduced the rhodium content of the alloy. This also degraded the mechanical properties of the tubing.

As alternate materials for platinum-20% rhodium, platinum-iridium alloys and platinum reinforced with thoria were proposed. Since relatively little was known of the properties of the latter system, this project was initiated with the view to studying the basic mechanical properties and the manufacturing properties. In particular, it was necessary to find what level of thoria would provide the optimum properties with regard to both the manufacturing and the usage of a platinum-thoria alloy. Having established this, the remainder of the project was then to be concerned with the production of tubing and wire test samples, electrical properties and methods of joining the thoriated alloy.

EXPERIMENTAL WORK AND RESULTS

1. Fabrication and Stress Rupture Testing of Platinum-Thoria Alloys

In this initial phase of the work, the object was to investigate the high temperature, stress-rupture properties of platinum-thoria alloys starting with 0.5% thoria. The thoria content of alloys tested was to be increased until the properties were unacceptable either from the point of view of mechanical strength or manufacturing procedures. The criterion for alloy suitability was improved stress rupture-properties along with the ability to be formed by cold working in the manner normal for platinum-base alloys. An exception to this was planned in that high thoria content alloys known to be unworkable by the techniques used for conventional platinum alloys were to be extruded, worked to wire, and tested for stress-rupture properties.

A Refining Lot No. 190-A of ammonium platinum chloride was reserved for this project. A spectrographic analysis of this material is given in Table I. This analysis showed the platinum content to be 99.994% with no base metals greater than six parts per million. Initial testing showed this material to be suitable in its response to stress-rupture testing and all further testing was done on platinum produced from Lot 190-A. Prior to the inception of this project, a "standard schedule" of powder production, thoriating, and wire production was worked out. This standard schedule involved only conventional powder metallurgy and metal fabrication procedures, and, unless otherwise noted, was used in making all the test samples and other materials in this report.

Stress-rupture tests on the various alloys produced were run on 0.050 inch diameter wire in air at 1450°C. Similar testing has been reported

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TABLE I

CERTIFICATE OF SPECTROGRAPHIC ANALYSIS No. 6609

Sample Identification: Platinum Powder, Lot #190-A

Requested by: NASA

Report No: A 49080

Analysis: Quantitative

p.p.m.		p.p.m.	
Rh	24	Ca	<1
Pd	<5	Al	1
Ir	ND	Ni	ND
Ru	ND	Cr	ND
Os	ND	Mn	<1
Au	16	Sb	ND
Ag	ND	B	ND
Pb	ND	Co	ND
Sn	ND	As	ND
Zn	ND	Bi	ND
Fe	<3	Cd	ND
Cu	<1	Mo	ND
Si	3	Te	ND
Mg	<1	Na	6

*Pt 99.994%

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By: A. J. Lincoln

A. J. Lincoln
Head, Instrumental Analysis Laboratory

AJL/rc

Date: June 29, 1971

* - BY DIFFERENCE < - LESS THAN > - GREATER THAN ND - NOT DETECTED
ALL VALUES REPORTED IN PARTS PER MILLION UNLESS OTHERWISE NOTED.

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for wrought platinum base alloys using the same technique and comparison results from these tests are given. Table II is a summary of the stress-rupture tests on platinum-thoria alloys. Note that when a test ran for over 1500 hours without a break, the test was terminated.

The alloy ultimately selected for further study was platinum-0.6% thoria. This alloy was workable without difficulty using the standard working schedule. Furthermore, it has excellent stress rupture properties. Alloys with higher thoria content could be extruded and have similar stress rupture properties, but would require substantially higher manufacturing costs, particularly in the form of tubing.

Figure 1 is a longitudinal section of the platinum-0.6% thoria alloy test sample which had been on test 1610 hours at 1450°C with a stress of 1120 psi. This sample did not fracture. The section was taken at the center of the specimen. The structure is quite exceptional in that after 9-1/2 weeks at temperature, the wire still shows an unusually small grain size and retains the elongated structure developed in the initial processing. Platinum wire at this time and temperature would show large grains extending across the entire section.

Figure 2 is a longitudinal section of the platinum-0.6% thoria alloy test specimen which had been on test 101 hours at 1450°C with a stress of 1456 psi (platinum-40% rhodium at this temperature and stress would have a stress-to-rupture time of about 12 hours). The picture is taken at the area of rupture. Figure 3 is the same specimen about one inch from the break. The break occured through the grain boundaries as is normal for platinum base alloys. There is relatively little grain boundary separation near the break. The elongated grain structure causes a much more jagged

TABLE II

Stress-Rupture Test Time (Time to Fracture in Hours)*
1450°C, Air Atmosphere, 0.050 Inch Diameter Wire

Composition	LOAD IN GRAMS			Comments
	800 (896psi)	1000 (1120psi)	1200 (1344psi)	
Pt + 0.5%ThO_2	1752 Removed 68 Furnace out 1200 Furnace out	1162 1512 Removed	1591 Removed	No difficulty in working with standard schedule.
Pt + 0.6%ThO_2**	1607 Removed 1571 Removed	1610 Removed	134 369	No difficulty in working with standard schedule
Pt + 0.7%ThO_2		1271	280	Some difficulty in working with standard schedule
Pt + 0.8%ThO_2		875	52.3	Very difficult to work cold. Bar divided into two pieces. Bar No.1 Some cracking
Pt + 0.8% ThO_2		25 39		Bar No. 2-more cracking than in Bar No. 1
Pt + 1.0% ThO_2		21.4	7.6	Very difficult to work cold. Standard schedule used with adjusted annealing.
Pt + 1.0% ThO_2	1584 Removed	1585 Removed		Extruded at 1200°C from one inch diameter to 0.190 inch diameter. Cold swaged to 0.100 inch diameter, annealed and drawn to 0.050 inch diameter
Pt + 1.2% ThO_2		1656 Removed		Initial bar hot swaged, the cold swaged and drawn to 0.050 inch diameter.
Pt + 1.5%ThO_2		1293 Removed		Extruded at 1200°C from 0.625 inch diameter to 0.100 inch diameter. Cold drawn to 0.050 inch diameter.
Pt + 2.0%ThO_2		22 22		Same as Pt + 1.5% ThO_2

TABLE II (Cont'd)

Pt	Less than one hour	Less than one hour	Less than one hour	
Pt-10% Rh	19	6	3	For comparison the values are given from previous work on standard wrought alloys.
Pt-20% Rh	45	20	15	
Pt-40% Rh	60	28	20	
Pt-10% Ir	16	10	8	
Pt-20% Ir	27	17	14	

* The notation "removed" indicates the sample was removed from test without fracture for the time indicated.

**Additional stress-rupture tests on Pt-0.6% ThO_2 gave the following results:
1456 psi - 101 hrs., 1568 psi. - 40.3 hrs., 1680 psi. - 8.0 hrs., 1792 psi. - 3.8 hrs.

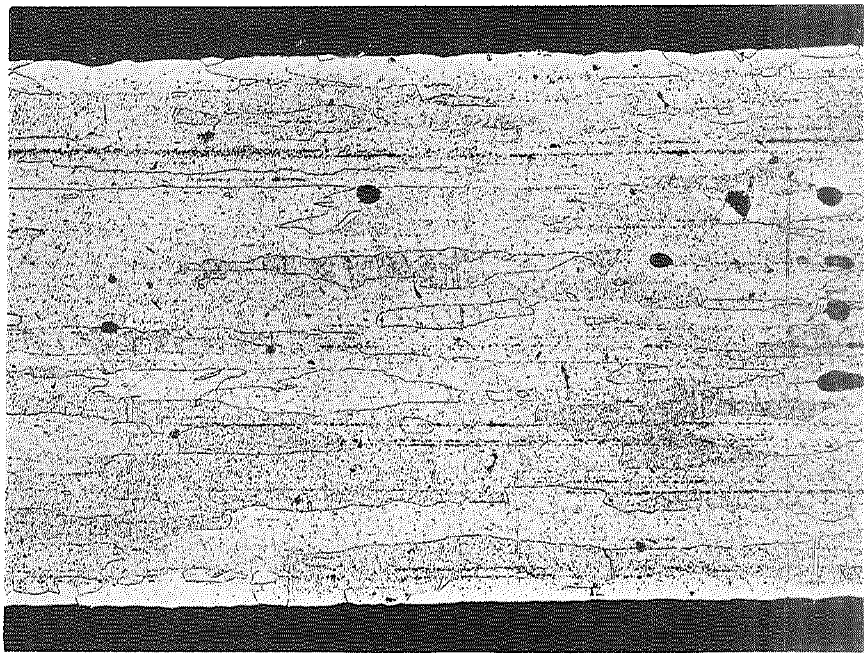


Figure 1. Film No. 8963 Mag. 100X

Longitudinal section of a platinum-0.6% thoria alloy, 0.050 inch diameter wire, on test for 1610 hours at 1450°C in air with a stress of 1120 psi.

