

U.S. DEPARTMENT OF COMMERCE
National Technical Information Service

AD-A025 500

HIGH TEMPERATURE THERMOCOUPLE SYSTEM
FOR ADVANCED AIRCRAFT TURBINE ENGINES

ENGELHARD MINERALS AND CHEMICALS CORPORATION

PREPARED FOR
AIR FORCE AERO PROPULSION LABORATORY

31 DECEMBER 1975

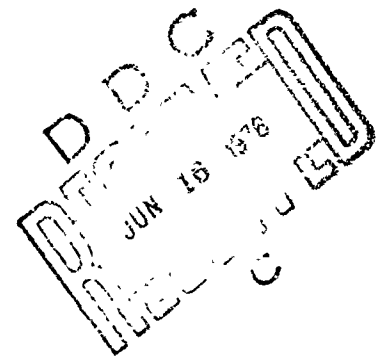
169078

AFAPL-TR-75-108

AD A 025 500

HIGH TEMPERATURE THERMOCOUPLE SYSTEM FOR ADVANCED AIRCRAFT TURBINE ENGINES

ENGELHARD MINERALS & CHEMICALS CORPORATION
ENGELHARD INDUSTRIES DIVISION
RESEARCH & DEVELOPMENT DEPARTMENT
MENLO PARK, EDISON, N.J.



DECEMBER 1975

FINAL REPORT FOR PERIOD 1 AUGUST 1974 - 31 DECEMBER 1975

Approved for public release; distribution unlimited

REPRODUCED BY
NATIONAL TECHNICAL
INFORMATION SERVICE
U.S. DEPARTMENT OF COMMERCE
SPRINGFIELD, VA. 22161

AIR FORCE AERO-PROPULSION LABORATORY
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio 45433

NOTICE

When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

This final report was submitted by Engelhard Minerals & Chemicals Corporation, under Contract F33615-74-C-2069. The effort was sponsored by the Air Force Aero-Propulsion Laboratory, Air Force Systems Command, Wright-Patterson AFB, Ohio under Project 3066, Task 306603, and Work Unit 30660368 with Charles A. Skira, AFAPL/TBC, as Project Engineer. D. A. Toenshoff and E. D. Zysk of Engelhard Minerals & Chemicals Corporation were technically responsible for the work.

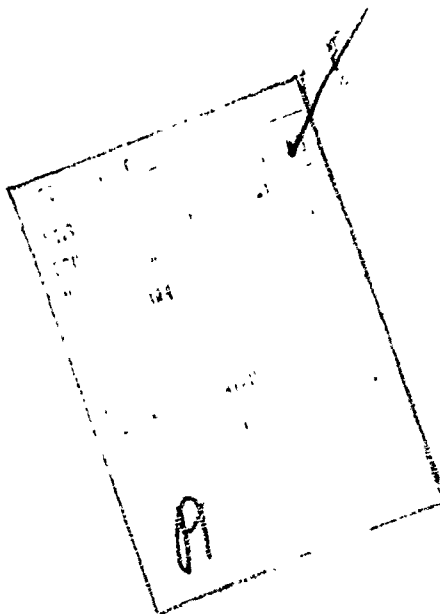
This report has been reviewed by the Information Office, ASD/OIP, and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nations.

This technical report has been reviewed and is approved for publication.

Charles A. Skira
CHARLES A. SKIRA, GS-11
Project Engineer

FOR THE COMMANDER

Charles E. Bentz
CHARLES E. BENTZ
Tech Area Manager, Controls



Copies of this report should not be returned unless return is required by security considerations, contractual obligations, or notice on a specific document.

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER AFAPL-TR-75-108	2. GOVT ACCESSION NO	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) High Temperature Thermocouple System		5. TYPE OF REPORT & PERIOD COVERED Technical Report (Final) 1 Aug. '74 - 31 Dec. '75
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) D. A. Toenshoff E. D. Zysk		8. CONTRACT OR GRANT NUMBER(s) F-33615-74-C-0069
9. PERFORMING ORGANIZATION NAME AND ADDRESS Engelhard Minerals & Chemicals Corp. Engelhard Industries Division Menlo Park, Edison, N. J. 08817		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 3066 306603 30660368
11. CONTROLLING OFFICE NAME AND ADDRESS Air Force Aero-Propulsion Lab (TBC) Wright Patterson AFB, Ohio 45433		12. REPORT DATE December 31, 1975
		13. NUMBER OF PAGES 104
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		15. SECURITY CLASS. (of this report) UNCLASSIFIED
		15a. DECLASSIFICATION/DOWNGRADING SCHEME
16. DISTRIBUTION STATEMENT (of this Report) Approved for Public Release; Distribution Unlimited		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Gas Turbine Engine Controls Gas Turbine Engine Temperature Sensor High Temperature Thermocouple System		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) This is a final report covering the development of a temperature measuring system for the very high temperature gas streams found in advanced jet engines. The measurement system is a thermocouple probe assembly suitable for interfacing to the Detroit Diesel-Allison GMA 200 Joint Technology Demonstration Engine. Average temperatures of the order of 2600-2700°F (1425-1480°C) will have to be measured and it is anticipated that hot spots to 3000°F (1650°C) will be encountered.		

Block 20. (continued)

This study covered the selection of an optimum thermocouple (Pt-40% Rh vs. Pt-0.6% ThO₂); compatible compensating extension lead wire (base metal) usable to around 750°C in air (Nichrome vs. EA 9R-682); high temperature resisting sheath and shield tube material (Pt-0.6% ThO₂); crushable MgO insulation; design of a temperature probe utilizing the optimum materials and vibration testing at 1450°C of one of the finished probes. The selected probe passed the high temperature vibration test successfully.

Based on the tests performed during this program the proposed probe, incorporating all of these new materials, offers interesting possibilities for the projected use. However only actual tests of this probe in an operating engine will prove this design and the new materials selected as components.

TABLE OF CONTENTS

<u>SECTION</u>	<u>TITLE</u>	<u>PAGE</u>
1.0	INTRODUCTION	1
2.0	PROGRAM OBJECTIVES AND METHODS OF ACHIEVEMENT	2
3.0	TECHNICAL CONSIDERATIONS	3
3.10	General	3
3.20	Probe Design	4
3.30	Materials	5
4.0	PROGRAM PLAN	8
5.0	DEVELOPMENT OF PROBE - PHASE I	8
5.10	Task A Candidate Material Selection	10
5.11	Thermocouple Alloys and Compensating Extension Wires	10
5.12	Sheath and Support Materials	11
5.13	Crushable MgO Insulation	11
5.20	Material Procurement or Fabrication	11
5.30	Qualifying Tests	13
5.31	Thermocouple Alloys and Compensating Extension Wires	13
5.32	Sheath and Support Material	16
5.40	Selection of Optimum Materials	16
5.50	EMF Reproducibility of Selected Thermoelements and Extension Wires	17
5.60	Proposed EMF vs. Temperature Relationship	17
6.00	DEVELOPMENT OF PROBE - PHASE II	18
6.10	Thermocouple Sensor Design	18
6.20	Fabrication of Prototype Probe	19
6.30	Vibration Testing of Prototype Probe	20

TABLE OF CONTENTS

<u>SECTION</u>	<u>TITLE</u>	<u>PAGE</u>
7.00	DISCUSSION	22
8.00	CONCLUSIONS	25
9.00	REFERENCES	26
APPENDIX A	PROBE DESIGN	63
	Design Analysis for T4.1 Thermocouple	64
	Drawing No. DDA 202000	70
APPENDIX B	VIBRATION TESTING OF FINISHED PROBE	71
	Test Specifications	72
	G.E. Exceptions	78
	Test Results	80

LIST OF ILLUSTRATIONS

<u>FIGURE</u>		<u>PAGE</u>
1	Stress-to-Rupture Strength of Pt, Pt-0.6% ThO ₂ , Pt-Rh Thoria and Pt-Rh alloys at 1450°C.	27
2	EMF Stability Test Equipment	28
3	Macrophotograph Showing Four Sides of Finished Probe	29
4	Macrophotograph Showing Top and Bottom View of Finished Probe	30
5	Longitudinal Section, Pt-0.6% ThO ₂ , As Worked	31
6	Longitudinal Section, Pt-0.6% ThO ₂ , After Exposure to 1200°C	31
7	Longitudinal Section, Pt-0.6% ThO ₂ , After Testing 2117 hours at 1450°C with a Stress of 900 psi	31
8	Longitudinal Section, Pt-40% Rh, After Testing 73 hours at 1450°C with a Stress of 900 psi	32
9	Longitudinal Section, Pt-40% Rh, Annealed Condition	32
10	Transverse Section, EA 9R-1545 (95%Pt-4%ThO ₂) Cermet, 87.55% of Theoretical Density	33
11	Transverse Section, EA 9R-1545 (95%Pt-4%ThO ₂) Cermet, 93.08% of Theoretical Density	33

LIST OF TABLES

<u>TABLE</u>		<u>PAGE</u>
1	Analysis of Platinum Powder Used In Fabricating Pt-0.6% ThO ₂ and Pt Cermet	34
2	EMF (millivolts) of Candidate Thermoelements Versus Platinum 67 As A Function of Temperature (IPTS 68)	35
3	EMF (millivolts) of Candidate Extension Wire Versus Platinum 67 As A Function of Temperature (IPTS 68)	36
4	Effect of Small Additions of Mn, Al and Si on EMF of 9R-567	37
5	Effect of Small Additions of Mn, Al, W, Si and Mo on EMF of 9R-567	38
6	Effect of Small Additions of Mn, Si and W On EMF of 9R-567	39
7	Effect of Small Additions of Mn and Si On EMF of 9R-567	40
8	Effect of Small Additions of Mn On EMF of 9R-567	41
9	Wanted EMF Vs. Pt67 For Negative Leg Compensating Extension Wire To Match Nichrome or Tophet D.	42
10	Stress-Rupture Test Results of Platinum and Platinum-Rhodium Alloys	43
11	High Temperature EMF Stability Test In Air Alloy 16444 (Pt-3% Rh)	44
12	High Temperature EMF Stability Test In Air Alloy 11791 (Pt-40% Rh)	45
13	High Temperature EMF Stability Test In Air Alloy 9R-763 (99.4% Pt - 0.6% ThO ₂)	46
14	EMF Stability Test In Air Tophet D	47
15	EMF Stability Test In Air Nichrome	48
16	EMF Stability Test In Air 9R-567	49
17	EMF Stability Test In Air 9R-567 Screening Tests (750°C - 1000°C)	50
18	Repeated EMF Stability Test In Air At 750°C Alloy 9R-567	51

LIST OF TABLES

<u>TABLE</u>		<u>PAGE</u>
19	Spectrographic Analysis of MgO Used In Finished Probe	52
20	EMF Reproducibility of Negative Leg Thermoelement (9R-763), EMF Vs. Pt67	53
21	EMF Reproducibility of Positive Leg Thermoelement (Pt - 40% Rh), EMF Vs. Pt67	54
22	EMF Reproducibility of EA 16444 (Pt-3% Rh) EMF Vs. Pt67	55
23	EMF Reproducibility of Alloy 9R-567, EMF Vs. Pt67	56
24	EMF Reproducibility of Nichrome, EMF Vs. Pt67	57
25	EMF Reproducibility of Alloy 9R-682, EMF Vs. Pt67	58
26	EMF Reproducibility of Alternate Extension Wire (Tophet C), EMF Vs. Pt67	59
27	Provisional EMF Vs. Temperature Relationship Pt-40% Rh Vs. Pt - 0.6% ThO ₂ , Target EMF of Selected Thermoelements	60
28	Target Nichrome Vs. Pt67 Values and Needed Matching Values of 9R-682 Vs. Pt67	61
29	EMF Vs. Temperature Relationships For Matched Couple and Matched Compensating Extension Wires Used in Probe	62

1.0 INTRODUCTION

The gas temperature of many gas turbine engines is determined by inserting thermocouple probes at the turbine outlet. In most cases, it is possible to extrapolate from the turbine outlet to obtain turbine inlet temperatures with sufficient accuracy to operate the gas turbine.

Advanced gas turbines, however, bring about dramatic changes to the above concept. Operating temperatures and pressures are being increased continually, requiring stringent engine control. To operate closer to engine stall and choke limits and turbine material structural limits requires accurate and rapid knowledge of the turbine inlet temperature. Since aircraft turbines of the future will utilize variable geometry and large amounts of cooling air, it will be extremely difficult to predict turbine inlet temperatures using turbine outlet temperatures. The closer the measurement can be made to the hottest point in the engine the more precisely the engine can be controlled.

Although various new methods of turbine temperature measurement are being developed, it appears that thermocouples are now and will be for the next five to ten years the most practical means of gas stream temperature measurement in the aircraft engine environment. Because of their simplicity, thermocouples are the most "cost effective" method of aircraft gas temperature measurement. In addition, the data or information obtained with a thermocouple can be transmitted easily to remote receivers.

To meet the high performance requirements of the future high operating temperature military aircraft, the Joint Technology Demonstration Engine Program has been established. Under this project, aircraft engine manufacturers are working under Government contracts to develop the new engines for the next five to ten year period. These engines will be used in supersonic aircraft having multi-mission capability. It has been estimated that operating temperatures within the new engines will be of the order of 2500°F (1370°C) to 2700°F (1480°C) with excursions to 3000°F (1650°C).

The Air Force, realizing that existing temperature measuring and control systems incorporating thermocouples are not adequate to provide the precise temperature control at the elevated operating temperatures that these future turbine engines will require, funded a program with Engelhard Industries under USAF Contract F-33615-74-C-2069, for the development of a new "High Temperature Thermocouple System".

2.0 PROGRAM OBJECTIVES AND METHODS OF ACHIEVEMENT

The main objective of this program was to develop a temperature measurement system for the very high temperature gas streams found in advanced jet engines. Average temperatures of the order of 2500-2700°F (1370°C - 1480°C) will have to be measured and controlled and it is anticipated that hot spots to 3000°F (1650°C) will be encountered.

The measurement system specified above would be a thermocouple probe assembly suitable for interfacing to the Allison GMA 200 Joint Technology Demonstration Engine. It would include suitable thermocouple wires (new); compensating extension wires (new) capable of being used in the operating environment for extended periods of time at 750°F - 1300°F (400-700°C) and having an EMF vs. temperature relationship approximately that of the thermocouple; support structure and thermocouple sheath of advanced materials and capable of withstanding the rigors of long time operation in the engine environment at the specified temperatures.

To reach the desired end, the program was divided into two phases. Phase I was directed towards the examination of suitable materials that could be used in a probe operating in the environment and high temperatures expected in advanced engines; subjecting these materials to qualifying tests and then selecting the optimum materials. Phase II was directed towards the design of a sensor capable of interfacing the Allison GMA 200 Joint Technology Demonstration Engine while using the optimum materials selected in Phase I; assembly of the designed probe; and vibration testing of the assembled probe.

3.0 TECHNICAL CONSIDERATIONS

3.1 General

There are several possible methods for measuring the temperature of the conduction gas in a gas turbine between 1000°C and 1650°C. These methods are in various stages of development and present varying degrees of promise as practical means for accomplishing the purpose.

1. Radiation pyrometer
 2. Fluidics
 3. Acoustical-sonic
 4. Gas temperature density
 5. Gas analysis
 6. Thermocouples
- (1) Radiation pyrometers are probably second best for this application, but this type of devices is complex and delicate. Certain problems such as eliminating dirt from the gas stream that accumulates in the optical system have not been fully resolved. In addition, the radiation pyrometer senses metal temperatures rather than gas temperatures, and this may not be always desirable. Metal temperatures change much more slowly than gas temperatures, and this thermal lag could be objectionable.
 - (2) Fluidic devices can be simple and, theoretically, have fast response characteristics. However, this system is not yet operational because of practical difficulties in manufacturing the sensor to the extremely close tolerances required and the development of a material that would maintain dimensional stability at elevated temperatures. This has resulted in problems with accuracy, repeatability and life.
 - (3) The speed of sound in the gas changes with temperature. If the method for determining the speed of sound in the short spaces existing in gas turbines could be developed, an accurate temperature sensor would result. Although several projects have been established during the last 20 years on this, none is known to have been practical to date, and it is not considered that such will be available within the next five years.
 - (4) The gas temperature density method operates on the change in attenuation of energy radiation with changes in the ambient temperature surrounding the radiator. This method is believed to be in early stages of development and would not be in a usable form for production applications within the next five years.

- (5) Gas analysis has been used in test standard operations and is especially suitable at temperatures in the 1500 to 1600°C level. However, the method is too slow to be practical in a production engine operated, for instance, in an aircraft.
- (6) Thermocouples (thermoelectric devices) have many features that make them attractive as temperature sensors for gas turbines. Among these are: nearly linear EMF output versus temperature, small size, simplicity, wide acceptance, and ease with which they may be integrated into engine controls. Thermocouples are the primary means of sensing temperatures in production gas turbines at the present time, and there is no question concerning the overall suitability of this type of sensor. Because of all of these favorable reasons, the thermocouple was proposed and accepted for development into a sensor for measuring and controlling temperature in advanced gas turbines.

3.2 Probe Design

In practice, the thermocouple probe is immersed in the moving gas and an EMF is produced proportional to the temperature of the hot junction which is in equilibrium with the gas immediately surrounding the probe. To function properly, the probe must reach a temperature very nearly equal to the gas temperature. In addition, it must respond rapidly to a change in the gas temperature. The immersed probe is subjected to very high temperatures, oxidation, erosion, contamination, and mechanical loading. Designing a probe to be accurate and reliable over a long period of time under these conditions often is contrary to design criteria aimed at reducing response time.

Response rates are maximum for a thermocouple probe with an exposed hot junction. The exposed junction, however, is susceptible to reactions with the gas, contamination, erosion, and mechanical failure. The performance of the probe must not be affected by these conditions over the required lifetime. The reliability requirement is easily satisfied by using a junction completely surrounded by protective sheathing. The rate of heat transfer through the sheathing and insulation, however is reduced, slowing the response.