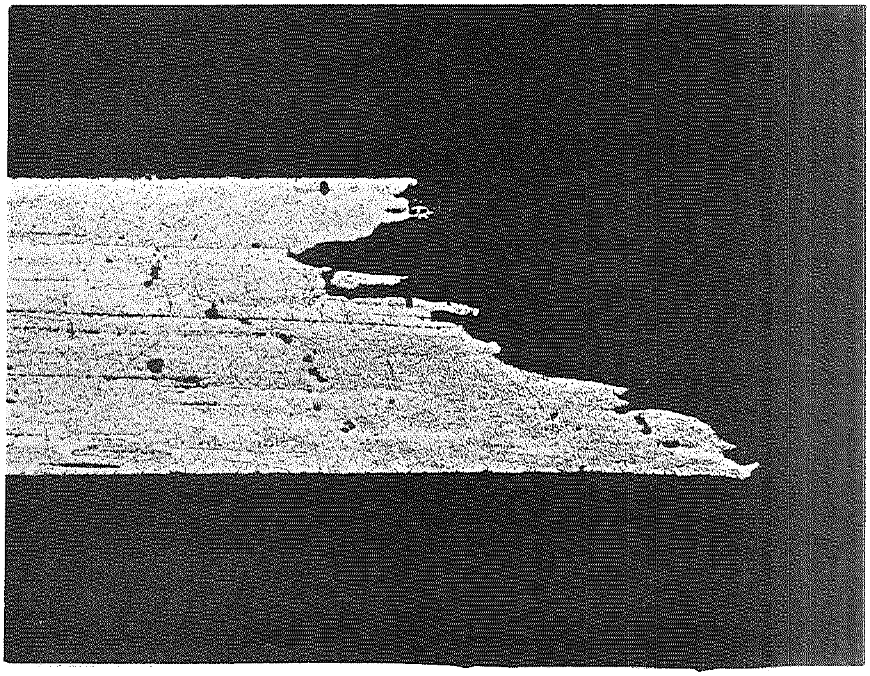


Figure 2. Film No. 8937 Mag. 50X

Longitudinal section of the fracture area of platinum-0.6% thoria alloy, 0.050 inch diameter wire, on test for 101 hours at 1450°C in air with a stress of 1456 psi.

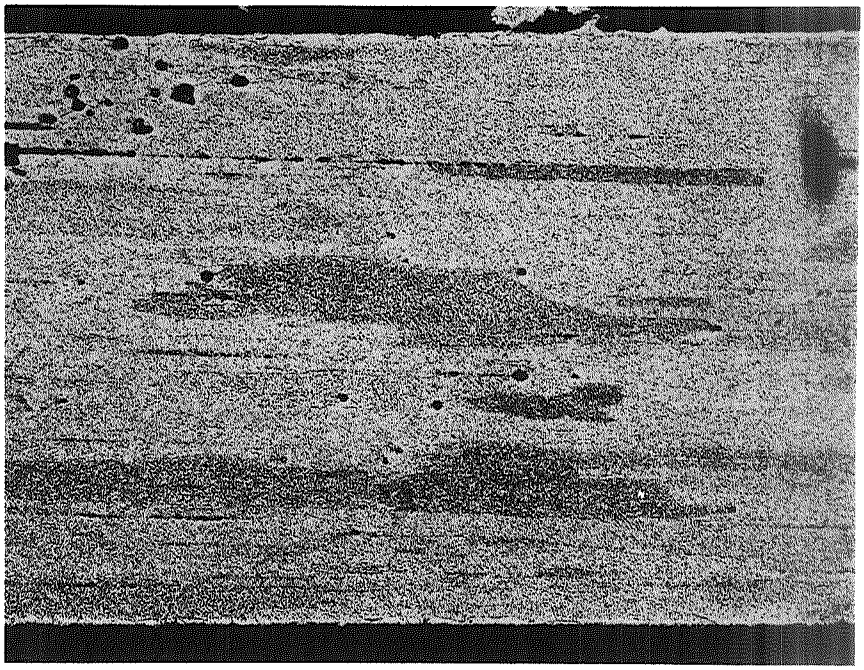


Figure 3. Film No. 8936 Mag. 100X

Same sample as in Fig. 2, about one inch from the fracture area.

and elongated break than is normal for wrought material. This type of "fibrous" break is characteristic of the thoriated material exhibiting long, stress-rupture lives. In wrought materials, the recrystallized grains are equiaxed in nature and result in a more "rounded" appearance at the break with perhaps the appearance of more ductility.

2. Mechanical Properties of Platinum-Thoria Alloys at Ambient Temperatures

Along with the high temperature stress-rupture properties, ambient temperature properties of the platinum thoria alloys were measured. These are necessary for manufacturing-metal working information and in the usage of these alloys at normal temperatures. In all cases, tests were performed on 0.020 inch diameter wire which had been reduced 75% in area from the last anneal.

Samples of wire were annealed in air for 15 minutes starting at 300°C and increasing at 100°C intervals. Tensile tests were performed at room temperature, giving values for ultimate tensile strength (psi) and elongation (percent in two inches). The instrument used for the testing was an Instron Table Model Testing Machine.

Table III summarizes the test results. Similar values for wrought platinum, platinum-rhodium and platinum-iridium alloys are included for comparison.

When it was decided to use platinum-0.6% thoria as the alloy from which tubing would be made, further tensile tests were run to determine for this alloy the yield strength (departure from straight line behavior on the tensile machine chart) and the value of the stress at 0.2% elongation offset. The values turned out to be 10,800 psi and 13,400 psi respectively. Values for pure platinum are similar.

TABLE III

Mechanical Properties of Platinum, Platinum-Thoria, Platinum-Rhodium, and Platinum-Iridium
Final Reduction 75%-0.020 Inch Diameter Wire
Samples Annealed 15 Minutes at Temperature
(Ultimate Tensile Strength-U.T.S.-psi; Elongation-% in Two Inches)

Temp. °C	Melted Pt.		Pt + 0.5%ThO_2		Pt + 0.6%ThO_2		Pt + 0.7%ThO_2		Extruded Pt + 1.0% ThO_2		Hot Worked Pt + 1.2% ThO_2	
	U.T.S.	Elong.	U.T.S.	Elong.	U.T.S.	Elong.	U.T.S.	Elong.	U.T.S.	Elong.	U.T.S.	Elong.
Hard Worked	44,000	1.0	52,000	1.0	56,500	1.0	57,000	1.0	60,000	1.0	59,500	1.0
300	42,000	1.0	50,000	1.0	55,000	1.0	57,000	1.0	59,000	1.0	58,000	1.0
400	44,000	1.2	49,000	1.0	54,000	1.0	56,000	1.0	57,000	1.0	56,000	1.0
500	43,200	1.4	49,000	1.0	52,000	1.0	54,000	1.0	57,000	1.0	56,000	1.0
600	21,800	3.9	47,000	1.5	49,000	1.2	51,000	2.0	56,000	1.0	48,000	1.0
700	17,700	21.8	41,000	2.0	38,000	2.3	38,000	2.0	53,000	1.5	32,000	5.0
800	17,300	25.3	35,000	3.5	31,000	6.0	31,000	4.0	46,000	2.0	31,000	9.0
900	17,300	26.5	31,000	4.0	30,000	9.0	31,000	7.0	38,000	3.0	31,000	10.0
1000	17,900	28.0	30,000	6.0	30,000	9.0	30,000	9.0	35,000	6.0	31,000	10.0
1100	17,600	23.0	28,000	8.0	30,000	10.0	30,000	11.0	35,000	7.0	31,000	9.0
1200	16,700	17.5	28,000	9.0	30,000	11.0	30,000	11.0	34,000	8.0	31,000	9.0
1300	15,400	9.5	28,000	9.0	30,000	10.0	30,000	11.0	34,000	8.0	31,000	9.0
1400	15,100	9.4	28,000	10.0	30,000	9.0	30,000	11.0	34,000	9.0	31,000	9.0