Sometimes, compromises and trade-offs have to be made between opposing requirements to enable a thermocouple probe to best fit a given application. In the present program, a relatively quick response time was considered to be essential and it was decided to design a temperature sensor with an exposed thermocouple hot junction. Because of the high temperatures required, this posed a problem in the selection of materials (thermocouple, compensating extension wire, sheath and support members) used in the probe assembly.

3.3 Materials

Known thermocouple systems that can measure temperatures of the magnitude required under this program are limited in number as for example:

1. Pt-10% Rh vs. Pt ISA Type S
2. Pt-13% Rh vs. Pt ISA Type R
3. Pt-30% Rh vs. Pt-6% Rh ISA Type B
4. W vs. W-26% Re
Doped W - 3% Re vs. W-25% Re
W-5% Re vs. W-26% Re
5. 60% Ir - 40% Rh vs. Ir
50% Ir - 50% Rh vs. Ir
40% Ir - 60% Rh vs. Ir
6. Pt - 40% Rh vs. Ir - 40% Rh (proposed by Glawe)¹

All of the enumerated couples have strong and weak points. For example (1) and (2) can be used in air for long periods of time without excessive drifting but the negative leg (platinum) is relatively weak mechanically at the anticipated high operating temperatures. Also, there is no adequate high temperature base metal extension lead wire. Of all of the couples listed, (3) is about the best suited for the operating environment; however, the EMF is rather low and there is no suitable high temperature base metal extension lead wire available.

The W-Re base metal thermocouple combinations can be used to around 2500°C in an inert atmosphere (helium, argon, etc.) but would never survive as bare metal thermocouples (even for a short time) in the environment encountered during engine operation. These combinations would have to be carefully protected. In addition, the thermoelements are relatively brittle, and there is no suitable high temperature extension lead wire available. In respect to the Ir-Rh combinations, they can tolerate some exposure to an oxidizing atmosphere at elevated temperatures without undergoing catastrophic failure (like the W-Re couples) but the thermo-elements are relatively fragile and the EMF of the couples is low. This low EMF complicates matters further because it makes the development of practical high temperature compensating extension lead wires an extremely difficult task.

Lastly, the Pt-40% Rh vs. Ir-40% Rh combination recently proposed by Glawe¹, can tolerate the anticipated high temperatures and oxidizing atmosphere but its EMF is relatively low (7.770 millivolts at 1500°C). This low EMF may make it difficult to develop a practical, high temperature, base metal compensating extension wire.

After due consideration of all of the above facts, it was proposed that certain noble metal thermocouple-base metal lead wire combinations be examined and if found acceptable be used to meet the objective of this program. These combinations had been previously partly developed by Engelhard Industries for the Air Force via sub-contracts from Detroit Diesel-Allison Division of General Motors Corporation.

¹ Glawe, George E., "New High-Temperature Noble-Metal Thermocouple Pairing, Rev. Sci. Instrum., Vol. 46, No. 8, August 1975.

These were:

- (a) Pt-40% Rh vs. Pt-3% Rh
- (b) Pt-40% Rh vs. oxide dispersion strengthened Pt-3% Rh
- (c) Pt-40% Rh vs. oxide dispersion strengthened Pt

Thermocouple (a) and a base metal lead wire combination were developed first under the previously mentioned contract. The EMF of the lead wire matches that of the thermocouple very closely over the range of 0 to 1000°C. Oxidation tests indicated that the lead wire would remain stable even though it were operated in air to 850°C for long periods of time. The availability of this lead wire could result in a substantial economic advantage because very short lengths of the noble metal thermocouple (say up to 1" to 2" long) would be used and long lengths of base metal extension wire (relatively inexpensive) would be utilized to complete the circuit. The lead wire-thermocouple junction could be located at around 700°C. Detroit Diesel-Allison has demonstrated the feasibility of the approach using Engelhard Industries Platinel (approximately 1/2" long) and ISA Type K compensating extension wire in probes to measure the turbine inlet temperature in the T-56 engine.

Thermocouple (b) was formulated when a question was raised about the high temperature strength of the Pt-3% Rh leg. A thorium oxide dispersed phase was added and the high temperature strength, as determined by stress-to-rupture tests at elevated temperatures was improved slightly. However, the high temperature strength of the oxide strengthened alloy was still significantly below that of the Pt-40% Rh alloy.

In the same program, in order to increase the EMF of the Pt-40% Rh vs. Pt-3% Rh combination, a thorium oxide dispersion strengthened platinum leg was substituted for the Pt-3% Rh leg. The EMF of the resulting combination, Pt-40% Rh vs. thoriated Pt, was higher (around 20%) than that of the Pt-40% Rh vs. Pt-3% Rh couple. Stress-to-rupture tests, at that time, on the thoriated platinum wire showed it to be very close to that of the Pt-40% Rh leg (recent tests have shown the thoriated Pt to have a strength higher than that of Pt-40% Rh). A base metal compensating extension wire whose EMF matches very closely that of the Pt-40% Rh vs. Pt thermocouple over the range of 200°-900°C was also developed.

For the present program, it was proposed that all three thermocouples be re-examined and the best combination be selected for manufacture into a test probe. In addition, since the program for Allison was a relatively brief one, the selected combination would be characterized and an EMF vs. temperature table be developed. Furthermore, reproducibility from lot to lot would be studied and if possible a commercial tolerance would be determined. This effort would result in the identification of an optimized long-life, reliable thermocouple combination for use continuously at temperatures to around 1500°C and intermittently at temperatures as high as 1650°C.

While work would be in progress to select the optimum thermocouple combination a portion of the effort could be directed towards optimization of the base metal lead wire for both the Pt-40% Rh vs. Pt-3% Rh and Pt-40% Rh vs. Pt-0.6% ThO₂ combinations, (the use of thoria as a strengthening agent does not significantly affect the EMF of the couple). In the case of thermocouple system (a) an EMF reversal plus to minus to plus EMF values occurs below 75°C. An attempt would be made, by alloying means, to shift this reversal below -50°C (ambient temperature in high altitude flight). In the case of the second lead wire (for Pt-40% Rh vs. Pt-0.6% ThO₂) an attempt would be made to improve the EMF match between couple and lead wire in the range of -50 to +200°C.

As part of this program, the EMF reproducibility from lot to lot of the finally selected lead wire would be determined. In addition, the chemical compatibility of the lead wire and the selected thermocouple wire with the environment (insulators, sheath) would be determined at the operating temperature.

The optimum sheath material (metal or ceramic) for the finished probe would be selected based on temperature, environmental, mechanical strength, and chemical compatibility considerations. Tests would be run to determine how well the selected sheath would withstand the operating temperature in air. The following materials would be considered Pt-20% Rh, Pt-30% Rh, Pt-40% Rh, oxide dispersion strengthened platinum, noble metal cermet and advanced technology ceramic, such as CVD silicon carbide and silicon nitride.

4.0 PROGRAM PLAN

The program was divided into two phases and each of the phases was then subdivided into tasks:

DEVELOPMENT OF PROBE (MATERIALS) PHASE I

Task A. Candidate Material Selection

- (a) Thermocouple Alloys and Compensating Extension Wires
- (b) Sheath and Support Material
- (c) Crushable MgO Insulating Material

Task B. Material Procurement or Fabrication

- (a) Purchase
- (b) Fabricate at Engelhard Industries

Task C. Qualifying Tests

- (a) Thermocouple Alloys and Compensating Extension Wires
 - (1) EMF of Candidate vs. US Thermoelectric Reference Standard (designated Pt67)
 - (2) Stress-to-Rupture Tests (thermocouple elements)
 - (3) EMF Stability Tests at Elevated Temperatures
 - (4) Compatibility at Elevated Temperatures; Thermoelements and Extension Wires With MgO Insulation
- (b) Sheath and Support Material
 - (1) Stress-to-Rupture Tests
 - (2) Compatibility at Elevated Temperatures With MgO Insulation

Task D. Selection of Optimum Materials

- (a) Thermocouple Alloys and Compensating Extension Wires
- (b) Sheath and Support Material
- (c) Crushable MgO Insulating Material

Task E. Determine Reproducibility From Lot to Lot

- (a) Thermoelement Alloys Selected vs. Pt67
- (b) Extension Wire Leg Alloys Selected vs. Pt67

Task F. Determine Tentative EMF vs. Temperature Relationship

- (a) Selected Thermocouple (-50°C to +1650°C)
Provisional Procurement Tolerances.
- (b) Selected Compensating Lead Wire (-50°C to 1000°C)
Suggested procurement approach.

DEVELOPMENT OF PROBE (DESIGN) PHASE II

Task H. Thermocouple Sensor Design

Task I. Fabrication of Prototype Probe

Task J. Vibration Testing of Prototype Probe

5.0 MATERIALS DEVELOPMENT - PHASE I

5.1 Task A - Candidate Material Selection

5.1.1 Thermocouple Alloys and Compensating Extension Wires

Candidate thermoelements and extension wires are as follows:

Thermoelements	Polarity	Engelhard Alloy No.	Extension Wires
Pt-3Rh Pt-40Rh	- +	16444 11791	9R567 Tophet D
Thoriated Pt-3Rh Pt-40Rh	- +	9R1530 11791	9R567 Tophet D
Thoriated Pt Pt-40Rh	- +	9R763 11791	9R567 Nichrome

Alloy 9R-567 Nominal Composition - 94.5%Ni-4.5%W
Producer: Engelhard Industries

Alloy Tophet D Nominal Composition - 35%Ni-20%Cr-45%Fe
Producer: Wilbur B. Driver Co., Newark, N.J.

Alloy Nichrome Nominal Composition - 60%Ni-15%Cr-24%Fe-1%Si-Trace Mn
Producer: Driver-Harris Co., Harrison, N. J.

5.1.2 Sheath and Support Materials

The following materials were selected as worthy of consideration for use in the projected application. All of the candidate materials selected had melting points above the desired maximum operating temperature (1650°C). Some of the materials under consideration had proven capabilities in air at 1650°C and these were notably the Pt-Rh alloys. Our aim in this program was to determine whether there were materials superior to the Pt-Rh alloys. Should this not be possible, then we could fall back and use the best of the Pt-Rh alloys.

80% Pt - 20% Rh	Alloy EA 7609
70% Pt - 30% Rh	Alloy EA 7146
60% Pt - 40% Rh	Alloy EA 8383
69.58% Pt - 30% Rh - 0.42% ThO ₂	Alloy 9R-1529
* 90% Ir - 10% Rh + .15% ThO ₂	Alloy 9R-876
95% Pt - 5% ThO ₂ (Cermet)	Alloy 9R-1545
99% Pt - 0.6% ThO ₂	Alloy 9R-763
CVD Silicon Carbide	
Silicon Nitride	

* Material developed under sub-contract from Detroit Diesel - Allison for use in a fluidics probe. This material also has been used successfully as a sheath material (protect W-Re thermoelements) in instrumentation temperature probes for short periods of time in advanced engines operating at temperatures considerably above those required under this contract.

5.1.3 Crushable MgO Insulation

Both MgO and Al₂O₃ insulation in crushable form were considered for use in the projected application. Because of its better "packing" ability and because of successful past experience in similar probes operating at lower temperatures, commercial quality MgO was selected for use in the final design.

5.2 Material Procurement or Fabrication

All of the thermoelement materials were obtained from within Engelhard Industries. Alloys EA 16444 (Pt-3%Rh) and EA 11791 (Pt-40%Rh) are standard alloys and were obtained as .050" diameter wire directly from the EI Carteret Operation. Alloy 9R-763 (99.4%Pt-0.6% ThO₂) and Alloy 9R-1530 (96.42% Pt - 3% Rh - 0.58% ThO₂) could have been obtained from the Carteret Operation but for control purposes it was decided to fabricate these alloys at the E.I. Research and Development Department at Menlo Park.

Two alloys, 9R-763 and 9R-1530, were fabricated by the Powder Metallurgy Section in R&D. In both cases thermocouple quality platinum powder (Table 1) was used and the thoria, in the correct proportions, was admixed. After isostatic pressing and sintering the bars were worked to wire (.050" diameter for stress-to-rupture testing and .040" diameter for use in the probe assembly).

In respect to the base metal compensating extension wires, Alloy 9R-567 and other experimental modifications of this alloy, these were melted, cast and fabricated into wire in the R&D Metal Processing Laboratory. Tophet D wire was obtained from the Wilbur B. Driver Company, Newark, New Jersey (some wire was at hand from previous work and recent samples were obtained through the courtesy of Dr. T. P. Wang of the WBD Co.). The Nichrome wire was obtained from the Driver-Harris Company, Harrison, New Jersey (likewise some wire was at hand from previous work and recent samples were obtained from Mr. C. L. Guettel of the Driver-Harris Co.).

Three Pt-Rh alloys intended for use as sheath or support materials were obtained from the EI Carteret Operation in the form of sheet (2" x 6" x .010" thick). These are EA7609 (80%Pt-20%Rh), EA 7146 (70%Pt-30%Rh), and EA 8383 (60% Pt - 40% Rh). One material, a 90%Ir-10%Rh alloy containing 0.15% thoria (9R-876), which has previously been hot extruded for another purpose, was requisitioned for possible application in this project and was obtained in the form of 1/8" diameter rod and tubing 1/4" O.D. by .020" wall. Two thoriated alloys, 9R 763 (99.6%Pt-0.6%ThO₂) and 9R-1529 (69.58%Pt-30%Rh-0.42%ThO₂), were isostatically pressed, sintered and partially worked at R&D and were then finished rolled to sheet (2" x 6" x 0.010" thick) at the Carteret Operation.

Four platinum cermet bars (9R-1545, 95% Pt-5% ThO₂) were fabricated using powder metallurgy techniques.

Bar #1

Isostatically pressed powder compact, 30,000 psi
Sintered in air at 1400°C for 20 hours.
Density 78.38% of theoretical
Resintered at 1600°C, 4 hours, vacuum 10⁻⁵ torr
Density 86.57% of theoretical

Bars #2 and #3

Isostatically pressed powder compact, 30,000 psi
Pre-sintered at 800°C, (1 hour) in vacuum of 10⁻⁵ torr
Sintered at 1600°C, 4 hours, vacuum 10⁻⁵ torr
Density Bar #2, 87.55% of theoretical
Bar #3, 87.31% of theoretical

Bar #4

Isostatically pressed powder compact, 30,000 psi
Pre-sintered at 600°C, 1 hour, vacuum 10^{-5} torr
 900°C, 4 hours, vacuum of 10^{-5} torr
Sintered at 1600°C, 6 hours, vacuum of 10^{-5} torr
Density 93.08% of theoretical

Two non metallic materials were considered for the sheath application. One of these was a CVD silicon carbide and the other was a silicon nitride. Deposits and Composites Inc., Reston, Virginia were contacted in respect to a CVD silicon carbide tube and quoted a price of \$800 for one experimental tube (lower price for larger quantities). Because of possible problems with the use of what may be brittle materials in an engine, CVD silicon carbide was dropped from further consideration. Since some silicon nitride, obtained from the Carborundum Company (for another experiment), was at hand plans were made to test it as a possible sheath material.

The MgO crushable insulators used in the finished probe were obtained from the Norton Company, Refractories Division, Worcester, Massachusetts. Because the ceramic during use in the thermocouple probe would be exposed to the products of combustion a commercial quality MgO (99.4% MgO) was considered to be adequate for the purpose.

5.3 Qualifying Tests

5.3.1 Thermocouple Alloys and Compensating Extension Wires

A series of qualifying tests were run on the candidate thermoelements and their respective compensating extension wire legs to determine the optimum thermocouple-extension wire system. Of all of the tests performed, the most critical, from the standpoint of their weight in making the final judgement, were: stress-to-rupture (indication of high temperature strength), EMF stability after exposure to high temperatures in air for relatively long periods of time and compatibility with air and the preferred MgO ceramic insulators (concerned mainly the compatibility of thoriated platinum or thoriated platinum-rhodium alloys).

The EMF vs. Pt67 was determined for all candidate thermoelements and their respective compensating extension wire legs. These tests, as well as all other similar tests made during this program, were performed in accordance with ASTM E220-72, "Standard Method for Calibration of Thermocouples By Comparison Techniques". All standards used are traceable to NBS via calibration certificates. Temperatures reported are on the International Practical Temperature Scale of 1968 (ITS 68) -- see Metrologia, Vol. 5, No. 2, April 1969, for additional details. The results of the present tests are shown in Tables 2 and 3. For the thermocouple elements, the EMF vs. Pt67 was determined over the range of -50°C to 1650°C, while for the extension wire legs, the EMF vs. Pt67 was determined over the range of -50°C to 1000°C.

In an attempt to improve the EMF vs. temperature match for the extension wires to the three candidate thermocouples, the composition of one extension wire alloy common to all three thermocouple combinations was modified slightly. This was done by adding very small amounts of Mn, Si, Al, Mo and/or W to a master 9R-567 alloy (94.5%Ni-4.5%W). The results of EMF tests vs. Pt67 are shown in Tables 4 through 8.

Using the EMF vs. Temperature Values determined under this contract for Pt-40%Rh vs. Pt-3%Rh and Pt-40%Rh vs. thoriated Pt (EMF of Pt-40%Rh vs. thoriated Pt-3%Rh similar to that of Pt-40%Rh vs. Pt-3%Rh) thermocouples, the "wanted" EMF for the negative (-) extension wire leg for each of the three thermocouple combinations was determined. This was done by subtracting the EMF vs. Pt67 values of the positive (+) extension wire leg (Tophet D or Nichrome as the case may be) from the EMF values of the respective matched thermocouple combinations (see Table 9). The effect of the small alloying additions to the master 9R-567 alloy can be fully assessed by comparing the EMF of samples of the new alloys (Tables 4-8) determined against Pt67, to the EMF of the "wanted alloy" vs. Pt67.

Stress-to-rupture tests were run at 1450°C in air on Pt-3%Rh, thoriated Pt-3%Rh, Pt-20%Rh, Pt-30%Rh, Pt-40%Rh, Pt-0.6% ThO₂ and platinum. All of the materials tested were candidates for consideration as thermocouple elements or as a sheath or structural member. The average life in hours (time to failure) was determined for each of these materials on .050" diameter wire under three different load conditions (1350 psi, 1120 psi and 900 psi). The results of these tests are summarized in Table 10 and shown graphically in Figure 1.

EMF stability tests were run on three of the four candidate thermocouple elements -- EA 16444 (Pt-3%Rh), EA 11791 (Pt-40%Rh) and 9R-763 (99.4%Pt - 0.6%ThO₂). These were run at 1450°C and 1650°C in air for 200 hours at each test temperature. All three materials were tested simultaneously as shown in Figure 2. The bead and approximately 1" of each thermocouple element protruded from the end of loosely fitting high purity (99.5% min. Al₂O₃) insulators. The exposed portion was held within an approximately four inch constant temperature zone at 1450°C or 1650°C depending upon the test. Each thermoelement was calibrated in another furnace vs. Pt67 before and after each aging test. The results of these aging tests may be found in Tables 11 through 13. In respect to the fourth candidate thermocouple element, 9R-1530 (96.42%Pt-3%Rh-0.58%ThO₂) inconclusive results were obtained and a retest was scheduled. However, it was decided not to complete these tests because the stress-to-rupture properties of this alloy were not appreciably better than that of its counterpart material EA 16444 (Pt-3%Rh) and much lower than that of 9R-763 (Pt-.6%ThO₂).

In like fashion EMF stability tests were run on the three candidate extension wire legs - Tophet D, Nichrome and 9R-567. The test procedure was similar to that for the three thermocouple elements as shown in Figure 2. However, for this test, a Lindberg Hevi-Duty muffle furnace was used.

This furnace was tilted slightly so that there would be some movement of laboratory air over the bead and exposed portion of the candidate extension wires (chimney effect). A Pt-10%Rh vs. Pt couple with an alumina protection tube was inserted to monitor and control the furnace temperature. The position of the "test package" (bundle of three wires in alumina insulators) was adjusted so that the bead and approximately 2" of the wires (about 1" of wires inside single bore tubes) were within an approximately 4" constant heat zone. The extension wires within this constant temperature zone were "aged" in this fashion in air, for 200 hours at $850^{\circ}\text{C} \pm 10^{\circ}\text{C}$.

After this treatment the three wire test package was removed from this furnace and inserted into another furnace used solely for calibration. The EMF of each of the three candidate materials was then determined versus Pt67 in the "as is" condition (bead and 1" of wires protruding out of insulators. Next the bead and approximately 1" of the exposed wires were cut off and the remaining wires were again calibrated vs. Pt67. The results of these stability tests are shown in Tables 14 through 16.

A quick examination of the results for Alloy 9R-567 showed an appreciable change in EMF after the aging test in air at 850°C . These results were surprising because in previous tests (heating 9R-567 in air for 24 hours at 750°C , 800°C , 850°C , 900°C , 950°C and 1000°C) it was shown that this alloy could be used in air for 24 hours to about 850°C without appreciable change in EMF (see Table 17). As a further check, the EMF stability was re-determined on Alloy 9R-567. This time the temperature was 750°C ; the time was 200 hours; and the test was conducted on samples from two different bars. The results of this test show the material to be quite stable after aging (Table 18).

The EMF stability tests on the base metal compensating extension wires as performed during this program are perhaps more severe than those to be encountered in actual service. In the latter case the extension wires will be encapsulated and not exposed to a moving stream of gas (containing air). For this reason it is anticipated that service life will be at least as good as that predicted by the stability tests if not better.

Of some concern was the possibility of incompatibility between the thoriated platinum and the thoria containing cermet with the MgO insulation at elevated temperatures. Compatibility tests were run in air at 1450°C and 1650°C with the critical items

such as thoriated platinum (9R-763) and the Pt cermet (9R-1545, 95%Pt-4%ThO₂) in contact with MgO. No reaction between the 9R-763 or 9R-1545 and the MgO was visually noted. In an earlier test, at 1650°C in air, silicon nitride volatilized and coated the other test specimens necessitating a re-test.

5.3.2 Sheath and Support Material

Stress-to-rupture tests at 1450°C in air were run on .050" diameter wire samples of Pt-20%Rh, Pt-30%Rh, Pt-40%Rh and Pt-0.6%ThO₂. The results of these tests were reported in Section 5.3.1 (see also Table 10 and Figure 1).

Compatibility tests in air at 1450°C and 1650°C were run with candidate sheath materials in contact with MgO. Silicon nitride vaporized very quickly at 1650°C. The remaining materials tested appeared to be compatible with MgO at the test temperature and test environment -- 9R-763 (Pt-0.6%ThO₂), 9R-1545 (95%Pt-5%ThO₂), Pt-20%Rh, Pt-30%Rh and Pt-40%Rh.

5.4 Selection of Optimum Materials

Based on test results obtained during the course of this program EA 11791 (Pt-40%Rh) vs. 9R-763 (99.4%Pt-0.6%ThO₂) were selected as the best thermocouple combination. The lead wires that would be used are Nichrome vs. 9R-682 (95%Ni-4.5%W-0.5%Mn). 9R-682 is a modified version (0.5 Mn added) of 9R-567. Of the three thermocouple combinations considered in this program, the selected couple has the highest EMF and the best high temperature strength (as determined by stress-to-rupture tests at 1450°C in air). The compensating wires selected match the EMF of the thermocouple wire used in the probe within .144 millivolts (144 microvolts) over the range of 0 to 1000°C. Over narrower temperature ranges the mismatch error will be much smaller.

Because of its superior stress-to-rupture properties at 1450°C, 9R-763 (99.4%Pt-0.6%ThO₂) was selected as the sheath and shield material. The runner up shield material was 9R-1545 (95%Pt-5%ThO₂) cermet which was not completely developed at the time the sheath material was selected. At present, it is anticipated but not proven that the addition of 5 weight percent (10.9 volume %) thoria to platinum may substantially improve the abrasion resistance of the resulting product.

Good commercial quality crushable MgO preforms manufactured by the Norton Company were chosen as the insulation material. An analysis on a sample from the lot of MgO preforms used in the final assembly was performed by Ledoux & Company and the results are shown in Table 19.

5.5 EMF Reproducibility of Selected Thermoelements and Extension Wires

In order to check the EMF reproducibility from lot to lot of each of the elements selected for the thermocouple and the negative extension lead wire some additional material was produced and the EMF vs. Pt67 was determined. The results of all tests run during the course of this program are shown in Tables 20 through 25. Also, in an attempt to see whether an alternate material, Tophet C, would have a more reproducible EMF from lot to lot than does Nichrome, Engelhard Industries files were searched to determine if this data were available from previous work. Old test results for Tophet C are shown in Table 26. Although Tophet C appears to have better reproducibility from lot to lot, the EMF vs. Pt67 for this material, in the upper temperature ranges (500°C to 1000°C), does not completely match that of Nichrome and hence cannot be substituted for Nichrome without a substantial composition change in the negative leg extension wire. Tophet C is a trade marked alloy produced by Wilbur B. Driver Company and has the following nominal composition: 61%Ni-15%Cr-bal. Fe.

5.6 Proposed EMF vs. Temperature Relationship

Based on results obtained in this program as well as experience with other thermocouple systems a provisional EMF vs. temperature relationship has been established for the Pt-40%Rh vs. thoriated Pt thermocouple and is shown in Table 27. Nominal EMF vs. Pt67 relationships for each thermoelement have been determined and also are shown in Table 27.

It is proposed, at the outset, that a tentative tolerance of $\pm 0.5\%$ at temperatures of 600°C to 1650°C be used as a guide if future procurement of this new thermocouple were required (see Table 27 for millivolt equivalents). At this point in time, with so little experience on the reproducibility of the thermoelements (in particular the thoriated Pt) this tolerance is more advisory rather than mandatory. With additional experience, a final tolerance can be set. The EMF vs. Pt67 values for the thermoelements shown in Table 27 are intended only for information and it is recommended that only the tolerance on the matched thermocouple be used for initial procurement of this particular combination.

In respect to the compensating extension wires, no EMF tolerance has been set because of the spread in EMF values obtained on samples from three lots. In all fairness, it should be pointed out that Nichrome as well as Tophet C and Tophet D are produced by their respective manufacturers for use as resistance wire (heating elements, etc.) and not as thermoelements. However, it appears that a good deal of control of their chemistry is exercised during processing in order to meet rigid resistance specifications. It is for this reason that these materials were actively considered for use as compensating extension wires for the proposed couples.

For future procurement, until additional information is obtained, it is suggested that Nichrome be purchased from its producer to the target EMF shown in Table 28. One approach would be to obtain samples from several lots from the producer and then determine the EMF vs. Pt67 at perhaps two points (say 400°C and 600°C). The EMF of the negative leg can then be "tailored" to give the desired EMF when matched to the selected Nichrome.

Of possible help here, to obtain optimum EMF matching with the selected Nichrome, are the EMF data shown in this report when small alloying modifications were made to EI Alloy 9R-567 (see Tables 4 through 8). One of these modified alloys, 9R-1522, when matched to the Nichrome used in the fabricated probes gave a slightly better EMF match to the selected thermocouple than did 9R-6C2. However, 9R-1522 was not used in the finished probe because of limited EMF stability experience.

6.0 DEVELOPMENT OF PROBE - PHASE II

6.1 Thermocouple Sensor Design

Measuring the temperature of a gas in a turbine presents problems that are generally more difficult to overcome than those encountered in the measurement of solid and surface temperatures. A thermocouple can indicate only the temperature of the thermocouple junction. In general, this will not be equal to gas temperature unless special precautions are taken. The degree to which the temperature of the junction approaches the true temperature of its environment depends on the exchange of energy between the thermocouple and the surrounding media.

A steady-state difference between thermocouple and gas temperature is commonly called an "error" but actually represents the balancing of four well defined phenomena²: (1) heat transfer to or from the probe by radiation, (2) heat transfer by conduction, (3) conversion of kinetic energy to thermal energy in the boundary layer around the thermocouple, and (4) heat transfer from the boundary layer to the junction by convection. During transients, the thermocouple will lag behind any change in gas temperature, due to its thermal capacity, resulting in a response-rate "error"². It is the responsibility of a probe designer to determine the differences which exist, and to correct them, or to design the probe such so that the differences or "errors" are acceptably small.

In the development of a particular thermocouple probe for a system, operating parameters must be considered carefully and enumerated after a thorough analysis. These parameters should include, among others, gas velocity, temperature and composition, and enclosure dimensions and temperature².

² Moffat, Robert J., "Gas Temperature Measurement, Temperature Its Measurement and Control in Science and Industry, C. M. Herzfeld, Ed-in-Chief (Reinhold Publishing Corp., New York, 1962), Vol. 3, Part 2, p553.

A basic probe design that has been used very successfully at Detroit Diesel-Allison in the measurement of gas turbine temperatures, albeit at lower operating temperatures (say 980°C to 1150°C), was chosen for this project. In the evolution of that particular design much consideration had been given to the factors that influence the temperature differences or "errors" mentioned above. However, since this probe had been used in practice at lower temperatures only the probe configuration was acceptable but factors relating to the mechanical integrity of the probe had to be examined and evaluated. This then was the area of concern for Engelhard Industries.

The design analyses at Engelhard Industries for the proposed thermocouple probe (T4.1 thermocouple) were based upon calculations formulated from the aerodynamic loading anticipated from the defined use conditions; simple stress concentration related to experimental stress-to-rupture data; and economic justification balanced against geometrical restraints and fabrication practicalities. Several meetings were held with representatives of Detroit Diesel - Allison to obtain necessary installation information so that the probe could physically interface the Allison GMA Gas Generator and function properly under its projected operating conditions. The mechanical properties, such as stress-to-rupture and Young's Modulus, needed for the stress analysis were obtained as a result of work on this project (stress-to-rupture) or previous work which was reported in the literature³ (Young's Modulus).

After consultation and input from Detroit Diesel-Allison and concurrence from WPAFB, a probe design was finalized as shown in DWG. NO. DDS 20200 in Appendix A. The design analysis for the new probe is also shown in Appendix A. The sheath and structural member material selected for the final probe design is EA Alloy 9R-763 (99.4%Pt-.06%ThO₂). Of the materials subjected to stress-to-rupture tests during this program, at 1450°C (including Pt-40%Rh), 9R-763 had the best values.

6.2 Fabrication of Prototype Probe

The fabrication of one prototype probe, built to specifications shown in EI Dwg. No. DDS 20200 (Appendix A) and its ultimate vibration testing at 1450°C were required by subject contract. However, because of the experimental nature of this probe, it was decided to put in process enough materials for five fully assembled sensors. The reasoning here was that should fabrication problems be encountered (due to brazing of components or in the ceramic compacting process) then at least one probe would be finished and tested to meet contract requirements. The anticipated

³ Papadakis, E. P., K. A. Fowler, L. C. Lynnworth, A. Robertson and E. D. Zysk, "Ultrasonic Measurements of Young's Modulus and Extensional Wave Attenuation In Refractory Metal Wires at Elevated Temperatures With Application to Ultrasonic Thermometry", J. Appl. Phys., Vol. 45, No. 6, June 1975, p2409.

assembly and fabrication problems did not arise and five finished probes were obtained -- all were considered acceptable. Various views of these finished probes are shown in photographs, Figures 3 and 4. The EMF vs. temperature relationships for the matched thermocouple wires and the matched compensating extension wires used in the finished probes may be found in Table 29.

Because a platinum with a dispersed thorium oxide phase (9R-763) was used as the shield tube, TC tube and the negative leg of the thermocouple, certain joining precautions were taken. In previous work⁴ it had been shown that some of the strength advantages obtained from the use of the dispersed oxide phase could be nullified or minimized should conventional oxy-hydrogen or arc welding techniques be used to join this material to another material (in the joining process the oxide strengthened material is melted). For this reason, the 9R-763 thermoelement was not welded at the hot junction to the Pt-40%Rh thermoelement but this junction was effected with a strap of a noble metal alloy having a melting point higher than that of the maximum operating temperature but slightly lower than pure platinum. In addition, the probe shield tube and TC sheath were joined as shown on the drawing using appropriate brazing alloys.

During the assembly of the probe the following sequence was followed: swaged assembly (TC wires, MgO insulation, 9R-763 sheath) was cut to desired size; sheath and insulation removed from each end; sheath trimmed; hot junction effected as shown on drawing; shield tube brazed to thermocouple sheath; thermocouple sheath (cooler end) brazed to Inconel 600 support tube with previously welded flange; lead wires connected; "potted" inside of support tube with cement; threaded extension wires and TC wires as shown on drawing through cap assembly (minus cap cover); tack welded cap assembly to top of flange; "potted" top of cap assembly with cement; tack welded cover on cap assembly; crimped tube (optional) on to extension wire. The brazing materials that were used are as follows:

VTG Braze 205 (75%Pt-20%Pd-5%Au)	junction strap and shield tube
Liquidus 1695°C	to TC sheath
Solidus 1655°C	

Engalloy 255 (82%Au-18%Ni)	TC sheath to Inconel 600
Eutectic 950°C	support tube

6.3 Vibration Testing of Prototype Probe

Vibration testing at $1450^{\circ}\text{C} \pm 25^{\circ}\text{C}$ of the finished probe was performed outside Engelhard Industries. Specifications outlining the requirements for this test were prepared and first submitted to four laboratories with a request to bid. A copy of this specification is shown in Appendix B.

⁴ Albert, H. J. and J. S. Hill, "Development of a Platinum-Thorium Oxide Alloy For Resistojet Thruster Use", Final Report NASA CR-111959, July 1971.

The four laboratories that were contacted are:

Associated Testing Laboratory
Burlington, Massachusetts

RCA
Astro-Electronics Division
Princeton, New Jersey

General Electric Company
Valley Forge Space Center
Philadelphia, Pennsylvania

Lockheed Electronics Company
Product and Systems Division
North Plainfield, N. J.

Three of the laboratories contacted responded with a "no bid". The fourth company, General Electric Company responded with a qualified bid (see letter, Appendix B). The General Electric Company bid was accepted and the vibration tests were performed on one prototype thermocouple probe in accordance with the Engelhard Industries Specifications and the GE exceptions to this specification.

The probe under the specified vibration test survived intact and was returned to Engelhard Industries. Visual examination of this returned probe disclosed that outside of a little discoloration (very little) the probe had not suffered any adverse effects. Electrical continuity tests on the returned probe showed that the thermocouple lead wire system was still intact. A written report covering the two days of vibration testing was submitted by the General Electric Company and is also shown in Appendix B.

7.0 DISCUSSION

It is well known that certain noble metals or combinations of these metals (alloys) have excellent high temperature oxidation resistance but have only fair to good mechanical strength at very high temperatures. One of the objectives of this program was to utilize the excellent elevated temperature oxidation resistance along with the best mechanical strength obtainable in noble metals or their alloys to achieve the optimum thermocouple combination.

Using stress-to-rupture tests as a discriminating criterion, Pt-40%Rh was selected as the positive leg of this couple and an oxide dispersion strengthened platinum (9R-763, 99.4%Pt-0.6ThO₂) as the negative leg.

The choice of Pt-40%Rh for one leg was logical because it has an excellent resistance to oxidation and has long been known to have the highest stress-to-rupture properties in air at elevated temperatures of all of the commercial noble metal alloys. In addition, it has been used with much success as a thermocouple element when matched to Pt-20%Rh as the other leg⁵. This particular combination (Pt-40%Rh vs. Pt-20%Rh) although enjoying an appreciable EMF stability when used at elevated temperatures nevertheless has not become a commercial success because of its low EMF response at elevated temperatures (3.237 millivolts at 1500°C and sensitivity at this temperature of .0043 millivolts/degree C).