TABLE III (Continued)

Temp. °C	Pt + 10% Rh		Pt + 20% Rh		Pt + 40% Rh		Pt + 10% Ir		Pt + 20% Ir		Pt + 30% Ir	
	U.T.S.	Elong.	U.T.S.	Elong.	U.T.S.	Elong.	U.T.S.	Elong.	U.T.S.	Elong.	U.T.S.	Elong.
Hard Worked	83,590	--	113,000	--	175,500	--	92,300	2.0	131,500	3.0	189,700	2.0
300	98,000	2.2	129,300	2.2	201,300	3.0	100,200	5.0	134,600	3.0	187,700	2.0
400	91,000	5.0	127,300	2.5	200,800	3.0	95,500	8.0	138,600	3.0	188,300	2.5
500	82,800	7.5	120,900	2.5	193,700	4.2	89,100	10.0	131,500	4.7	188,000	2.5
600	77,000	8.5	99,500	6.0	165,600	6.0	88,000	11.2	128,400	12.5	187,700	6.2
700	74,800	8.7	95,000	7.7	145,600	10.2	87,000	12.0	129,100	12.5	200,500	10.5
800	65,000	8.7	92,000	9.0	124,100	12.2	81,200	13.0	125,600	12.5	203,700	11.0
900	43,700	35.0	60,000	36.0	84,300	49.5	73,200	14.7	120,200	13.3	184,000	11.5
1000	42,900	35.0	59,800	38.7	82,800	52.0	55,600	29.2	113,900	13.3	165,500	14.0
1100	42,000	35.5	59,000	38.5	81,200	54.0	52,500	32.5	105,100	15.8	143,200	19.0
1200	42,000	36.7	58,000	37.0	79,500	52.5	50,100	32.0	92,700	22.2	136,800	21.5
1300	41,000	33.0	57,000	38.0	77,200	51.5	48,500	29.0	89,000	18.5	131,000	17.0
1400	39,000	29.0	55,000	38.0	75,600	44.0	46,100	27.2	86,000	16.0	117,700	12.2

Further testing was also done on platinum-0.6% thoria to determine the hot tensile strength of this alloy. In this test, the wire was 0.050 inches diameter and is heated to the test temperature. The details of the testing method and a summary of the results including comparison values for platinum are given in Table IV.

3. Resistivity vs. Temperature for Platinum-0.6% Thoria

The resistivity of platinum-0.6% thoria was measured on 0.010 inch diameter wire. The wire was bifilar wound on an alumina cross and inserted in an alumina, closed-end tube using platinum lead wires from the alloy to junction head. The wires were annealed for one hour at 1400°C prior to test and the tests were made at static temperatures, i.e., no change of resistance at temperature on the measuring bridge out to the fourth decimal place. The furnace is a special calibration furnace heavily lagged and controlled for temperature stability.

The temperature was measured using an NBS calibrated platinum vs platinum-10% rhodium thermocouple and a Honeywell Rubicon B Potentiometer. The resistance was measured on a Leeds and Northrup Type ER Thermometer Bridge.

The measured values for resistivity vs. temperature are given in Table V and these values plotted in Figure 4. The resistivity measured on pure platinum under the same test conditions was very close to that of platinum-0.6% thoria. At the highest temperature (1450°C), the point of maximum difference, the resistivity of platinum was 336 ohms/circ. mil foot and that of platinum-0.6% thoria was 342 ohms/circ. mil foot, a difference of less than 2%.

Another possible use for the platinum-thoria alloy in the resistojet project is for electrical heater coils. A piece of platinum-0.6% thoria wire,

TABLE IV

Hot Tensile Tests in Air on Platinum - 0.6% Thoria With Comparison Tests for Pure Platinum

Temperature (°C)	Ultimate Tensile Strength (PSI)		Elongation (% in 2 inches)	
	Pt + 0.6% ThO_2	Pt	Pt + 0.6% ThO_2	Pt
Ambient	50,000	44,000	--	1
300	43,000		3	
400	38,200		3.25	
500	24,000	23,500	5	4
600	13,300	9,700	52	28
700	10,700	8,700	52	25
800	9,700	8,200	45	30
900	8,175	6,600	40.5	25
1000	6,650	5,600	38	29
1100	5,200	4,100	30.5	32
1200	4,600	3,100	24.5	31
1300	4,080	2,300	20.5	31
1400	3,680	1,800	17.5	30

Apparatus and Accuracy. A modified Dillon Universal tensile tester was adapted to accept a horizontal tensile specimen. The modified tester used a horizontal furnace having a uniform heat zone of 5 inches; this extended heat zone, in turn, accommodated the large elongation of platinum and its alloys at the higher temperatures.

The accuracy of the modified Dillon tensile tester was checked against a Baldwin-Southwark tensile tester and an Instron tensile tester; in either instance an error of less than 3% was determined.

Test Specimens. The standard tension test specimens used in these studies were 22 inches in length, 0.050 inches in diameter-the wire having been drawn without annealing from a diameter of 0.100 inches to one of 0.050 inches, with a last anneal at 0.100 inches.

In order to measure the elongation, the wire was marked off with a concentrated solution of ferric chloride which on heating produces a black distinguishable mark. At extreme elevated temperatures where elongation can exceed 40%, the center of the spread mark was taken as the gage point. The ferric chloride band was kept to a minimum by exercising extreme care during the marking of the specimens.

Experimental. The rate of tensile pull was 0.5 inches per minute. The sample was heated for five minutes prior to starting the tensile pull. Prior tests have shown negligible variations of tensile strength when specimens were heated for intervals ranging between 5 and 15 minutes.

TABLE V

Resistivity Test
Alloy 9R 763 (Pt + 0.6% ThO_2)

Temperature Measurement - NBS Calibrated Pt vs Pt 10 Rh Standard Thermocouple
With Honeywell Rubicon B Potentiometer
Resistance Values - Determined on an L & N Bridge - Type ER

Temperature °C	Resistivity ohms/mil. ft.
25	68
173	103
211	112
337	141
395	154
489	173
674	211
731	222
844	244
974	266
1109	290
1220	308
1420	337