As indicated by the test results in this project as well as by the results of previous work⁴, the use of an oxide dispersion strengthened platinum as a negative thermoelement offers some interesting possibilities. Information obtained from tests show that there is relatively little effect of 0.6% thorium on the room temperature mechanical properties. This is also true for room temperature and high temperature electrical properties such as resistance and EMF vs. Pt67. For all practical manufacturing purposes the thoriated material behaves as does commercially pure platinum. The advantage of adding thorium shows up in the high-temperature, long term strength properties. The stress-to-rupture tests reported here show that in this type of testing, the 99.4% Pt - 0.6% ThO₂ alloy has a much greater life than platinum - 40% rhodium (1450°C, 900 psi, 2000 hours for thoriated Pt and 73 hours for Pt-40%Rh).

The observation in dispersion strengthened platinum of relatively unaffected room temperature properties and enhanced high temperature mechanical properties also has been reported for other materials such as nickel and copper. While the theoretical basis for improved high temperature properties has not yet been firmly established, it appears that grain-boundary sliding plays an important role in high-temperature yielding and creep. When most of the grain boundaries are parallel with the stress axis, that is, when there is a highly elongated microstructure, there is, on the average, a low resolved shear stress on the boundaries, and this minimizes the overall

⁵ Bedford, R. E., "Reference Tables For Platinum-40% Rhodium/Platinum-20% Rhodium Thermocouples", Rev. Sci. Instr. Vol. 36, No. 11, November 1965, p1571.

amount of sliding. The maximum sliding should occur when the "Grain Aspect Ratio" or GAR is unity (equi-axed grains). It appears probable that a coarse equiaxed structure should have better high temperature strength than a fine equiaxed structure because fewer boundaries will reduce the total amount of grain boundary sliding. Apparently, the long elongated grains result from the addition of thorium to platinum plus the thermo-mechanical processing required to fabricate this material. Of additional interest is the fact that even after long times at elevated temperatures (of the order of 1450°C) the elongated microstructure of the thoriated platinum is retained.

An example of thoriated platinum microstructure (in the as worked condition - outside of stress rupture furnace) may be found in Figure 5. In another photomicrograph, Figure 6, same wire, an annealed portion of this material is shown (in stress rupture furnace at 1200°C for 2117 hours - away from break). A quick examination of both photomicrographs discloses a marked similarity in structure (both have elongated grains). For further information, a section, Figure 7, was taken through the same thoriated platinum wire after stress-to-rupture testing (vicinity of break). It can readily be seen that the "break" follows a tortuous path across the section of wire - elongated grains still present. For comparison purposes a section through a Pt-40%Rh wire in the vicinity of a break after stress-to-rupture testing and another section through this wire showing a fully annealed structure may be seen in Figures 8 and 9. In respect to Figure 8, it is clearly shown that cavities have formed in the grain boundaries perpendicular to the direction or axis of the stress. When these cavities join failure of the wire occurs. In this case, the failure line across the wire is short and direct.

Since thorium oxide is thermodynamically a very stable oxide, it was anticipated that very little if any decomposition would take place during use of the thoriated platinum alloy at elevated temperatures. The results of the stability tests performed at 1450°C and 1650°C for 200 hours indicate that the EMF of the thoriated platinum is not significantly affected after this exposure. Detailed results of the test are shown in Table 13.

The thoriated platinum material was also chosen for the thermocouple sheath as well as the shield tube (tip of the probe). The reasons for choosing this material for this purpose are the same as those outlined in support of its use as a thermoelement. No difficulty was encountered in fabricating the sheath or the closed end shield tube. In the latter case the closure was accomplished by spinning the end of the tube and then sealing it with molten platinum.

In respect to the shield tube backup material, cermet alloy 9R-1545 (95%Pt-5% ThO₂), some success was achieved. A density of 93.08% of theoretical was finally obtained (on the fourth try). This material is machinable and appears to be resistant to thermal shock. Two samples were quenched into water after heating at 1500°C for 15 minutes. No cracking or distortion was noticed after this test. The physical characteristics of these samples were as follows:

Sample 1

Density 86.57% of theoretical. Machined to test size; 2.359" long, O.D. .462", I.D. at top .270", I.D. at bottom .122". The .270" I.D. covered almost the whole length of the tube or to within .100" of bottom where the I.D. became .122".

Sample 4

Density 93.08% of theoretical. In as pressed and sintered condition. Test size was 1.470" long by about .436" O.D. (average) and .129" I.D. (average).

With a little more effort the density can be increased through improved fabrication techniques. A density of over 90% of theoretical may be adequate but a higher density would be preferred.

At this point, the cermet shows some promise for use as a shield tube for thermocouples operating at temperatures to around 1650°C where resistance to abrasion (carbon particles, dust etc. in the gas stream) would be required. The microstructure of two of the bars (#2 and #4) produced in this program are shown in Figures 10 and 11. The distribution of the thorium oxide throughout the cross section is good.

Assembly of the selected components into the finished probe presented no insurmountable problems. Perhaps because of anticipated difficulties, mainly in the brazing of the thoriated platinum components and the compaction of the MgO insulated thermocouple, greater than usual care may have been taken during fabrication and assembly and this in turn resulted in fewer actual problems and the successful assembly of all probes in process.

One of the five thermocouples assembled, picked at random, was submitted to the General Electric Company for vibration testing according to the specifications shown in Appendix B. The thermocouple survived all testing as demonstrated by continuity, insulation, resistance and visual inspection. Of particular interest is the fact that all brazed connections in the probe survived the vibration test.

8.0 CONCLUSIONS

A review of the results obtained during this program, as documented in this report, has resulted in the following conclusions:

- A. The Pt-40%Rh vs. 99.4%Pt-0.6%ThO₂ thermocouple offers interesting possibilities for the measurement and control of gas turbine temperatures to approximately 1450°C with short time excursions to 1650°C.
- B. Both thermoelements selected have good oxidation resistance and have relatively good EMF stability in air at the projected gas turbine operating temperatures.
- C. Of all of the noble metals and alloys considered, the two thermoelements selected appear to have the best strength at 1450°C in air (as determined by stress-to-rupture tests).
- D. The EMF generated by the selected thermocouple is higher than that of all of the presently known standard noble metal thermocouples (ISA types R, S and B) that are capable of operating in air at 1450°C. Likewise, it has a higher sensitivity per degree Celsius, as for example 21.5 $\mu\text{V}/^\circ\text{C}$ at 1500°C, (Type R 14 $\mu\text{V}/^\circ\text{C}$, Type S 12 $\mu\text{V}/^\circ\text{C}$ and Type B 11.6 $\mu\text{V}/^\circ\text{C}$).
- E. Matched base metal compensating extension wires have been developed that generate an EMF which may approximate that of the selected thermocouple within ± 150 mv over the range of -30°C to 1000°C (Nichrome vs. EI Alloy 9R-682). Additional work is needed to determine how well these compensating extension wires can be reproduced from lot to lot. Based on test data from one melt only it appears that increasing the manganese content in 9R-682 from 0.50% to 0.70% helps to bring the thermocouple/extension wire EMF a little closer (see Alloy 9R-1522, Table 5).
- F. Of the noble metals and alloys available at this time for use as a thermocouple shield tube or thermocouple sheath, the oxide dispersion strengthened platinum appears to be the best. However, the platinum cermet material under study but not fully developed under this contract may offer another property that may be advantageous at elevated temperatures - abrasion resistance.
- G. A thermocouple probe design was developed during the course of this program. The successful completion of the vibration test is one indication that this probe may be used in the projected application. Only actual tests in the Allison GMA 200 Gas Turbine will prove whether the design is sound and the component materials will withstand the severe operating conditions.

9.0 REFERENCES

1. Glawe, George E., "New High-Temperature Noble-Metal Thermocouple Pairing, Rev. Sci. Instrum., Vol. 46, No. 8, August 1975
2. Moffat, Robert J., "Gas Temperature Measurement", Temperature Its Measurement and Control In Science and Industry, C. M. Herzfeld, Ed-in-Chief (Reinhold Publishing Corp., New York, 1962), Vol. 3, Part 2, p553.
3. Papadakis, E.P., K.A. Fowler, L.C. Lynnworth, A. Robertson and E. D. Zysk, "Ultrasonic Measurements of Young's Modulus and Extensional Wave Attenuation In Refractory Metal Wires at Elevated Temperatures With Application To Ultrasonic Thermometry", J. Appl. Phys., Vol. 45, No. 6, June 1975, p2409.
4. Albert, H. J. and J. S. Hill, "Development of a Platinum-Thorium Oxide Alloy For Resistojet Thruster Use", Final Report NASA CR-111959, July 1971.
5. Bedford, R.E., "Reference Tables For Platinum-40% Rhodium/Platinum-20% Rhodium Thermocouples, Rev. Sci. Instr. Vol. 36, No. 11, November 1965, p1571.

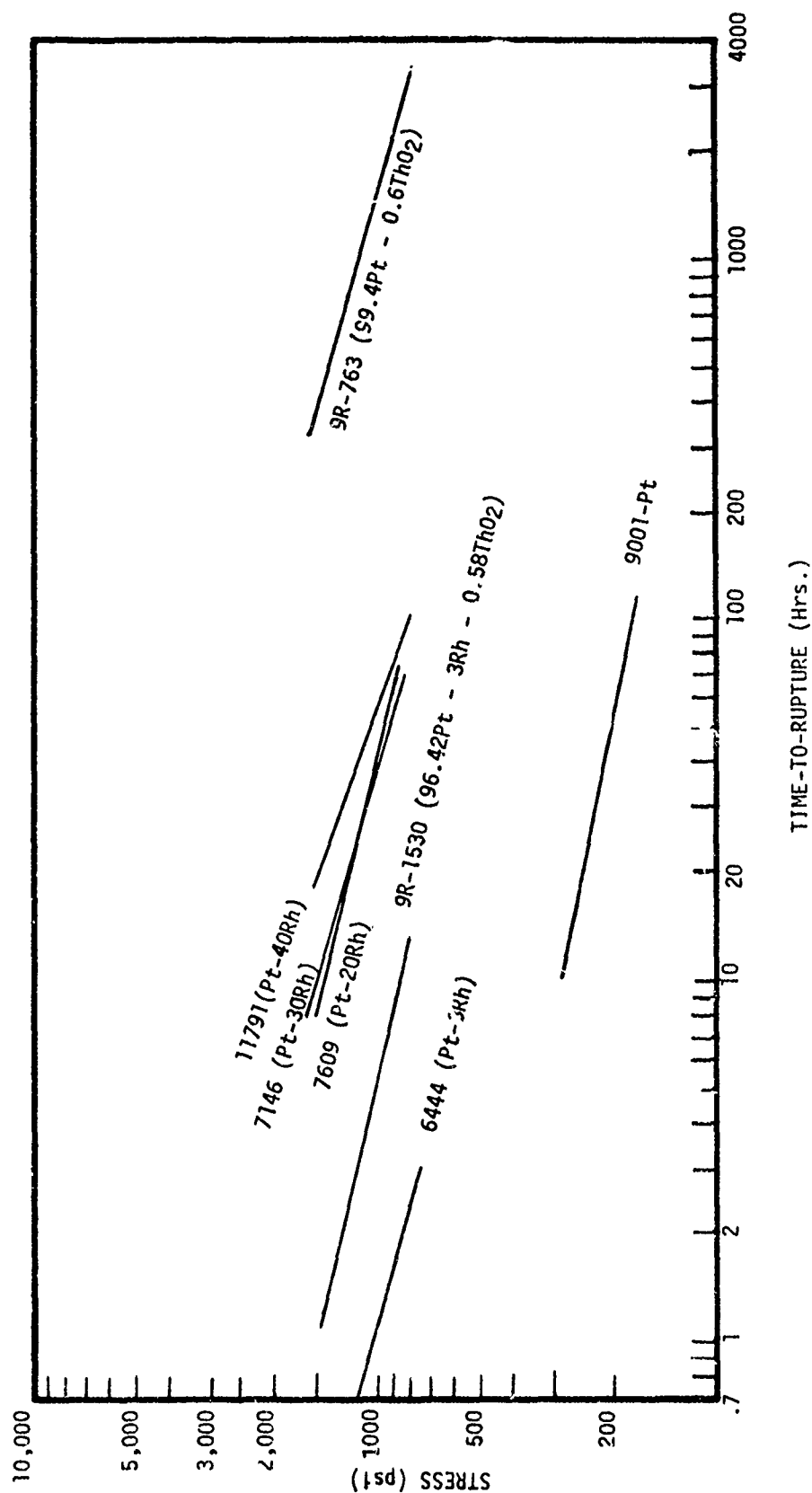
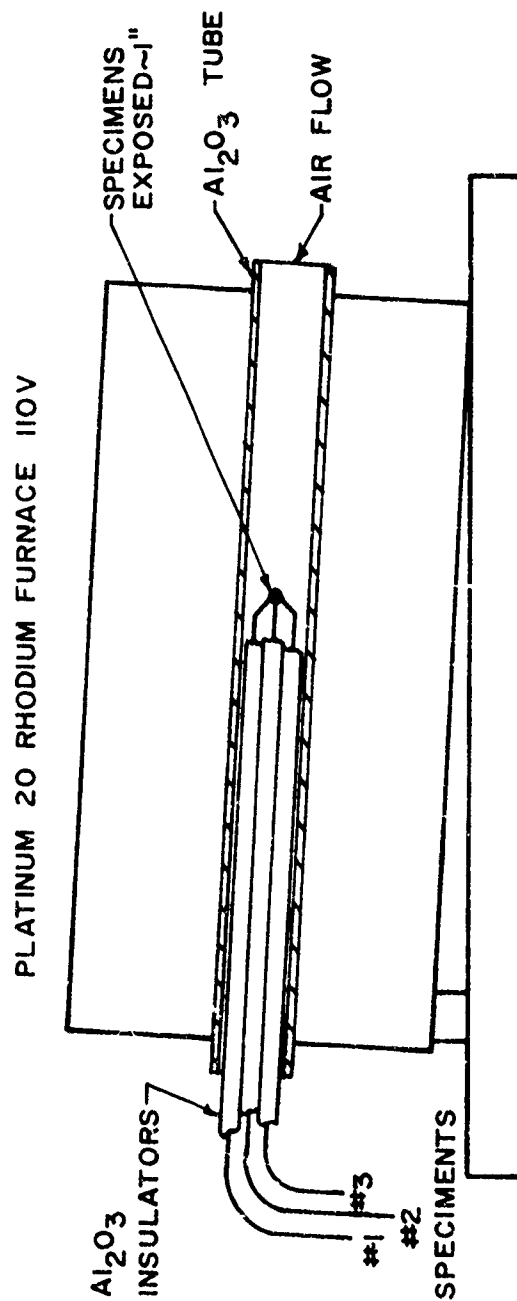


Figure 1 Stress vs. Time-To-Rupture For Pt, Pt-0.6%ThO₂, Pt-Rh-Thoria and Pt-Rh Alloys at 1450°C in air.

FIGURE 2
EMF STABILITY TEST EQUIPMENT



NOTES:

1. SAMPLES TESTED AS SHOWN IN AIR AT EITHER 1450°C OR 1650°C FOR 200 HOURS. CONTROL COUPLE Pt-6%Rh vs. Pt-30%Rh
2. I.D. OF Al_2O_3 MUFFLE TUBE IS 1/2".
3. FURNACE TILTED AS SHOWN TO PROMOTE AIR FLOW (CHIMNEY EFFECT).
4. THREE TEST SPECIMENS SHOWN WERE 9R-763 (99.4% Pt-0.6% ThO_2), EA16444 (Pt-3% Rh) AND EA11791 (Pt-40% Rh).

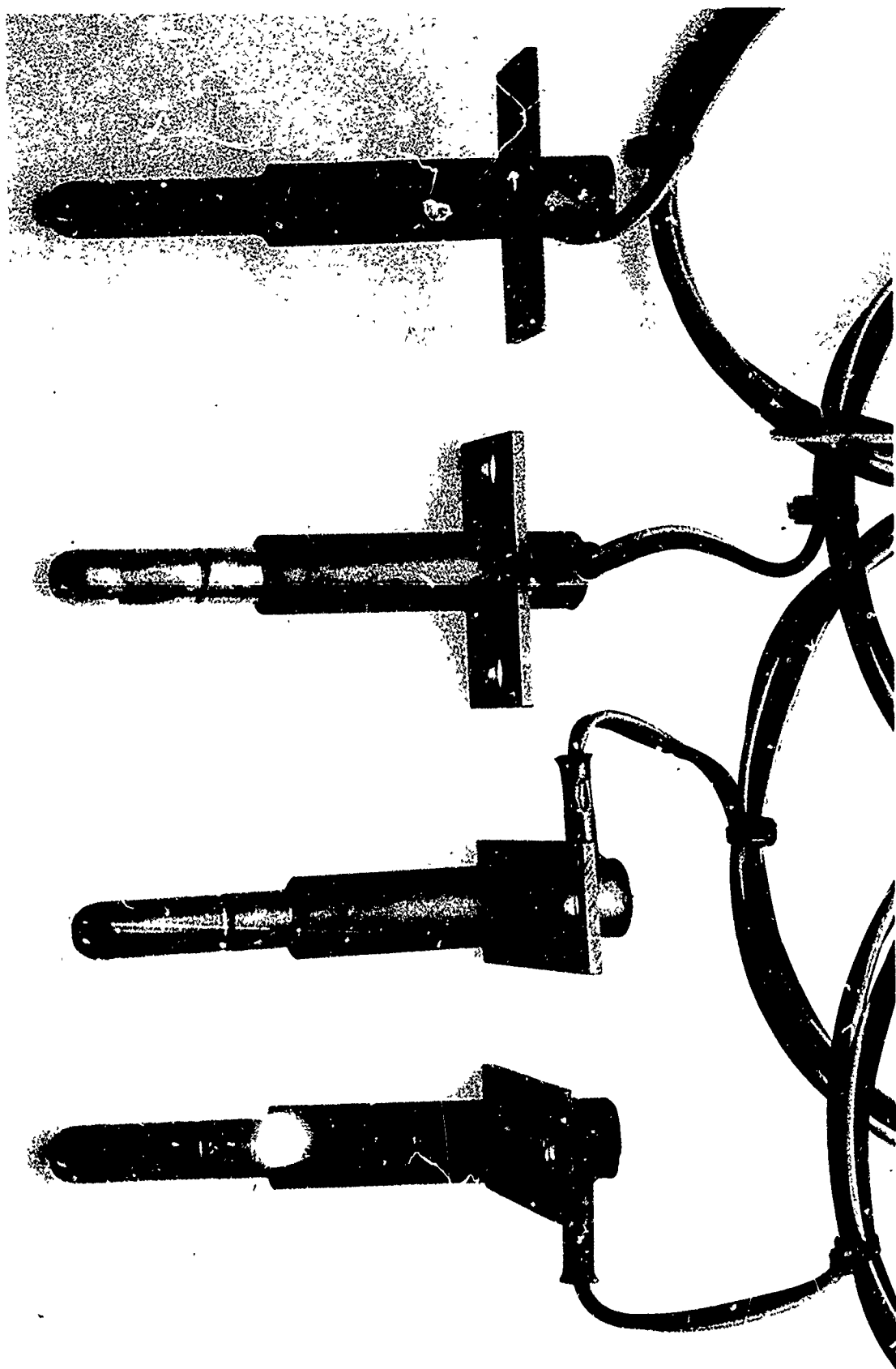


Figure 3 Film No. X1398-C Macro photograph Showing Four Sides of Finished Probe.

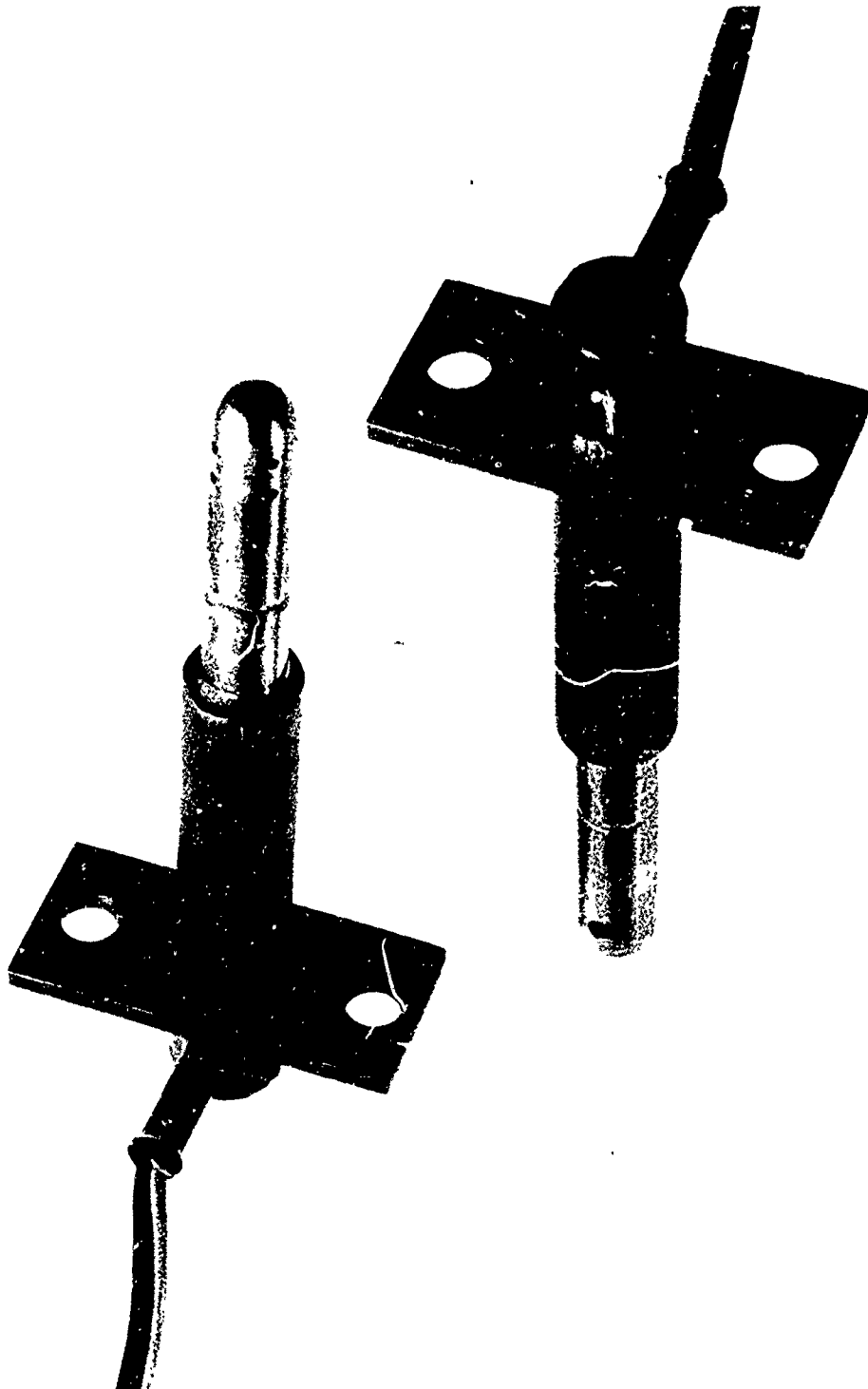


Figure 4 Film No. X1398-5 Top and Bottom View of Finished Probes.



Figure 5 Film No. 10511-A 50X

Longitudinal Section, Pt-0.6%ThO₂
.050" dia. wire, as worked condition
outside of stress-rupture furnace.
Same wire as in Fig. 6, lesser depth
of polish.



Figure 6 Film No. 10508-A 50X

Longitudinal Section, Pt-0.6%ThO₂
.050" dia. wire, within stress-rupture
furnace (~ 1200°C, 2117 hours), stress
900 psi.

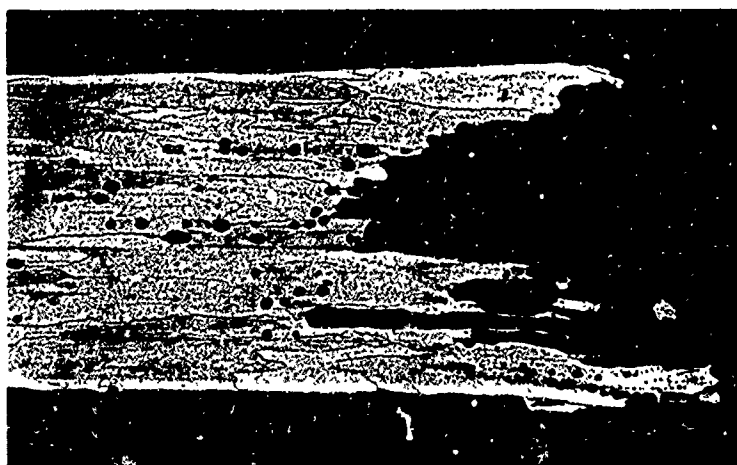


Figure 7 Film No. 10509-A 50X

Longitudinal section, fracture area of a Pt-0.6%ThO₂
alloy, .050" dia. wire, on test for 2117 hours at
1450°C in air with a stress of 900 psi.

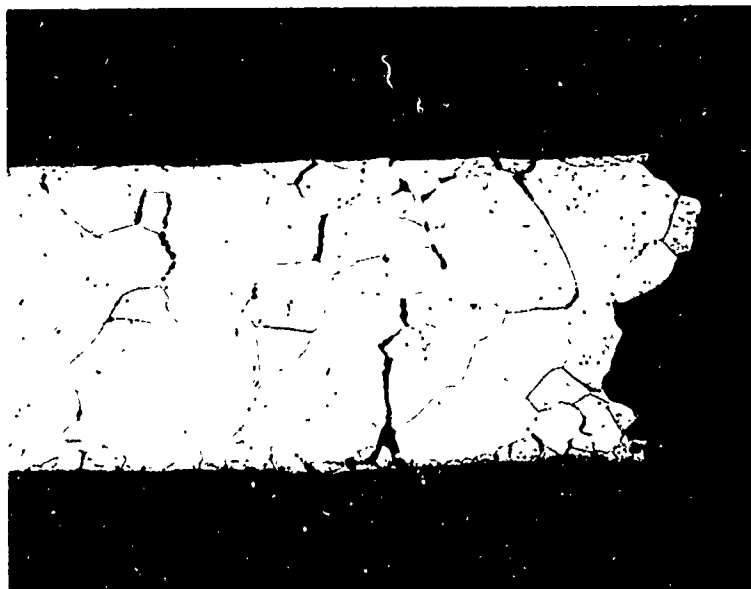


Figure 8 Film No. 10506-A Mag. 50X

Longitudinal section of the fracture area of a 60% Pt-40% Rh alloy, 0.050" diameter wire, on test for 73 hours at 1450°C in air with a stress of 900 psi.

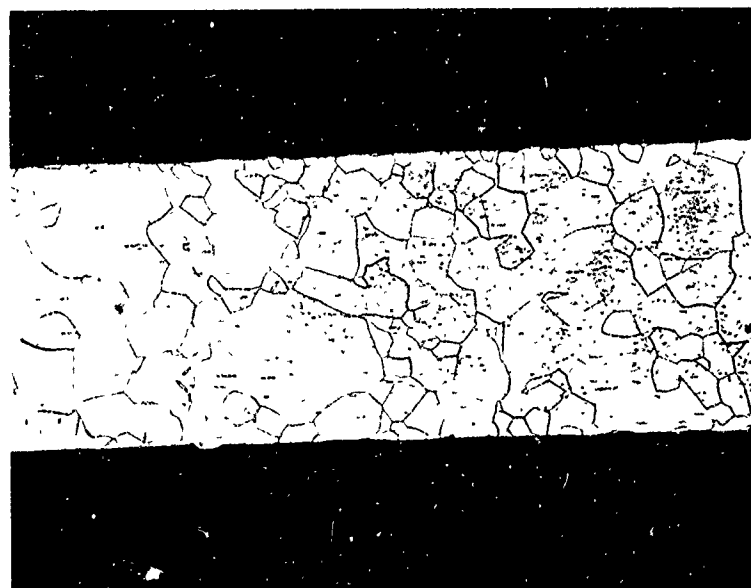


Figure 9 Film No. 10513-A Mag. 50X

Longitudinal section of a 60%Pt-40%Rh alloy, 0.050" diameter wire, annealed condition.

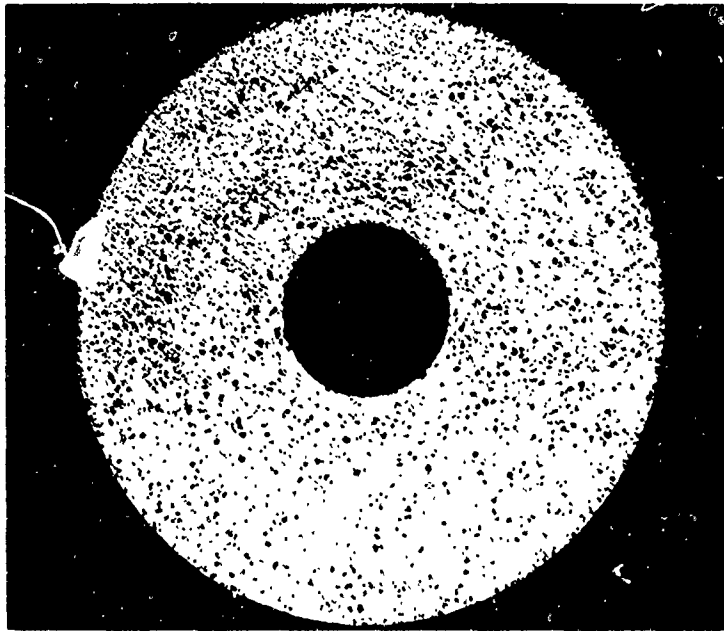


Figure 10 Film No. 10519-A Mag. 8X

Transverse section through EA 9R-1545 (95%Pt - 5%ThO₂) cermet, isostatically pressed at 30,000 psi and sintered for 1 hour at 800°C in vacuum followed by a sinter at 1600°C, 4 hours, vacuum 10⁻⁵ torr. Density of bar, 87.55% of theoretical.

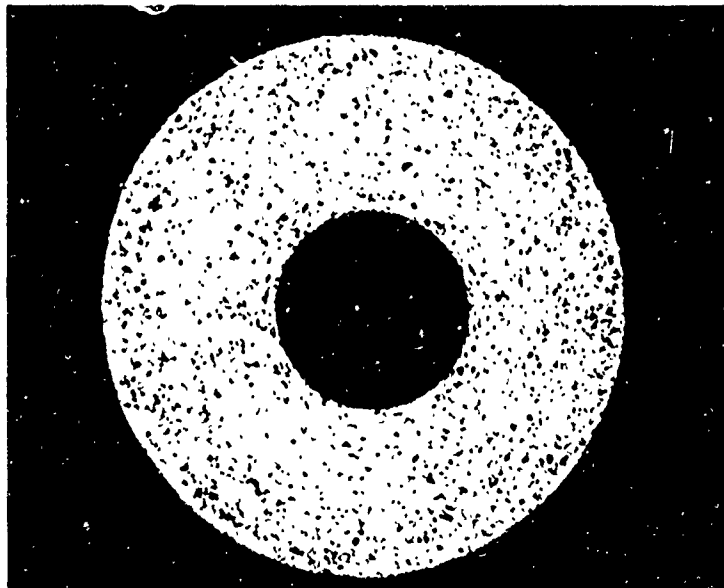


Figure 11 Film No. 10523-A Mag. 8X

Transverse section through EA 9R-1545 cermet, isostatically pressed at 30,000 psi and sintered 600°C, 1 hour, vacuum of 10⁻⁵ torr. Density 93.08% of theoretical.

TABLE 1

ENGELHARD
 ENGELHARD INDUSTRIES DIVISION
 ENGELHARD MINERALS & CHEMICALS CORPORATION

ANALYTICAL SERVICES DEPARTMENT
 P.O. BOX 2307
 NEWARK, NEW JERSEY 07114
 (201) 589-5000

CERTIFICATE OF SPECTROGRAPHIC ANALYSIS No. 9340

Sample Identification: Platinum Powder Lot # 03774529

Requested by: WPAFB

Report No: A 72571

Analysis: Quantitative

p.p.m.		p.p.m.	
Rh	ND	Ca	ND
Pd	6	Al	< 1
Ir	ND	N	< 1
Ru	ND	Cl	< 1
Ps	ND	Mn	< 1
Au	6	Sb	ND
Ag	4	B	ND
Pb	ND	Co	ND
Sn	ND	As	ND
Zn	ND	Bi	ND
Fe	ND	Cd	ND
Cu	< 1	Mo	ND
Si	17	Te	ND
Mg	ND	Na	5

* Pt 99.996%

ENGELHARD
 ENGELHARD INDUSTRIES DIVISION
 ENGELHARD MINERALS & CHEMICALS CORPORATION

By: *A. J. Lincoln*
 A. J. Lincoln
 Co-Director, Analytical Services Department

AJL/pc

Date: December 10, 1975

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

TABLE 2

EMF (millivolts) of Candidate Thermoelements Versus
Platinum 67 As A Function of Temperature (IPTS 68)

	Temperature °C		Reference Junction 0°C	
Thermoelement	11791	9R-1530	16444	9R-763
Test No.	{ 4016, 4017 4020	3710	3734	{ 4000 4002
Bar No.	9833	201815	201923	201813
T (°C)				
-50	-.216			-0.001
-40	-.180			0.000
30	-.140			0.000
20	-.099			0.000
10	-.053			0.000
0	0	0	0	0
25	+.140	+.103	.098	+0.001
50	.300	.211	.204	+0.002
75	.470	.325	.314	0.002
100	.659	.441	.426	0.003
150	1.073	.683	.659	0.004
200	1.533	.934	.901	0.005
250	2.033	1.187	1.145	0.007
300	2.567	1.444	1.391	0.008
350	3.133	1.700	1.639	0.009
400	3.732	1.957	1.883	0.010
500	5.052	2.465	2.372	0.012
600	6.393	2.973	2.860	0.014
700	7.894	3.484	3.347	0.016
800	9.502	3.998	3.837	0.018
900	11.215	4.519	4.329	0.020
1000	13.030	5.040	4.830	0.022
1100	14.942	5.575	5.338	0.023
1200	16.935	6.110	5.850	0.026
1300	19.016	6.645	6.366	0.030
1400	21.135	7.179	6.865	0.031
1500	23.295	7.940	7.351	0.033
1600	25.459	8.424	7.821	0.034
1650	26.561	8.894	8.050	0.036

Alloys 11791 60%Pt-40%Rh
 9R-1530 96.42%Pt-3.00%Rh-0.58%ThO₂
 16444 97%Pt-3%Rh
 9R-763 99.4%Pt-0.6%ThO₂

TABLE 3

EMF (millivolts) of Candidate Extension Wire Versus
Platinum 67 As A Function of Temperature (ITS 68)

Temperature °C		Reference Junction 0°C	
Material	Nichrome	9R-567	Tophet D
Test No.		{ 3697 3937	3929
Bar No.	1695	175429	534919
<u>T (°C)</u>			
-50	-.262	-.326	-.127
40	.209	-.259	-.108
30	.155	-.191	.087
20	.104	-.127	.061
10	.050	-.063	.033
0	0	0	0
+25	+.181	+.150	+.101
50	+.391	.280	.223
75	.616	.394	.364
100	.867	.492	.519
150	1.418	.645	.877
200	2.038	.760	1.293
250	2.719	.885	1.759
300	3.454	1.073	2.269
350	4.232	1.315	2.824
400	5.058	1.576	3.416
500	6.831	2.134	4.728
600	8.765	2.725	6.195
700	10.876	3.352	7.833
800	13.161	4.020	9.641
900	15.611	4.738	11.627
1000	18.227	5.499	13.783