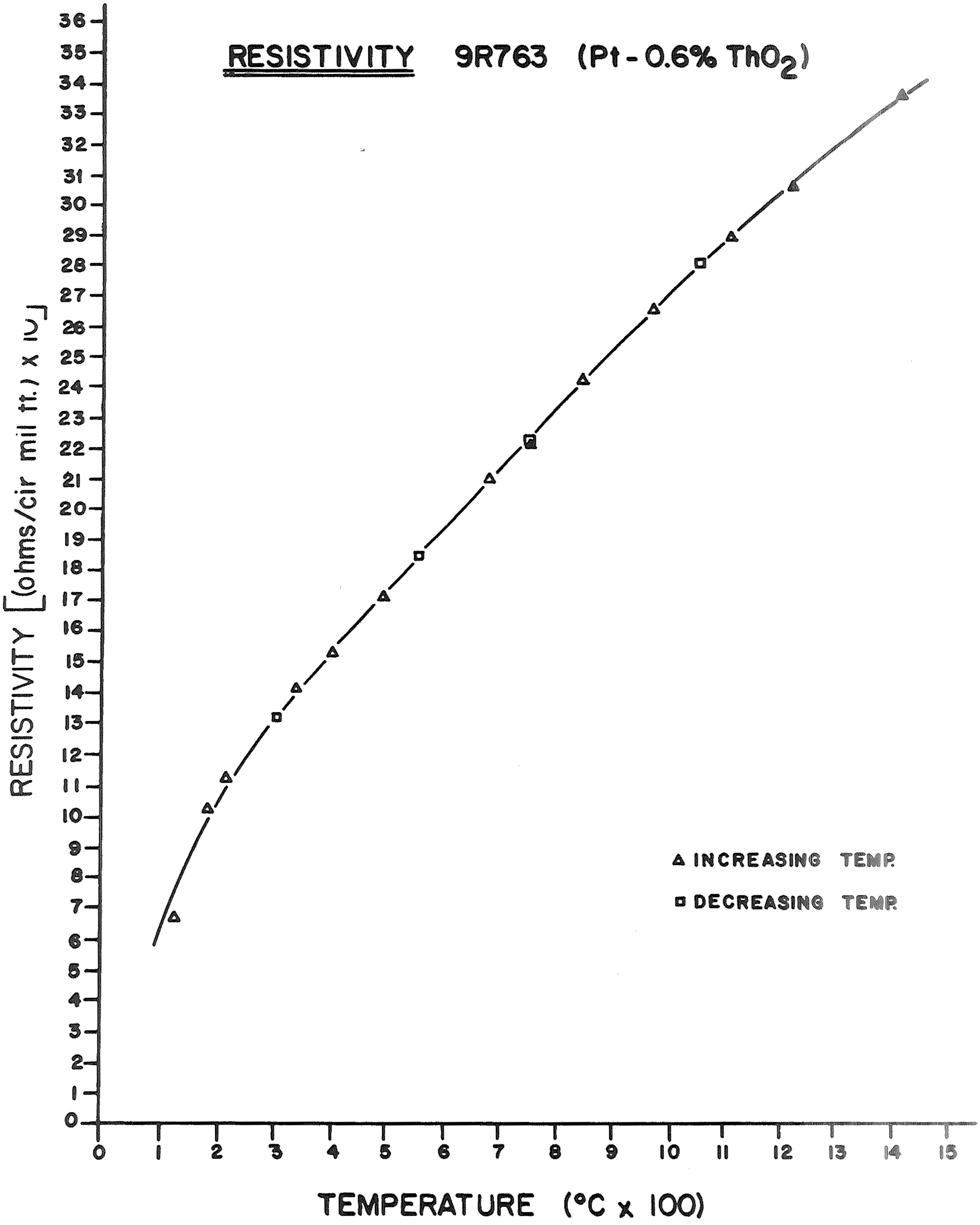


Figure 4.

Electrical resistivity of a platinum-0.6% thoria alloy as a function of temperature.

0.005 inches diameter and 20 feet long was sent for the manufacture of test coils on 3 March 1971, to Mr. Bob Murch, TRW Systems Group, Inc., 1 Space Park, Redondo Beach, California 90278.

4. Tubing Manufacture

Seamless tubing was prepared of platinum-0.6% thoria. In the laboratory a small sheet bar was prepared using the standard technique. This bar was rolled and cross-rolled with intermediate anneals at 1000°C to a flat sheet approximately five inches square by 0.065 inches thick.

This sheet was then transferred to the Engelhard Industries Division, Mill Products Department tube room and all further operations were performed in that area. The first operation was to punch out a 4-3/4 inch diameter round blank from the sheet. This blank was then cupped through a series of punch operations with two intermediate anneals at 1000°C to form a shell one inch in diameter with an 0.033 inch wall thickness.

The shell was then swaged and drawn with intermediate annealing to the final size of 0.054 inches diameter with a wall thickness of 0.007 inches. No difficulty was encountered in fabricating this tubing. Its working qualities were much like that of platinum. A piece of this tubing, in the cold worked state, 19-1/2 inches long, was sent for testing on 11 May 1971, to Carl Halbach of the Marquardt Company, 16555 Saticoy Street, Van Nuys, California 91409.

Cross sections and longitudinal sections of the tubing as-worked and annealed for 15 minutes at 1000°C are shown in Figures 5 to 8.

5. Joining Methods

Platinum-thoria alloys are produced by powder metallurgy and depend for their properties on a proper dispersion of the thoria. If the alloy is melted, as in simple fusion welding by arc or torch, the thoria

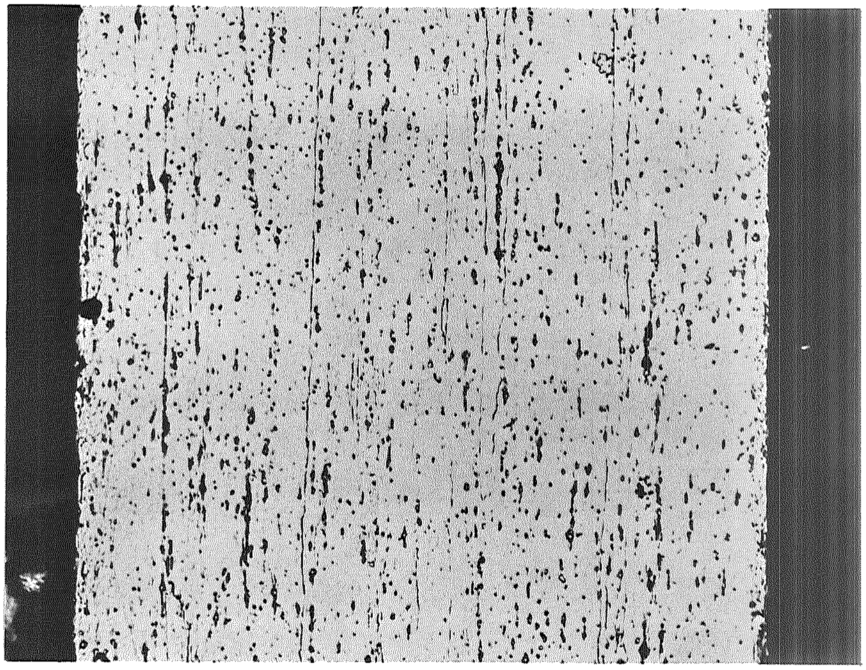


Figure 5. Film No. 8934 Mag. 500X

Platinum-0.6% thoria tubing, cold worked, longitudinal section

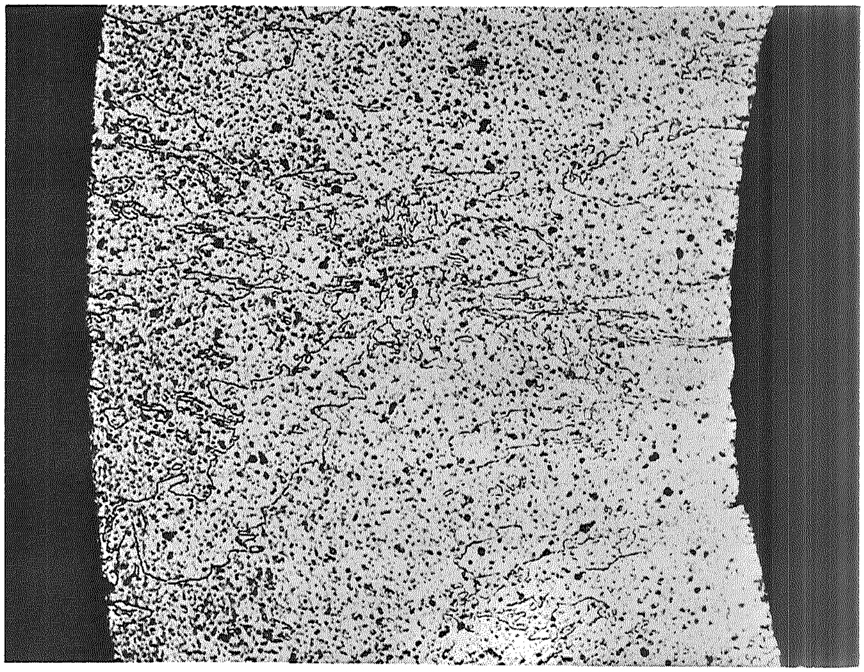


Figure 6. Film No. 8935 500X

Platinum-0.6% thoria tubing, cold worked, cross section.