Nichrome - T.M. Driver-Harris Co. (60%Ni-15%Cr-24%Fe-1%Si-Trace Mn)

Tophet D - T.M. Wilbur B. Driver Co. (35%Ni-20%Cr-45%Fe)

9R-567 - Engelhard Industries (94.5Ni-4.5W)

TABLE 4
Effect of Small Additions of Mn, Al and Si on EMF of 9R-567
EMF of Each Alloy Versus Pt 67 (IPTS 68)

Temperature °C		EMF Millivolts						Reference Junction 0°C			
Additions		0.1%Mn	0.2%Mn	0.5%Mn	0.1%Al	0.2%Al	0.5%Al	0.1%Si	0.2%Si	0.5%Si	
Alloy No.	9R-567	9R-903	9R-904	9R-682	9R-1518	9R-1519	9R-1520	9R-905	9R-906	9R-1517	
Heat No.	175429	175354	175355	175356	175360	175361	175362	175357	175358	175359	
°C											
100	.492	.451	.398	.349	.388	.347	.161	.431	.373	.210	
200	.760	.681	.593	.532	.567	.507	.204	.659	.571	.330	
300	1.073	.955	.851	.822	.816	.751	.396	.953	.861	.609	
400	1.576	1.445	1.332	1.331	1.266	1.179	.745	1.445	1.338	1.039	
500	2.134	1.996	1.875	1.906	1.766	1.659	1.194	1.990	1.867	1.518	
600	2.725	2.555	2.427	2.493	2.296	2.179	1.583	2.590	2.433	2.045	
700	3.352	3.163	3.028	3.139	2.854	2.726	2.067	3.185	3.035	2.608	
800	4.020	3.804	3.662	3.831	3.459	3.321	2.573	3.842	3.683	3.229	
900	4.738	4.495	4.349	4.571	4.108	3.961	3.160	4.548	4.380	3.903	
1000	5.499	5.237	5.085	5.366	4.805	4.653	3.792	5.311	5.135	4.636	

9R-567 95.5Ni-4.5W
 9R-903 95.41Ni-4.49W-0.1Mn
 9R-904 95.31Ni-4.49W-0.2Mn
 9R-682 95.0Ni-4.5W-0.5Mn
 9R-1518 95.41Ni-4.49W-0.1Al
 9R-1519 95.31Ni-4.49W-0.2Al
 9R-1520 95.025Ni-4.478W-0.497Al
 9R-905 95.41Ni-4.49W-0.10Si
 9R-906 95.31Ni-4.49W-0.20Si
 9R-1517 95.025Ni-4.478W-0.497Si

TABLE 5

Effect of Small Additions of Mn, Al, W, Si and Mo on EMF of 9R-567

EMF of Each Alloy Versus Pt 67 (ITS 68)

Additions Alloy No. Heat No. Test No.	Temperature °C		EMF Millivolts						Reference Junction 0°C		
	None	Mn	Mn	Mn	Al & W	Al & W	Si & W	Si & W	Si & W	Mo	Mo
	9R-567 175429 3697 3937	9R-1521 175434 3712 3911	9R-1522 175435 3711 3912	9R-1523 175436 3713 3913	9R-1524 175437 3714 3914	9R-1525 175438 3716 3915	9R-1526 175439 3715 3916	9R-1527 175440 3717 3917	9R-1528 175441 3719 3918		
°C											
-50	-.326	-.227	-.223	-.158	-.208	-.208	-.214	-.361	-.408		
-40	-.259	-.179	-.175	-.123	-.164	-.164	-.168	-.284	-.326		
-30	-.191	-.132	-.127	-.090	-.123	-.121	-.125	-.214	-.241		
-20	-.127	-.086	-.082	-.079	-.081	-.081	-.082	-.138	-.158		
-10	-.063	-.041	-.041	-.028	-.039	-.041	-.039	-.070	-.081		
0	0	0	0	0	0	0	0	0	0		
25	.150	.103	.100	.063	.093	.073	.097	.171	.200		
50	.280	.191	.185	.114	.178	.176	.184	.328	.385		
75	.394	.265	.258	.151	.254	.248	.259	.468	.555		
100	.492	.325	.318	.179	.321	.312	.325	.592	.714		
150	.645	.417	.406	.211	.445	.423	.439	.806	.998		
200	.760	.494	.479	.247	.585	.538	.558	.987	1.258		
250	.885	.599	.584	.335	.783	.705	.727	1.184	1.540		
300	1.073	.786	.773	.485	1.032	.933	.956	1.446	1.882		
350	1.315	1.029	1.014	.663	1.310	1.190	1.215	1.756	2.266		
400	1.576	1.295	1.279	.859	1.609	1.465	1.492	2.081	2.675		
500	2.134	1.864	1.849	1.268	2.266	2.062	2.095	2.781	3.547		
600	2.725	2.473	2.456	1.759	2.992	2.713	2.751	3.531	4.477		
700	3.352	3.121	3.105	2.275	3.781	3.421	3.461	4.326	5.467		
800	4.020	3.815	3.801	2.845	4.640	4.192	4.237	5.172	6.519		
900	4.738	4.561	4.549	3.472	5.571	5.027	5.073	6.073	7.633		
1000	5.499	5.360	5.349	4.152	6.568	5.925	5.975	7.026	8.787		
9R-567	95.5Ni-4.5W										
9R-1521	94.931Ni-4.473W-0.596Mn	9R-1523	94.088Ni-5.419W-0.493Al		9R-1526	94.648Ni-4.856W-0.496Si					
9R-1522	94.836Ni-4.469W-0.695Mn	9R-1524	93.904Ni-5.604W-0.492Al		9R-1527	95.31Ni-4.49W-0.20Mo					
		9R-1525	94.742Ni-4.762W-0.496Si		9R-1528	95.025Ni-4.478W-0.497Mo					

TABLE 6
Effect of Small Additions of Mn, Si and W on EMF of 9R-567
EMF of Each Alloy Versus Pt67 (IPTS 68)

Additions Alloy No. Heat No. Test No.	Temperature °C		EMF Millivolts				Reference Junction 0°C		
	Si & W 9R-1536 175491 3821	Si & W 9R-1537 175493 3822	Mn 9R-1538 175493 3823	Mn 9R-1539 175494 3824	Mn 9R-1540 195495 3825	Mn & W 9R-1541 175496 3826	None 9R-567 175429 3697 3937		
°C									
-50	.260	.232	.239	.231	.128	.241	-.326		
-40	.214	.190	.196	.190	.106	.200	-.259		
-30	.165	.147	.150	.146	.083	.152	-.191		
-10	.056	.030	.053	.051	.031	.106	-.127		
0	0	0	0	0	0	0	0		
25	.145	.127	.138	.132	.076	.137	.150		
50	.276	.243	.256	.244	.137	.255	.280		
75	.389	.342	.357	.339	.188	.359	.394		
100	.485	.423	.444	.421	.228	.446	.492		
150	.640	.566	.576	.544	.287	.582	.645		
200	.766	.686	.680	.640	.342	.691	.760		
250	.904	.829	.797	.752	.438	.815	.885		
300	1.110	1.040	.984	.937	.629	1.008	1.073		
350	1.363	1.294	1.225	1.175	.872	1.255	1.315		
400	1.638	1.567	1.486	1.437	1.139	1.524	1.576		
500	2.221	2.152	2.045	1.992	1.715	2.098	2.134		
600	2.843	2.778	2.639	2.583	2.332	2.709	2.725		
700	3.509	3.452	3.268	3.213	2.995	3.360	3.352		
800	4.220	4.175	3.941	3.882	3.709	4.052	4.020		
900	4.980	4.955	4.664	4.604	4.478	4.800	4.738		
1000	5.793	5.782	5.429	5.373	5.296	5.598	5.499		

9R-567 95.5Ni-4.5W
9R-1536 94.555Ni-4.950W-0.495Si
9R-1537 94.461Ni-5.044W-0.495Si
9R-1538 94.742Ni-4.464W-0.794Mn
9R-1539 94.648Ni-4.460W-0.892Mn
9R-1540 94.555Ni-4.455W-0.990Mn
9R-1541 94.695Ni-4.611W-0.694Mn

TABLE 7

Effect of Small Additions of Mn and Si On EMF of 9R-567

EMF of Each Alloy Versus Pt 67 (IPTS 68)

Additions Alloy No. Heat No. Test No.	Temperature °C				EMF Millivolts					Reference Junction 0°C				
	None	Mn	Mn	Mn	Mn	Mn	Mn	Mn	Si	Si	Si	Mn & Si	Mn	
	9R-567 175449 3854	9R-1558 175497 3855	9R-1559 175498 3857	9R-1560 175499 3858	9R-1564 39251 3862	9R-1565 39252 3864	9R-1557 175450 3856	9R-1561 175500 3861	9R-1566 39253 3863	9R-682 175379 1932				
°C														
25	.147	.102	.105	.110	.075	.064	.096	.075	.095					
50	.276	.187	.196	.206	.136	.119	.180	.145	.182					
75	.386	.261	.275	.288	.185	.161	.256	.209	.261					
100	.482	.323	.341	.358	.227	.190	.325	.265	.332	.348				
150	.626	.423	.448	.474	.289	.246	.453	.379	.470	.530				
200	.737	.516	.547	.577	.352	.302	.595	.524	.627	.530				
250	.856	.643	.679	.716	.463	.406	.798	.732	.845	.822				
300	1.040	.855	.895	.938	.666	.606	1.053	.983	1.118					
350	1.288	1.120	1.163	1.209	.920	.860	1.339	1.263	1.425					
400	1.536	1.407	1.452	1.506	1.499	1.137	1.647	1.565	1.753	1.329				
500	2.087	2.024	2.076	2.139	1.797	1.734	2.312	2.222	2.462	1.895				
600	2.668	2.685	2.744	2.820	2.444	2.373	3.032	2.942	3.237	2.499				
700	3.285	3.395	3.459	3.577	3.140	3.060	3.822	3.735	4.078	3.149				
800	3.942	4.157	4.226	4.326	3.887	3.804	4.678	4.601	4.993	3.834				
900	4.645	4.971	5.049	5.162	4.687	4.595	5.602	5.541	5.977	4.572				
1000	5.396	5.838	5.928	6.050	5.538	5.446	5.598	6.547	7.033	5.367				

9R-567 95.5Ni-4.5W
 9R-1558 94.752Ni-4.554W-0.69Mn
 9R-1559 94.733Ni-4.573W-0.694Mn
 9R-1560 94.714Ni-4.592W-0.694Mn
 9R-1564 94.592Ni-4.451W-0.951Mn
 9R-1565 94.573Ni-4.455W-0.971Mn
 9R-1557 94.414Ni-5.092W-0.494Si
 9R-1561 94.275Ni-5.133W-0.592Si
 9R-1566 94.27Ni-5.035W-0.197Mn-0.494Si
 9R-682 95.0Ni-4.5W-0.5Mn

TABLE 8

Effect of Small Additions of Mn on EMF of 9R-567

EMF of Each Alloy Versus Pt 67 (IPTS 68)

Temperature °C	EMF Millivolts		Reference Junction 0°C
Additions Alloy No. Heat No. Test No.	Mn 9R-682 175399 3954-3951	Mn 9R-1562 39258 3952-3950	Mn 9R-1563 35259 3953-3949
°C			
-50	-.243	-.186	-.185
-40	-.191	-.145	-.145
-30	-.142	-.108	-.107
-20	-.092	-.069	-.069
-10	-.045	-.033	-.035
0	0	0	0
25		+.084	+.089
50		.155	.165
75		.214	.227
100	+.367	.260	.278
150		.331	.357
200	.565	.395	.427
250		.498	.535
300	.868	.691	.733
350		.938	.982
400	1.380	1.209	1.255
500	1.952	1.785	1.840
600	2.561	2.410	2.467
700	3.210	3.073	3.144
800	3.906	3.788	3.865
900	4.650	4.555	4.640
1000	5.440	5.377	5.468

9R-567 95.5Ni-4.5W
 9R-682 95.0Ni-4.5W-0.5Mn
 9R-1562 94.629Ni-4.459W-0.912Mn
 9R-1563 94.611Ni-4.458W-0.931Mn

TABLE 9

Wanted EMF vs. Pt67 For Negative Leg Compensating Extension Wire
To Match Nichrome or Tophet D

T (°C)	Temperature °C (IPTS 68)		EMF Millivolts				Reference Junction °C	
	Pt-40%Rh vs. Pt-0.6%ThO ₂	Nichrome (+) vs. Pt67	Wanted (-) Leg vs. Pt67	Pt-40%Rh vs. Pt-3%Rh	Tophet D (+) vs. Pt67	Wanted (-) Leg vs. Pt67		
-50	-0.216	-0.262	-0.046					
-40	-0.180	-0.209	-0.029					
-30	-0.140	-0.155	-0.015					
-20	-0.090	-0.144	-0.054					
-10	-0.052	-0.050	+0.002					
0	0	0	0	0	0	0		
+25	+0.138	+0.181	+0.043	+0.060	+0.101	+0.041		
50	0.298	0.391	0.093	+0.120	0.223	0.103		
75	0.466	0.616	0.150	0.180	0.364	0.184		
100	0.654	0.867	0.213	0.240	0.519	0.279		
200	1.526	2.038	0.512	0.640	1.293	0.653		
300	2.558	3.454	0.896	1.180	2.269	1.089		
400	3.720	5.058	1.338	1.850	3.416	1.566		
500	4.988	6.831	1.843	2.640	4.728	2.089		
600	6.376	8.765	2.389	3.550	6.195	2.645		
700	7.871	10.876	3.005	4.560	7.833	3.273		
800	9.475	13.161	3.686	5.680	9.641	3.961		
900	11.178	15.611	4.433	6.900	11.627	4.727		
1000	12.985	18.227	5.242	8.220	13.783	5.563		

NOTES:

1. EMF vs. Pt67 values for Nichrome (+) bar #1695 and Tophet D (+) bar #534919 were used to determine wanted (-) EMF vs. Pt67. The calculated wanted (-) leg values were the targets during this program.
2. Tophet D is (+) leg of compensating extension wire for Pt-40%Rh vs. Pt-3%Rh couple and Nichrome is (-) leg of compensating extension wire used on Pt-40%Rh vs. Pt-0.6%ThO₂ couple.

TABLE 10
Stress-Rupture Test Results of Platinum And
Platinum-Rhodium Alloys

<u>Alloy No.</u>	<u>Composition</u>	<u>Load - PSI</u>	<u>Avg. Life - Hours</u>
16444	Pt-3%Rh	1350	0.2
		1120	0.7
		900	1.6
9R1530	96.42%Pt, 3%Rh, 0.58% ThO ₂	1350	1.5
		1120	3.5
		900	7.7
7609	Pt-20%Rh	1350	14.0
		1120	26.8
		900	53.1
7146	Pt-30%Rh	1350	12.8
		1120	25.5
		900	69.2
11791	Pt-40Rh	1350	24.6
		1120	39.6
		900	72.5
9R763	Pt-0.6% ThO ₂	1350	506.0
		900	2117.0
9001 *	Pt	275	12.0
		225	30.0
		175	100.0

* Data from previous tests - note the small loads

NOTES

1. Wire .050" diameter.
2. Load
800 grams (900 psi)
1000 grams (1120 psi)
1200 grams (1350 psi)
3. Test temperature 1450 ± 10° Celsius
4. Each point reported (load and time to rupture) is an average of three tests. Alloy 9R-763 is an exception to this rule -- because of limited material, only one sample was run at a load of 800 grams (900 psi) and one at a load of 1200 grams (1350 psi).

TABLE 11

High Temperature EMF Stability Test In Air

Alloy 11791 (Pt-40%Rh), .020" Diameter

EMF vs. Pt67

Reference Junction 0°C

EMF, Millivolts

Test No.	3707	3840		3341	
Temp. °C	Before Stability Test EMF	After 200 Hrs. at 1450°C EMF	* Difference EMF	After 200 Hrs. at 1650°C EMF	* Difference EMF
0	0	0	0	0	0
25	+.138	+.143	+.005	+.143	+.005
50	.296	.300	.004	.300	.004
75	.470	.472	.002	.472	.002
100	.656	.653	.004	.659	.003
150	1.069	1.075	.006	1.074	.005
200	1.529	1.536	.007	1.535	.006
250	2.027	2.036	.009	2.035	.008
300	2.564	2.573	.009	2.572	.008
350	3.130	3.138	.008	3.138	.008
400	3.724	3.736	.012	3.735	.011
500	5.003	5.013	.010	5.012	.009
600	6.390	6.405	.015	6.404	.014
700	7.890	7.907	.017	7.905	.015
800	9.502	9.512	.010	9.510	.008
900	11.222	11.229	.007	11.227	.005
1000	13.041	13.048	.007	13.046	.005
1100	14.959	14.966	.007	14.964	.005
1200	16.960	16.960	0	16.958	-.002
1300	19.030	19.025	-.005	19.023	-.007
1400	21.135	21.133	-.002	21.131	-.004
1500	23.247	23.255	+.008	23.253	+.006
1600	25.379	25.385	.006	25.383	.004
1650	26.445	26.450	.005	26.448	.003

* Difference EMF obtained by subtracting "Before Stability Test EMF" from "After Stability Test EMF".