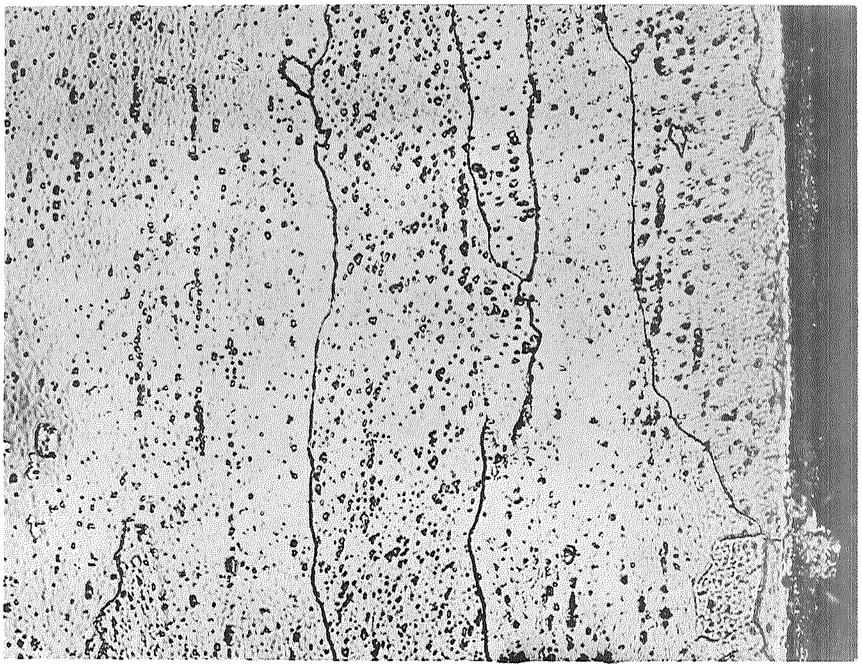


Figure 7.   Film No. 8962   Mag. 500X

Platinum-0.6% thoria tubing, annealed, longitudinal section.

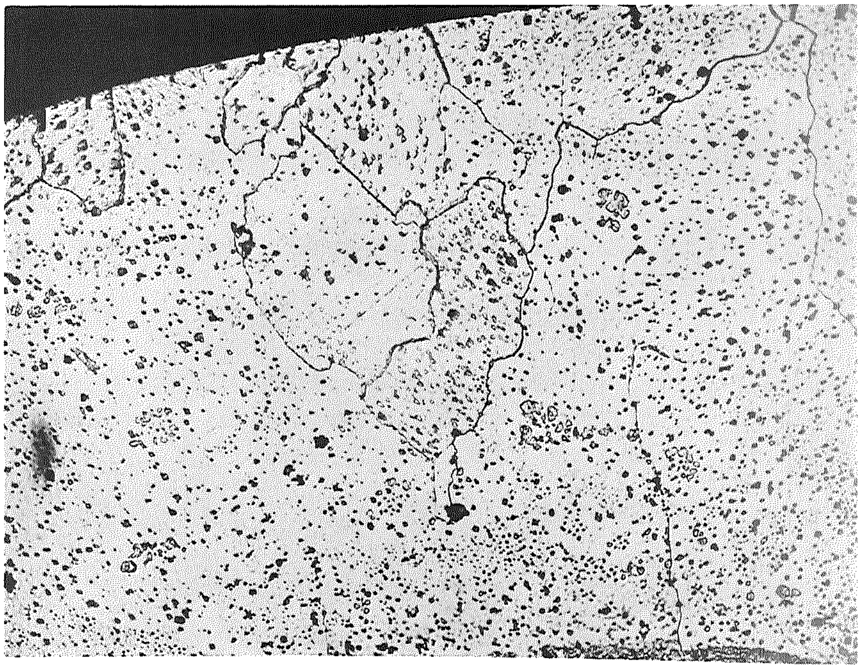


Figure 8.   Film No. 8961   Mag. 500X

Platinum-0.6% thoria tubing, annealed, cross section.

dispersion is destroyed. In addition, the thoria may also cause a defective joint on melting by collecting at the juncture of the weld and parent metal. It has been suggested that a small fusion weld in which the heat for welding was produced rapidly, and quickly dissipated, could possibly avoid some of the detrimental aspects of fusion welding. For this reason it was decided to try electron-beam welding as a joining technique. Electron-beam welding has the virtue of being able to produce small fusion zones, precisely controlled, with very localized heat input resulting in a very small heat-affected zone. Both butt welds and lap welds were produced by electron-beam welding.

A method of joining thoriated platinum without melting is obviously very desirable. Three methods chosen for investigation were dap welding, diffusion bonding and spot welding. Dap welding is a form of forge welding. Since platinum does not form an appreciable oxide film during heating, it is possible to join platinum by heating in air to about 800°C and hammering the area to be welded. Such a process, done by a skilled operator, will produce a sound weld in pure platinum and many platinum alloys.

Diffusion bonding is similar to forge welding in that a combination of heat and pressure is used to make the weld. Figure 9 shows the fixture used. The fixture consists of stainless steel flanges connected by molybdenum bolts fitted with Inconel nuts. The sheet to be welded was placed between Inconel slugs and inserted between the flanges. The nuts were tightened to complete the assembly. The full assembly was then heated in vacuum to 1100°C, for two hours. The thermal expansion of the assembly produced the necessary compressive force to bond the strips by interdiffusion at the weld interface.

The spot welding was done in a conventional spot welder. The weld was produced by a line of spot welds across the strips. Both a single line and a double line were used. The time-temperature cycle of the spot welder was adjusted to produce no melting at the spot welded interfaces.

Brazing is also a feasible process for joining. Obviously, the braze material must melt and wet the material to be joined. However, although the joint cannot operate too near or above the brazing material's melting point, a properly designed brazed joint can operate at high temperatures and does not degrade the parent metal by melting it.

To demonstrate the feasibility of brazing platinum-thoria alloys, a platinum-20% gold alloy (melting point about 1400°C) was chosen. Heating was done both by torch and by induction heating. A higher melting point alloy could be used if good temperature control is used to avoid melting the parent metal (The melting point of platinum is 1767.6°C).

In all cases the samples for joining tests were 0.020 inch thick sheet, 1/2 inch wide. After joining, the central section of the strip was machined to a 1/4 inch width in a standard tensile specimen shape and annealed at 1100°C. The samples were then pulled in a tensile machine and the ultimate tensile strength and the elongation recorded. Table VI summarizes the test results. Figures 10 and 11 show some of the samples pulled. Figures 12 through 17 are representative sections of the test samples showing the joint areas.

The electron-beam welded samples showed some porosity at the weld. This has been noted previously in similar welds of this type material. This might be due to (1) coagulation of minor porosity always present in powder metallurgy material, (2) collection of residual gas in the metal, or

TABLE VI

SUMMARY OF TENSILE TESTS ON WELDED AND BRAZED TEST SPECIMENS OF PLATINUM-0.6% THORIA SHEET

Specimen Type of Weld	Tensile Strength PSI	% Elongation
Blank (solid sample, no weld)	27,000	
	"	43
"	"	37
Butt Weld- Electron Beam	27,000	31
	28,000	31 (Broke at weld)
	28,000	34
Lap Weld- Electron Beam	27,000	24
	26,000	23
	22,000	7 (Broke at weld)
Dap Weld	28,000	24
	28,000	25
High Temp. Braze Pt-20%Au Braze Alloy	30,000	14
	30,000	14
Single Line Spot Weld	30,000	19
	27,500	8 (Broke at weld)
Double Line Spot Weld	30,000	15
	29,000	15
Diffusion Bond	28,000	25
	27,000	25

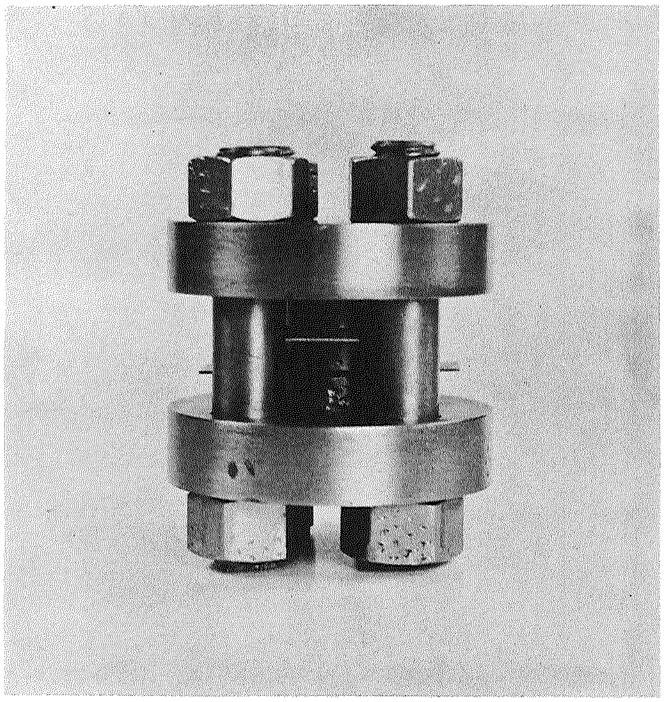


Figure 9. Film No. X 894

Diffusion bonding fixture.