TABLE 12
High Temperature EMF Stability Test In Air
Alloy 16444 (Pt-3% Rh), .020" Diameter
EMF vs. Pt67

Reference Junction 0°C

EMF, Millivolts

Test No.	3707	3840		3841	
Temp. °C	Before Stability Test EMF	After 200 Hrs. at 1450°C EMF	* Difference EMF	After 200 Hrs. at 1650°C EMF	* Difference EMF
0	0	0	0	0	0
25	+.098	+.104	+.006	+.104	+.006
50	.204	.213	.009	.213	.009
75	.314	.322	.008	.322	.008
100	.426	.435	.009	.435	.009
150	.659	.666	.007	.666	.007
200	.901	.908	.007	.907	.006
250	1.145	1.153	.008	1.152	.007
300	1.390	1.398	.008	1.397	.007
350	1.638	1.644	.006	1.643	.005
400	1.881	1.888	.007	1.887	.006
500	2.370	2.374	.004	2.373	.003
600	2.857	2.861	.004	2.860	.003
700	3.343	3.350	.007	3.350	.007
800	3.832	3.837	.005	3.837	.005
900	4.323	4.333	.010	4.333	.010
1000	4.823	4.829	.006	4.829	.006
1100	5.329	5.335	.006	5.335	.006
1200	5.839	5.841	.002	5.841	.002
1300	6.351	6.352	.001	6.353	.002
1400	6.848	6.846	-.002	6.852	.004
1500	7.334	7.329	-.005	7.335	.001
1600	7.804	7.797	-.007	7.803	-.001
1650	8.262				

* "Difference EMF obtained by subtracting "Before Stability Test EMF" from "After Stability Test EMF".

TABLE 13

High Temperature EMF Stability Test In Air
 Alloy 9R-763 (99.4%Pt-0.6%ThO₂), .020" Diameter
 EMF vs. Pt67

Reference Junction 0°C

EMF, Millivolts

Test No.	3818	3838		3839	
Temp. °C	Before Stability Test EMF	After 200 Hrs. at 1450°C EMF	* Difference EMF	After 200 Hrs. at 1650°C EMF	* Difference EMF
0	0	0	0	0	0
25	+.002	+.004	+.002	+.004	+.002
50	.002	.012	.010	.007	.005
75	.003	.015	.012	.011	.008
100	.003	.015	.012	.011	.008
150	.005	.016	.011	.014	.009
200	.006	.016	.010	.016	.010
250	.007	.016	.009	.017	.010
300	.008	.018	.010	.019	.011
350	.010	.019	.009	.021	.011
400	.011	.020	.009	.023	.012
500	.013	.024	.011	.027	.014
600	.014	.027	.013	.031	.017
700	.016	.030	.014	.035	.019
800	.018	.033	.015	.039	.021
900	.020	.037	.017	.044	.024
1000	.022	.039	.017	.047	.025
1100	.023	.042	.019	.050	.027
1200	.025	.044	.019	.053	.028
1300	.028	.048	.020	.058	.030
1400	.031	.051	.020	.061	.030
1500	.034	.052	.018	.060	.026
1600	.035	.054	.019	.061	.026
1650	.036	.056	.020	.062	.026

* "Difference EMF" obtained by subtracting "Before Stability Test EMF" from "After Stability Test EMF".

TABLE 14

EMF Stability Test In Air

Alloy Tophet D (Bar #534319), .040" Diameter

EMF vs. Pt67

Reference Junction 0°C

EMF, Millivolts

Test No.	3815	3837*		3849**	
Temp. °C	Before Stability Test EMF	After 200 Hrs. at 850°C EMF	Difference EMF	After 200 Hrs. at 850°C EMF	Difference EMF
0	0	0	0	0	0
25	+.102	+.102	0	+.102	0
50	.223	.225	+.002	.225	+.002
75	.362	.368	.006	.366	.004
100	.519	.526	.007	.523	.004
150	.877	.890	.013	.884	.007
200	1.292	1.310	.018	1.301	.009
250	1.755	1.782	.027	1.771	.016
300	2.266	2.298	.032	2.286	.020
350	2.821	2.856	.035	2.842	.021
400	3.414	3.456	.042	3.436	.022
500	4.728	4.775	.047	4.752	.024
600	6.193	6.259	.066	6.221	.028
700	7.830	7.899	.069	7.855	.015
800	9.640	9.715	.075	9.664	.024
900	11.625	11.705	.080	11.647	.022
1000	13.781	13.864	.083	13.801	.020

* Re-calibrated as tested condition.

** Re-calibrated after cutting off head and 1" off hot end.

Table 15
EMF Stability Test In Air
Alloy Nichrome (Bar #1695), .040" Diameter
EMF vs. Pt67

Reference Junction 0°C			EMF, Millivolts		
Test. No.	3816	3836*		3850**	
Temp. °C	Before Stability Test EMF	After 200 Hrs. at 850°C EMF	Difference EMF	After 200 Hrs. at 850°C EMF	Difference EMF
0	0	0	0	0	0
25	+.181	+.180	-.001	.182	.001
50	.386	.386	0	.388	.002
75	.616	.615	-.001	.615	-.001
100	.862	.867	+.005	.866	.004
150	1.415	1.418	.003	1.420	.005
200	2.036	2.041	.005	2.044	.008
250	2.717	2.726	.009	2.727	.010
300	3.451	3.463	.012	3.462	.011
350	4.234	4.250	.016	4.245	.011
400	5.062	5.083	.021	5.078	.016
500	6.855	6.891	.036	6.862	.007
600	8.793	8.829	.036	8.805	.012
700	10.918	10.952	.034	10.921	.003
800	13.201	13.254	.053	13.218	.017
900	15.663	15.710	.047	15.674	.011
1000	18.287	18.332	.045	18.285	-.002

* Re-calibrated, as tested condition

*** Re-calibrated, after cutting off bead and 1" off hot end.

TABLE 16
EMF Stability Test In Air
Alloy 9R-567 (Bar #175446), .040" Diameter
EMF vs. Pt67

Reference Junction 0°C

EMF, Millivolts

Test. No.	3805	3835*		3851**	
Temp. °C	Before Stability Test EMF	After 200 Hrs. at 850°C EMF	Difference EMF	After 200 Hrs. at 850°C EMF	Difference EMF
0	0	0	0	0	0
25		.150		.149	
50		.255		.270	
75		.348		.369	
100	.490	.439	-.051	.454	-.036
150		.526		.592	
200	.763	.603	-.160	.694	-.069
250		.685		.807	
300	1.080	.836	-.244	.985	-.095
350		1.068		1.241	
400	1.595	1.349	-.246	1.516	-.079
500	2.161	1.898	-.263	2.047	-.114
600	2.762	2.401	-.361	2.603	-.159
700	3.400	2.927	-.473	3.206	-.194
800	4.076	3.553	-.523	3.814	-.262
900	4.801	4.126	-.675	4.528	-.273
1000	5.572	4.800	-.772	5.282	-.290

* Re-calibrated, as tested condition

** Re-calibrated, after cutting off bead and 1" off hot end.

TABLE 17
EMF Stability Test In Air
Alloy 9R-567 (Bar #175194) Wire .040" Diameter
EMF vs. Pt67

EMF, Millivolts

Reference Junction 0°C

Test No.	0906	0945	0946	0947	0955	0961	0956	Difference
Temp. °C		Difference	Difference	Difference	Difference	Difference	Difference	Difference
100	.471	.478	.476	.484	.477	.445	.243	-.228
200	.722	.734	.737	.744	.723	.677	.334	-.388
300	1.024	1.038	1.043	1.051	1.019	.959	.421	-.543
400	1.532	1.550	1.555	1.561	1.517	1.446	.890	-.652
500	2.092	2.113	2.117	2.120	2.066	1.990	1.355	-.737
600	2.691	2.711	2.715	2.716	2.647	2.561	1.812	-.879
700	3.323	3.340	3.345	3.345	3.262	3.163	2.310	-1.013
800	3.985	4.009	4.014	4.011	3.915	3.800	2.834	-1.151
900	4.698	4.719	4.724	4.719	4.613	4.478	3.388	-1.310
1000	5.459	5.483	5.488	5.478	5.354	5.202	3.985	-1.474

Test #906 Before Stability Test
#945 Heated 24 hours at 750°C in air.
#946 Heated 24 hours at 800°C in air.
#947 Heated 24 hours at 850°C in air.
#955 Heated 24 hours at 900°C in air.
#961 Heated 24 hours at 950°C in air.
#956 Heated 24 hours at 1000°C in air.

EMF Stability Test in Air
Alloy 9R-567 .034" Diameter

EMF vs. Pt67

EMF, millivolts

Reference Junction 0°C

Bar # Test #	Temp. °C	Before Stability Test EMF	After 200 Hrs. at 750°C EMF	Difference EMF
39254 3899	0	0	0	0
	100	+ .496	+ .489	.007
	200	.765	.753	.012
	300	1.080	1.061	.019
	400	1.579	1.570	.007
	500	2.136	2.125	.009
	600	2.724	2.713	.011
	700	3.347	3.335	.012
	800	4.012	3.998	.014
	900	4.722	4.702	.020
	1000	5.482	5.456	.026

9R-567 95.5Ni, 4.5W

NOTE: Test wire was loosely inserted into .082" I.D. high purity Al_2O_3 (99.5%+) open end tube. Tip of wire was approximately 1/2" from end of tube. The tube with the wire was then inserted into a furnace muffle (open ended tube about 5/8" I.D.). The furnace containing the test assembly was "tilted" 1" to promote a draft through the muffle tube. Temperature was maintained within the muffle at $750^{\circ}C \pm 10^{\circ}C$.

TABLE 19

Ledoux & Company
EST. 1900

CABLE ADDRESS "LEDOUX TEANECK"
TELEX 134340

DM



359 Alfred Avenue, Teaneck, New Jersey 07666 • Telephones: 201 837 7160
212 947 0953

INDEPENDENT CONTROL, AND RESEARCH CHEMISTRY, INSTRUMENTAL AND CHEMICAL ANALYSIS • SAMPLING,
WEIGHING, SHIPPERS' REPRESENTATION, BENEFICIATION, AND STORAGE OF ORES AND METALS

REPORT OF ANALYSIS

No. 956633

October 17, 1975

Date Received: 10/2/75

Our analysis of the sample of MgO Insulators

From Engelhard Industries

Marked: PO#90205

and submitted to us, shows:

BY SPECTROGRAPHIC ANALYSIS

ALUMINUM-----	0.1%
BORON-----	0.002%
CALCIUM-----	0.1%
CHROMIUM-----	0.003%
COPPER-----	0.001%
IRON-----	0.04%
MAGNESIUM-----	High
MANGANESE-----	0.003%
MOLYBDENUM-----	0.001%
NICKEL-----	0.005%
SILICON-----	0.1%
TITANIUM-----	0.003%
ZIRCONIUM-----	0.005%

Note: Other elements not detected

To Engelhard Industries

LEDOUX & COMPANY

High 10—100% Medium 1—10% Low 0.1—1% Trace less than 0.1%

TABLE 20

EMF Reproducibility of Negative Leg Thermoelement
 EMF of Alloy 9R-763 (Pt-0.6% ThO₂) Vs. Pt 67 (ITS 68)

Temperature °C		EMF Millivolts				Reference Junction 0°C	
Bar # Test #	Bar A 3708	201813 3818	Bar B 3844	Bar C 4000 4002	Bar D 3999 4001	Bar E 3708	Bar F 2924
T (°C)							
-50				-.001	-.009		
-40				0	-.007		
-30				0	-.007		
-20				0	-.005		
-10				0	-.005		
0				0	0		
+25		+.001	+.006	+.001	.000		
50		+.002	+.006	.002	+.003		
75		+.002	+.008	.002	.006		
100	+.003	+.003	+.009	.003	.009	+.016	
150				.004	.013		
200	+.006	+.006	+.010	.005	.018	+.030	
250				.007	.024		
300	+.007	+.008	+.013	.008	.028	+.045	
350				.009	.033		
400	+.010	+.011	+.016	.010	.037	.057	
500	+.013	+.013	+.019	.012	.046	.072	+.016
600	+.016	+.014	+.022	.014	.054	.085	
700	+.018	+.016	+.024	.016	.062	.096	
800	+.021	+.018	+.027	.018	.070	.112	
900	+.024	+.020	+.031	.020	.078	.121	
1000	+.026	+.022	+.033	.022	.086	.132	
1100	+.028	+.024	+.036	.023	.093	.143	
1200	+.030	+.026	+.039	.026	.102	.154	+.029
1300	+.032	+.030	+.046	.030	.117	.166	
1400	+.034	+.034	+.049	.031	.126	.176	
1500	+.034	+.035	+.055	.033	.135		
1600	+.036			.034	.147		
1650				.036	.151		

1. Powder Lot #05774529, Bars A and B and Bar 201813 fabricated from this lot.
2. Powder Lot #05774539, Bar C fabricated from this lot. Used for stress to rupture tests and in finished probes.
3. Powder Lot #05774537, Bar D fabricated.
4. Bar E fabricated from commercial lot of platinum powder.
5. Bar F values determined in previous work.

TABLE 21
EMF Reproducibility of Positive Leg Thermoelement
EMF of Pt-40%Rh Vs. Pt-67 (ITS 68)

Bar # Test #	52466 0979	74300 2438	9833 4016 4017 4020	8833 3840	3707	202132 4040 4041 4042
T (°C)						
-50			-.216			-0.211
40			-.180			-0.169
30			-.140			-0.136
20			-.099			-0.092
-10			-.053			-0.048
0			0			0
+25			+.140	+.143		+.136
50			.300	+.300	+0.274	.293
75			.470	+.472		.464
100	+.662	+.660	.659	+.660	+0.656	.649
150			1.073			1.058
200	1.537	1.535	1.533	1.536	1.529	1.516
250			2.033			2.013
300	2.563	2.571	2.567	2.577	2.564	2.543
350			3.133			3.112
400	3.730	3.740	3.732	3.736	3.724	3.704
500	5.000	5.016	5.052	5.012	5.003	4.985
600	6.390	6.409	6.393	6.403	6.390	6.376
700	7.887	7.910	7.894	7.901	7.890	7.879
800	9.493	9.519	9.491	9.501	9.502	9.494
900	11.202	11.231	11.199	11.217	11.222	11.218
1000	13.013	13.044	13.008	13.026	13.041	13.050
1100	14.927	14.960	14.913	14.937	14.959	14.976
1200	16.927	16.959	16.901	16.926	16.960	16.982
1300	18.985	19.021	18.947	18.989	18.994	19.090
1400	21.061	21.119	21.093	21.081	21.095	21.272
1500	23.194	23.238	23.249	23.209	23.201	23.476
1600			25.409	25.335	25.329	25.689
1650			26.486	26.395	26.390	

1. Analysis #N45547 indicates 39.99%Rh present in Bar #9833.
This wire was used in finished probe.

TABLE 22
EMF Reproducibility of Alloy EA16444 (Pt-3%Rh)
EMF Vs. Pt-67 (ITS 68)

Temperature °C	EMF Millivolts		Reference Junction 0°C
Alloy No. Bar # Test #	16444 201921 3732	16444 201922 3733	16444 201923 3734
-50	.205	.204	.205
-40	.161	.160	.161
-30	.120	.119	.120
-20	.078	.079	.080
-10	.039	.038	.039
0	0	0	0
25	.098	.096	.098
50	.204	.201	.204
75	.314	.310	.314
100	.426	.419	.426
150	.659	.649	.659
200	.901	.886	.901
250	1.145	1.125	1.145
300	1.390	1.366	1.391
350	1.638	1.610	1.639
400	1.881	1.849	1.883
500	2.370	2.327	2.372
600	2.857	2.804	2.860
700	3.343	3.280	3.347
800	3.832	3.749	3.837
900	4.323	4.240	4.329
1000	4.823	4.728	4.830
1100	5.329	5.224	5.338
1200	5.839	5.724	5.850
1300	6.351	6.224	6.366
1400	6.848	6.710	6.865
1500	7.334	7.177	7.351
1600	7.804	7.628	7.821
1650	8.033	7.848	8.050

TABLE 23
EMF Reproducibility of Alloy 9R-567 (Ni-4%W)
EMF Vs. Pt-67 (IPTS 68)

Temperature °C		EMF Millivolts		Reference Junction 0°C	
	Bar # Test #	175194 (standard)	195429 3690	175430 3691	
	T (°C)	EMF	EMF	EMF	
	100	.471	.492	.490	
	200	.722	.760	.755	
	300	1.024	1.073	1.068	
	400	1.532	1.576	1.573	
	500	2.092	2.134	2.132	
	600	2.691	2.725	2.724	
	700	3.323	3.352	3.353	
	800	3.985	4.020	4.023	
	900	4.698	4.738	4.740	
	1000	5.459	5.499	5.509	

TABLE 24

EMF Reproducibility of Nichrome

EMF Vs. Pt-67 (IPTS 68)

Temperature °C		EMF Millivolts		Reference Junction 0°C	
	Bar # Test #	B1852 3940	1695 4013	A1700 3939	A 2419
	T (°C)	EMF	EMF	EMF	EMF
	-50	-0.271	-0.262	-0.265	
	-40	-0.227	-0.209	-0.221	
	-30	-0.177	-0.155	-0.171	
	-20	-0.126	-0.104	-0.121	
	-10	-0.064	-0.050	-0.063	
	0	0	0	0	
	25	+0.172	+0.181	+0.168	
	50	+0.367	0.391	+0.359	
	75	0.585	0.616	0.573	
	100	0.824	0.867	0.806	0.839
	150	1.357	1.418	1.328	
	200	1.962	2.038	1.918	1.989
	250	2.623	2.719	2.565	
	300	3.342	3.454	3.267	3.373
	350	4.104	4.232	4.016	
	400	4.914	5.058	4.810	4.955
	500	6.655	6.831	6.525	6.695
	600	8.566	8.765	8.393	8.610
	700	10.638	10.876	10.435	10.697
	800	12.883	13.161	12.651	12.957
	900	15.299	15.611	15.029	15.382
	1000	17.900	18.227	17.566	17.977

Wire from Bar 1695 was used in finished probes.