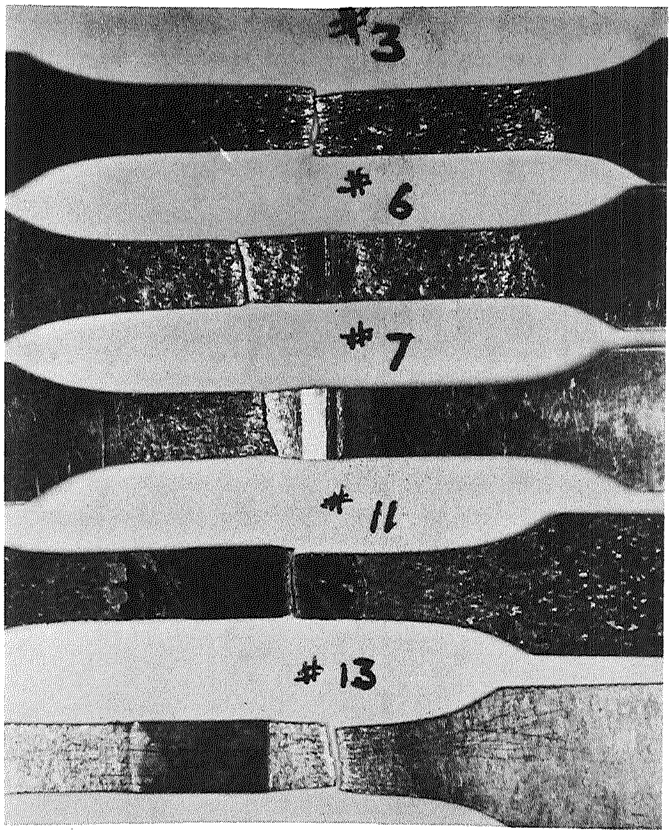


Figure 10. Film No. 8933 Mag. 1.5X

Test samples after tensile testing; Coding refers to samples numbers. Sample No. 3 is the blank.

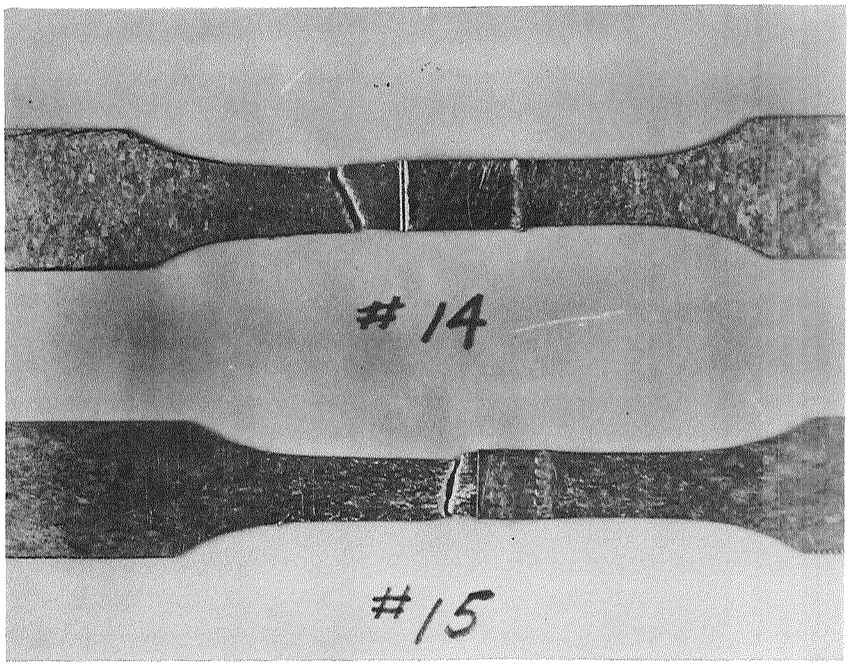


Figure 11. Film No. 8939 Mag. 1.5X

Test samples after tensile testing; coding refers to sample numbers.

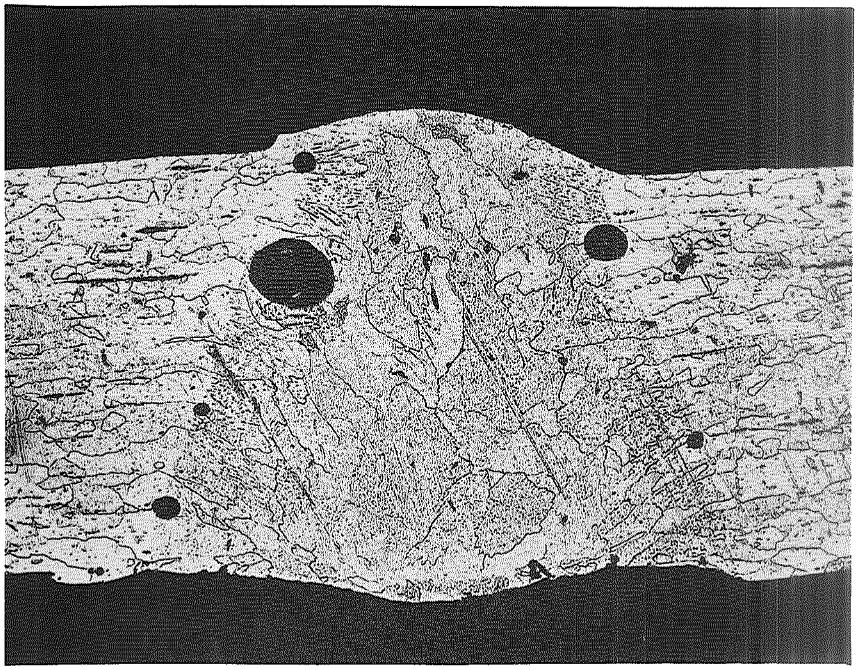


Figure 12. Film No. 8930 Mag. 100X

Electron beam butt weld. Sample No. 6.

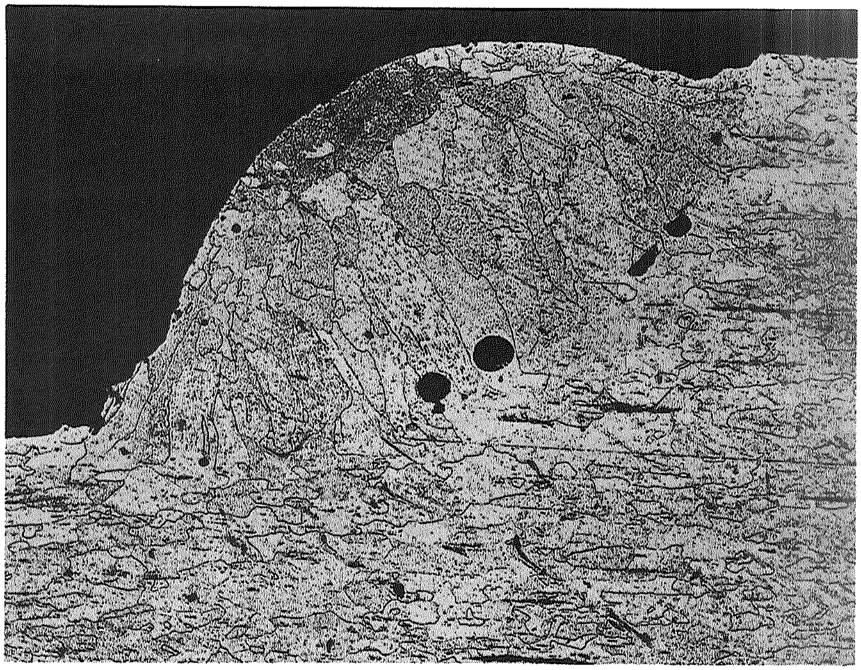


Figure 13.   Film No. 8931   Mag. 100X

Electron beam lap weld. Sample No. 7.

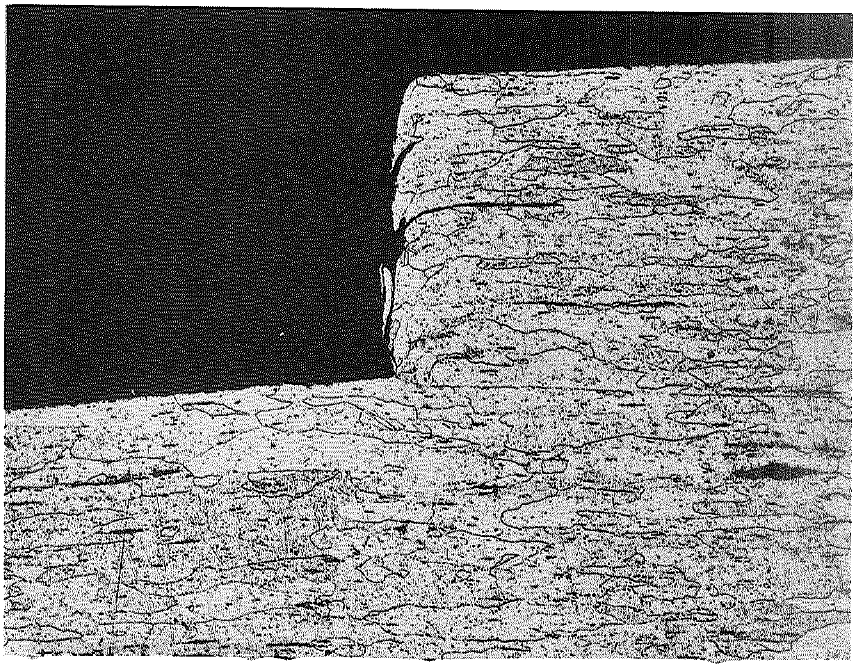


Figure 14.   Film No. 8932   Mag. 100X

Dap weld (forge weld) showing one end of the lap. Sample No. 13.

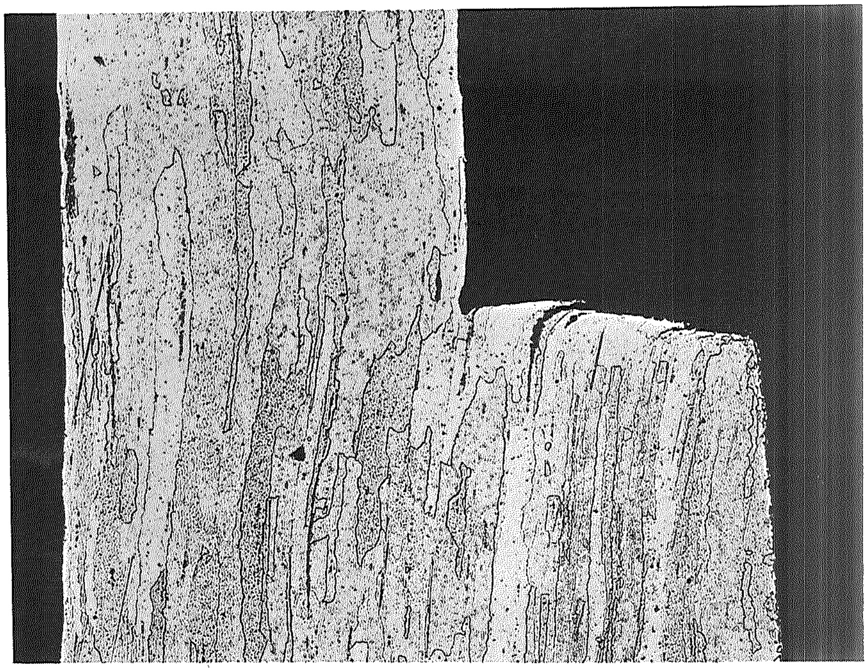


Figure 15. Film No. 8927 Mag. 100X

Diffusion weld showing one end of the lap.
Sample No. 11

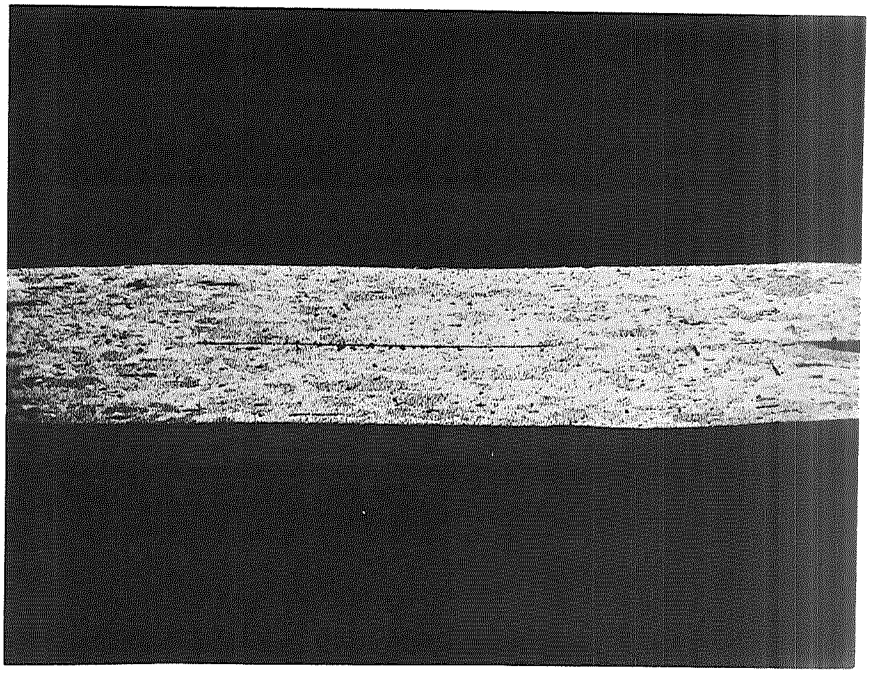


Figure 16. Film No. 8941 Mag. 25X

Double line spot weld showing welds at both ends of lap. Sample No. 15.

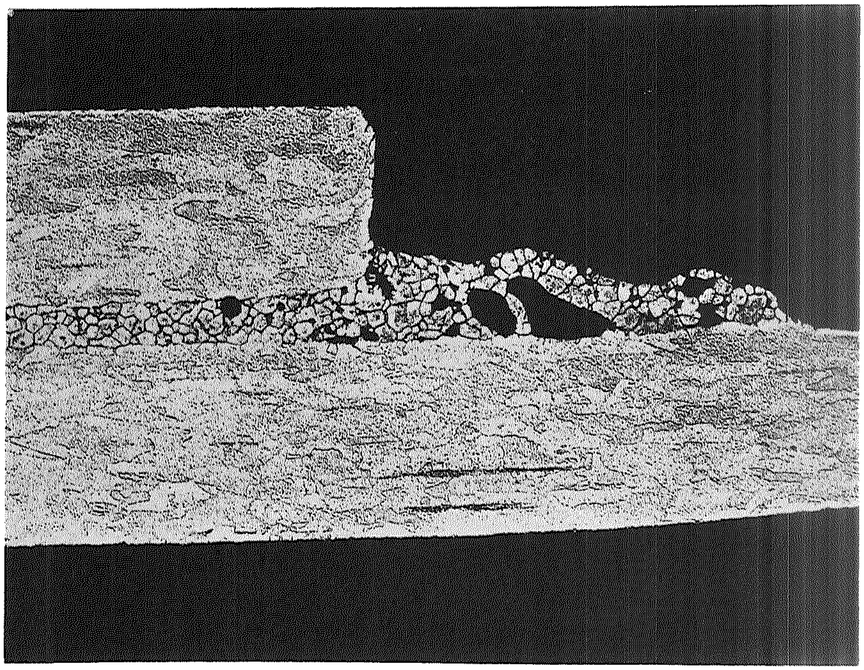


Figure 17. Film No. 8940 Mag. 50X

High temperature brazed joint using Pt-20% Au brazing alloy. Sample No. 14 shows one end of the lap.

(3) decomposition of the thoria in the intense electron beam. Despite the porosity, the welds had good tensile strength. Still, this is a fusion weld, and the cross section shows that the structure of the weld is completely different from the parent metal. Although the ambient temperature tensile properties are adequate, the change in structure due to the fusion weld would probably mean the loss of the exceptional high temperature stress-rupture properties at the weld. This would, of course, not be a problem if the weld were outside the hot zone in a fabricated structure.

The dap welded, diffusion bonded and spot welded cross sections show no molten zone. This is obviously the best type of structure for such material since the thoria dispersion remains intact and active in reducing high temperature creep.

The brazed structure shows no melting of the platinum-thoria alloy and good wetting in the joint. The Pt-20% Au alloy melts at about 1400°C. This limits the temperature of operation of such a system although with accurate temperature control, less gold could be used, raising the temperature of melting. Furthermore, the diffusion of gold from the braze alloy into the platinum would also raise the braze melt temperature and perhaps increase the strength of the parent metal.

CONCLUSIONS AND RECOMMENDATIONS

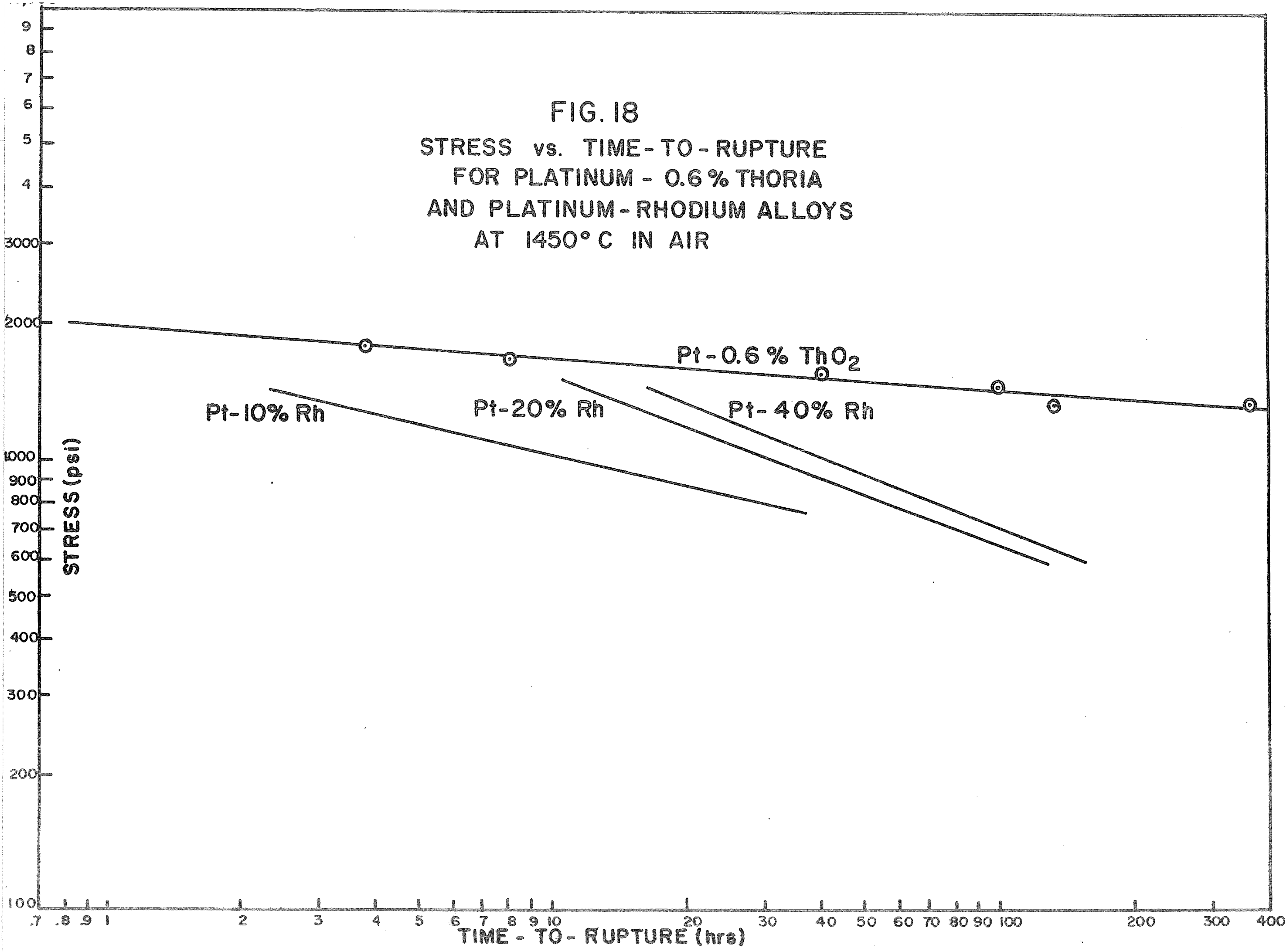
The tests reported above show that there is relatively little effect of 0.6% thoria on the room temperature mechanical and electrical properties of platinum. For all practical manufacturing purposes the thoriated material behaves as does commercially pure platinum. The advantage of adding thoria shows up in the high-temperature, long term properties. The stress-rupture tests reported here show that in this type of testing, the platinum -.6% thoria alloy has a much greater life than platinum-40% rhodium. In earlier testing the platinum-40% rhodium alloy had been found to be the best commercial alloy in stress-rupture tests at 1450°C. Figure 18 is a plot of stress vs. time-to-rupture for platinum-0.6% thoria and some platinum-rhodium alloys, at 1450°C, the data taken from Table II. The superiority of platinum-0.6% thoria in this type of testing is clearly evident in this plot. Since platinum-iridium alloys are more readily oxidized, they would show up as inferior to both the platinum-0.6% thoria and platinum-rhodium alloys.

The metallographic cross sections of platinum-0.6% thoria on test for extended periods at 1450°C show an elongated grain structure. This elongated structure is quite different from platinum or platinum-rhodium wrought alloys held at high temperatures for extended periods. In the latter case the recrystallized grains would be equiaxed, and as time progressed the grain growth would result in very large grains.

The observation in dispersion hardened materials of relatively unaffected room temperature properties and enhanced high temperature properties has been reported for other materials such as nickel and copper. While the theoretical basis for improved high temperature properties has not yet

FIG. 18

STRESS vs. TIME-TO-RUPTURE FOR PLATINUM - 0.6% THORIA AND PLATINUM-RHODIUM ALLOYS AT 1450° C IN AIR



been firmly estblished, the most recent publications tend to focus on the particles in the grain boundaries, their interactions with dislocations and their modification of the grain boundaries as sources and sinks for vacancies. The action of the grain boundaries is obviously crucial in any theoretical development since this is the principal difference in cross sections of wrought versus dispersion strengthened materials. Particularly so since it is observed that the fracture in the stress rupture testing invariably follows the grain boundaries. On a phenomenological basis, it can be observed that since the fracture follows the grain boundaries, a stable, elongated, fine grain structure presenting a minimum of grain boundary perpendicular to the applied stress would be the most desirable. This, of course, describes the platinum-0.6% thoria alloy, and while not a fundamental explanation of the high temperature properties, it does help to rationalize the improvement in these properties.

The joining tests demonstrate that the platinum-0.6% thoria alloy can be reliably fabricated by a number of techniques other than simple fusion welding. The particular method to be employed in a given situation will depend on the structure to be fabricated. However, when the alloys are properly joined, the weld will be as strong as the parent metal. Brazing is feasible with platinum-gold alloys, the choice of alloy being governed by the operating temperature required and the degree of temperature control which can be exercised in the brazing operation.

It has been suggested that the thoriated platinum alloys may not be stiff enough at room temperature to withstand the shock forces acting on the resistojet. Platinum-iridium alloys do have the necessary stiffness but may not be sufficiently oxidation resistant. One method for overcoming this difficulty would be to make a three-layer tube, the inner and outer layers being platinum-0.6% thoria, and the central layer being platinum-30% iridium. Such material has been produced in other alloys, and seems feasible in this combination. It would be useful to run tests on such a system to determine the diffusion rate of iridium from the central layer to the inner and outer layers.