TABLE 25
EMF Reproducibility of Alloy 9R-682
EMF Vs. Pt-67 (IPTS 68)

Temperature °C		EMF Millivolts			Reference Junction 0°C	
Bar # Test #	175399 3954 3951	175369 1851 1852	39260 3989 3995	39261 3990 3996	39262 3991 3997	175379 1932
T (°C)						
-50	-0.243	-0.250	0.252	-0.252	-0.251	
-40	-0.191	-0.202	0.206	-0.207	-0.205	
-30	-0.142	-0.149	0.154	-0.153	-0.152	
-20	-0.092	-0.097	0.100	-0.101	-0.100	
-10	-0.045	-0.048	0.047	-0.049	-0.048	
0	0	0	0	0	0	
+25		+0.110	+0.116	+0.114	+0.114	
50		0.206	0.216	0.213	0.211	
75		0.285	0.301	0.297	0.290	
100	+0.367	0.314	0.371	0.367	0.363	+0.348
150			0.480	0.475	0.467	
200	0.565	0.543	0.568	0.565	0.552	0.530
250			0.679	0.680	0.660	
300	0.868	0.838	0.870	0.875	0.848	0.822
350			1.112	1.122	1.090	
400	1.380	1.350	1.375	1.388	1.353	1.329
500	1.952	1.918	1.942	1.963	1.915	1.895
600	2.561	2.525	2.546	2.576	2.515	2.499
700	3.210	3.175	3.190	3.232	3.155	3.149
800	3.906	3.870	3.877	3.926	3.838	3.834
900	4.650	4.613	4.618	4.676	4.578	4.572
1000	5.440	5.398	5.409	5.474	5.365	5.367

Wire from Bar #39262 was used in finished probes.

TABLE 26

EMF Reproducibility of Alternate Extension Wire Positive Leg
 EMF Tophet C Vs. Pt67 (IPTS 68)

Temperature °C		EMF Millivolts		Reference Junction 0°C	
Bar	82621-4	82381-5	82921-10	62641-2	82251-15
Test	2415	2413	2413	2414	2414
T (°C)	EMF	EMF	EMF	EMF	EMF
100	+0.863	+0.061	+0.861	+0.860	+0.861
200	2.041	2.036	2.036	2.031	2.036
300	3.452	3.444	3.444	3.438	3.444
400	5.057	5.046	5.046	5.035	5.046
500	6.815	6.803	6.803	6.789	6.800
600	6.663	8.648	8.648	8.632	8.645
700	10.806	10.786	10.785	10.765	10.783
800	13.058	13.034	13.031	13.008	13.031
900	15.475	15.448	15.444	15.417	15.446
1000	18.055	18.029	17.993	17.993	18.026

Tophet C TM Wilbur B. Driver Co.
 Nominal Composition 61%Ni-15%Cr-Bal. Fe

TABLE 27

Provisional EMF Vs. Temperature Relationship Pt-40%Rh Vs. Pt-0.6 ThO₂
 Target EMF Vs. Pt67 of Selected Thermoelements

Temperature °C (IPTS 68)	EMF Millivolts		Reference Junction 0°C	
T (°C)	Pt-40%Rh Vs. Pt-0.6%ThO ₂	Tentative Tol. + 1/2% of Temp. For Couple	Thermoelement Pt-40%Rh Vs. Pt67	Thermoelement Pt-0.6%ThO ₂ Vs. Pt67
-50	-0.216		-0.217	-0.001
-40	-0.180		-0.180	+0.000
-30	-0.140		-0.140	+0.000
-20	-0.090		-0.090	+0.000
-10	-0.052		-0.053	+0.001
0	0		0	0
+25	+0.138		+0.140	+0.002
50	0.298		0.300	0.002
75	0.466		0.470	0.004
100	0.654		0.659	0.005
200	1.526		1.533	0.007
300	2.558		2.567	0.009
400	3.720		3.731	0.011
500	4.988		5.001	0.013
600	6.376	+0.042	6.391	0.015
700	7.871	+0.053	7.888	0.017
800	9.473	+0.054	9.492	0.019
900	11.178	+0.077	11.199	0.021
1000	12.985	+0.090	13.008	0.023
1100	14.895	+0.105	14.921	0.026
1200	16.879	+0.120	16.908	0.029
1300	18.938	+0.137	18.970	0.032
1400	21.087	+0.154	21.122	0.035
1500	23.238	+0.165	23.276	0.038
1600	25.394	+0.176	25.435	0.041
1650	26.473	+0.182	26.516	0.043

NOTES:

1. Tolerance of + 1/2% on Pt-40%Rh vs. Pt-0.6%ThO₂ advisory only. Additional work needed to develop final tolerance.
2. EMF vs. Pt67 for thermoelements Pt-40%Rh and Pt-0.6%ThO₂ for information only. This is a target EMF and is included for information only. Tolerance suggested for matched couple only.

TABLE 28

Target Nichrome vs. Pt67 Values and Needed Matching Values of 9R-682 vs. Pt67

Temperature °C (IPTS 68)	EMF Millivolts	Reference Junction 0°C
T °C	Nichrome Vs. Pt67	9R-682 Vs. Pt67
-50	-0.26	-0.25
-40	-0.21	-0.20
-30	-0.16	-0.15
-20	-0.11	-0.10
-10	-0.05	-0.04
0	0	0
+25	+0.18	+0.12
50	+0.39	0.21
75	0.62	0.30
100	0.87	0.36
150	1.42	0.46
200	2.04	0.55
250	2.72	0.66
300	3.45	0.85
350	4.23	1.09
400	5.06	1.35
500	6.83	1.91
600	8.77	2.51
700	10.88	3.15
800	13.16	3.83
900	15.61	4.57
1000	18.23	5.36

NOTES:

1. Nichrome is available commercially as a resistance (electrical) wire and not usually sold as a thermoelement. It is suggested that samples be obtained from producer from 2 or 3 different lots. Purchase wire of lot from which sample comes closest to values vs. Pt67 indicated above.
2. Values for 9R-682 vs. Pt67 are advisory only and are intended to show magnitude of EMF needed for the negative leg compensating extension wire.

TABLE 29

EMF Vs. Temperature Relationships For Matched Couple
And Matched Compensating Extension Wires Used In Probe

Temperature °C (IPTS 68)	EMF Millivolts	Reference Junction 0°C
T (°C)	Pt-40%Rh Vs. Pt-0.6%ThO ₂ A	Nichrome Vs. 9R-682 B Difference A - B
-50	-0.212	+0.201
-40	-0.175	+0.171
-30	-0.136	+0.003
-20	-0.092	+0.002
-10	-0.046	+0.044
0	0	0
+25	+0.148	+0.068
50	0.293	0.180
75	0.467	0.323
100	0.651	0.504
150	1.064	0.951
200	1.521	1.486
250	2.019	2.059
300	2.551	2.605
350	3.117	3.142
400	3.713	3.707
500	4.980	4.918
600	6.366	6.250
700	7.859	7.721
800	9.464	9.323
900	11.169	11.033
1000	12.984	12.862
1100	14.894	
1200	16.883	
1300	18.951	
1400	21.086	
1500	23.236	
1600	25.394	
1650	26.473	

APPENDIX A
PROBE DESIGN

APPENDIX A

Design Analysis For T4.1 Thermocouple

D. A. Toenshoff

Introduction:

The design of the subject thermocouple was based upon calculations formulated from the aerodynamic loading anticipated from the defined use conditions; simple stress concentration related to experimental stress to rupture data; and economic justification balanced against geometrical restraints and fabrication practicalities.

Structural & Economic Analyses

Geometrical restraints are imposed upon the design by the intended application and are shown in Figure A-1. The pertinent environment conditions provided were:

1. Mass Flow Rate - 144 lbm/ft²-sec.
2. Gas Stream Temperature - 1400°C with excursions to 1650°C (2550°F to 3000°F).
3. Static Gas Pressure - 160 lbf/in² abs.

From this criteria, and assuming for the first approximation a 5 to 1 cylindrical configuration, the following engineering parameters were calculated for an assumed temperature of 1650°C (worst case):

1. Mach No. ~ 0.4
2. Gas Density - ~ 1248 lbm/ft³
3. Reynolds No. (Based on diameter) - $\sim 1 \times 10^5$
4. Flow Velocity - $V = 1150$ ft/sec.
5. Fluid Viscosity - $\sim 3.86 \times 10^{-5}$ lbm/ft - sec.

From the equation for the Conservation of Linear Momentum for a control volume analysis:

$$1. \quad \vec{F}_S = \frac{\partial}{\partial t} \int_V \rho \vec{V} d\tau - \int_V \vec{\beta} \rho d\tau + \oint_S \vec{V} (\rho \vec{V} \cdot d\vec{A})$$

and assuming steady state flow ($\frac{\partial}{\partial t} \rightarrow 0$) and ignoring secondary effects ($\vec{\beta} \rightarrow 0$) the general statement (1) reduces and simplifies to the familiar equation:

$$2. \quad \sum \vec{F} = \frac{1}{2} C_D \rho \vec{V}^2 A$$

Where C_D = coef. of drag (~ 0.8)
 ρ = fluid density
 V = fluid velocity
 A = projected Area of obstruction

Making the appropriate substitutions, and estimating the thermocouples projected length as 1.90 inches equation (2) can be expressed as:

$$3. F = 25.8 (D) \text{ lbf}$$

Where D = probe diameter, inches

Equation (3) presents a simple linear relationship of fluid force imposed on the projected thermocouple sheath in the gas stream at 1650°C, expressed as a function of the sheath diameter.

By assuming that the flexural stress would be the predominant mode for failure, and realizing that the thermocouple probe can be treated as a simple cantilever beam we have;

$$4. \sigma = Mc/I$$

Where σ = flexural stress (lbf/in²)
M = Bending moment (lbf-in)
C = Distance to outer fiber (in)
I = 1st Area Moment of Inertia (in⁴)

Equation (4) can be used as an expression for the stress pattern anticipated under the assumed and defined conditions as previously set forth.

From equation (4) it can be readily seen that the stress varies directly with the bending moment, which in turn varies in proportion to the square of the thermocouple length and linearly with the diameter. The section modulus (I/C), generally taken as a statement of structural rigidity causes the resulting stress to vary inversely to the cube of the diameter.

By combining equations (3) and (4), substituting $\sigma = 2100$ psi (equivalent to a 100 hour stress to rupture life at 1450°C for Pt0.6Th02), and simplifying, we have:

$$5. D^4 - .1127D^2 = d^4$$

Where D = Thermocouple sheath
outside diameter (in.)
d = Thermocouple sheath
inside diameter (in.)

Equation (5) provides a simple statement of the thermocouples dimensions required to satisfy assumed allowable stress for the derived flow conditions.

By employing equation (5) a calculation can be made for candidate designs based on geometric configuration and "first cut" intrinsic economic advantage. Table I presents the results of this calculation. Relative intrinsic value was obtained by assuming a precious metal value of \$200/oz.

Reproduced from
best available copy. 

TABLE I
Dimensional Design Candidates
T4.1 Thermocouple

<u>Sheath Dimensions (Inches)</u>			<u>Relative Intrinsic Value, \$/Unit Length</u>
<u>O.D.</u>	<u>I.D.</u>	<u>Wall</u>	
.625	.574	.025	105.25
.5625	.504	.029	107.37
.500	.430	.034	112.03
.4375	.350	.043	118.58
.375	.250	.062	134.45
.250	-	-	-

As can be seen from Table I, for the defined conditions of fluid force and allowable stress, a .250" O.D. thermocouple is structurally inadequate. Also, for the defined conditions, it is evident from equation (4) that the load bearing capabilities of the thermocouple vary to the third power of the diameter, hence as the diameter decreases a proportionally larger wall thickness is required resulting in an equally proportionate weight (and hence intrinsic value) increase.

Practical considerations associated with the thermocouples manufacture, dimensional compatibility with related components and the geometrical restraints imposed by the end use dictate that sheath diameters from .500 O.D. and less be given weighed advantage over those of a larger size. The differential increase in relative intrinsic value, as shown, does not impose severe penalties for making these practical compromises.

By assuming a number of wall thicknesses for candidate thermocouple designs of nominal 1/2", 7/16", and 3/8" O.D. dimensions and substituting these values in equation (4), a tabulation can be readily obtained for the resulting stress as a function of wall thickness. This calculation was conducted and is presented in graphic form in Figure A-2. Overlaid on Figure A-2 is the stress to rupture time (in hours) for Pt0.6%ThO₂ thermocouple sheath material. From this graph, it can readily be appreciated, that as previously expressed, the stress bearing capability, varying as the diameter to the third power, materially increases for larger diameter. With this increased stress bearing capability extended estimated life is also realized.

A simplified economic ratio analysis was conducted to assess the possibility of identifying the optimum relationship between relative intrinsic value and estimated hours of life. Table II presents the finding of this analysis.

TABLE II
Simplified Economic Ratio Analysis
Estimated Life Versus Relative Intrinsic Value

<u>Stress to Rupture Life at 1450°C (Hours)</u>	<u>Relative Intrinsic Value Per Unit Length Per Unit Estimated Life (\$/Inch/Hr.)</u>		
	<u>1/2" O.D.</u>	<u>7/16" O.D.</u>	<u>3/8" O.D.</u>
1500	.22	N/A	N/A
1000	.26	N/A	N/A
500	.40	.46	N/A
300	.54	.59	N/A
200	.72	.79	1.08
100	1.12	1.20	1.34
50	1.65	1.68	2.09

By performing a graphic regression analysis with estimated life and relative intrinsic value per unit length per unit life it can be demonstrated that no significant advantage exists between the nominal 1/2" and 7/16" O.D. dimensions, while both these candidate design sizes are economically preferred over the 3/8" diameter. Due to the relativity of the stress bearing capability versus nominal wall thickness for the three diameters under consideration as shown in Figure A-2, no optimum break even point was obtained in the regression analysis.

Based on the theoretical calculations and economic analysis as presented herein, and in consideration of manufacturing practicalities consistent with the objective for conservative design balanced against reasonable economics it is concluded that the thermocouple should be designed such as to include a nominal 7/16" O.D. shield tube structure with a .065" wall. This design then provides an estimated life expectancy at 1450°C in excess of 200 hours with the good prospect of extending the usable life at a later date by a factor of 7 after consideration of the in use performance.

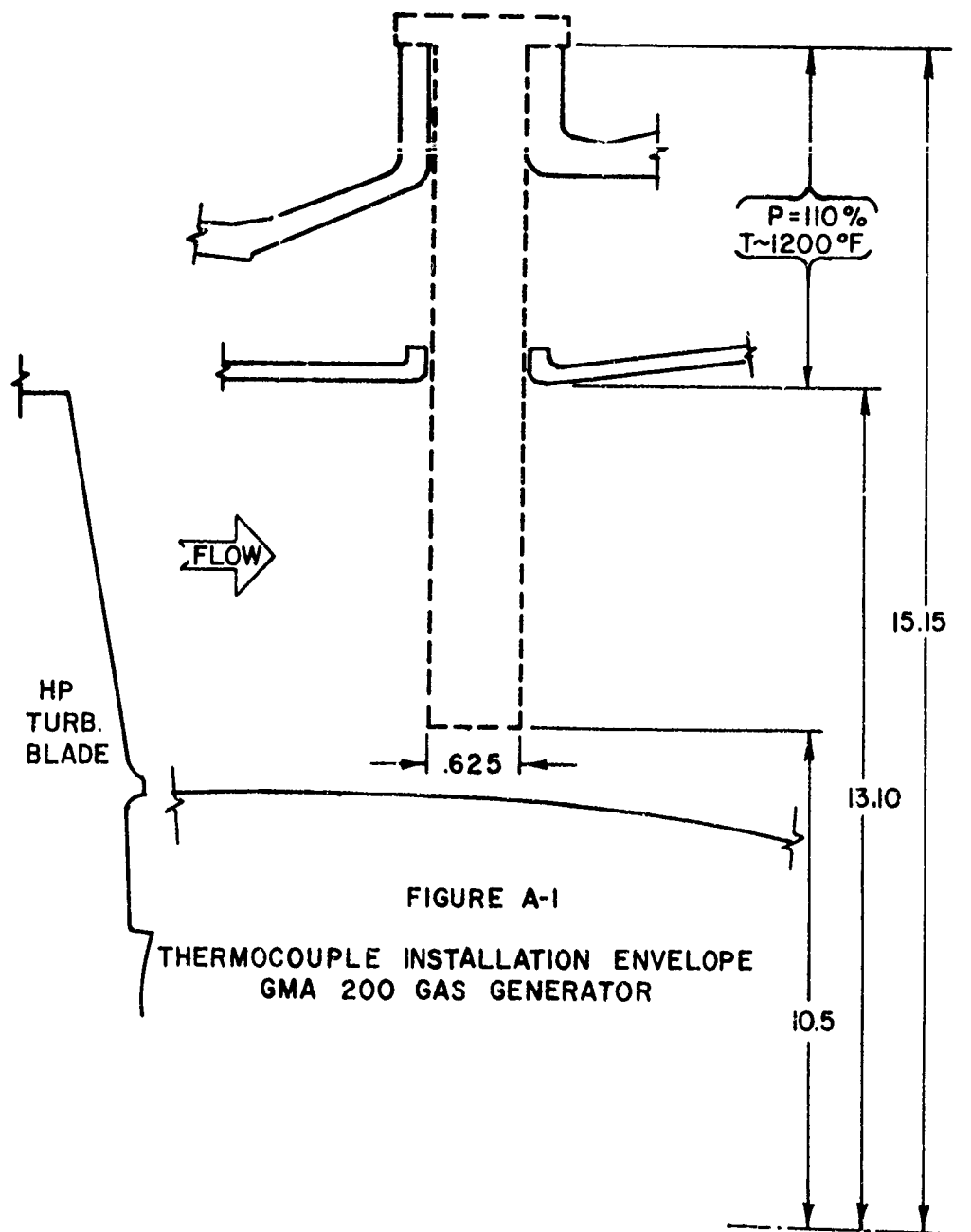


FIGURE A-1
THERMOCOUPLE INSTALLATION ENVELOPE
GMA 200 GAS GENERATOR

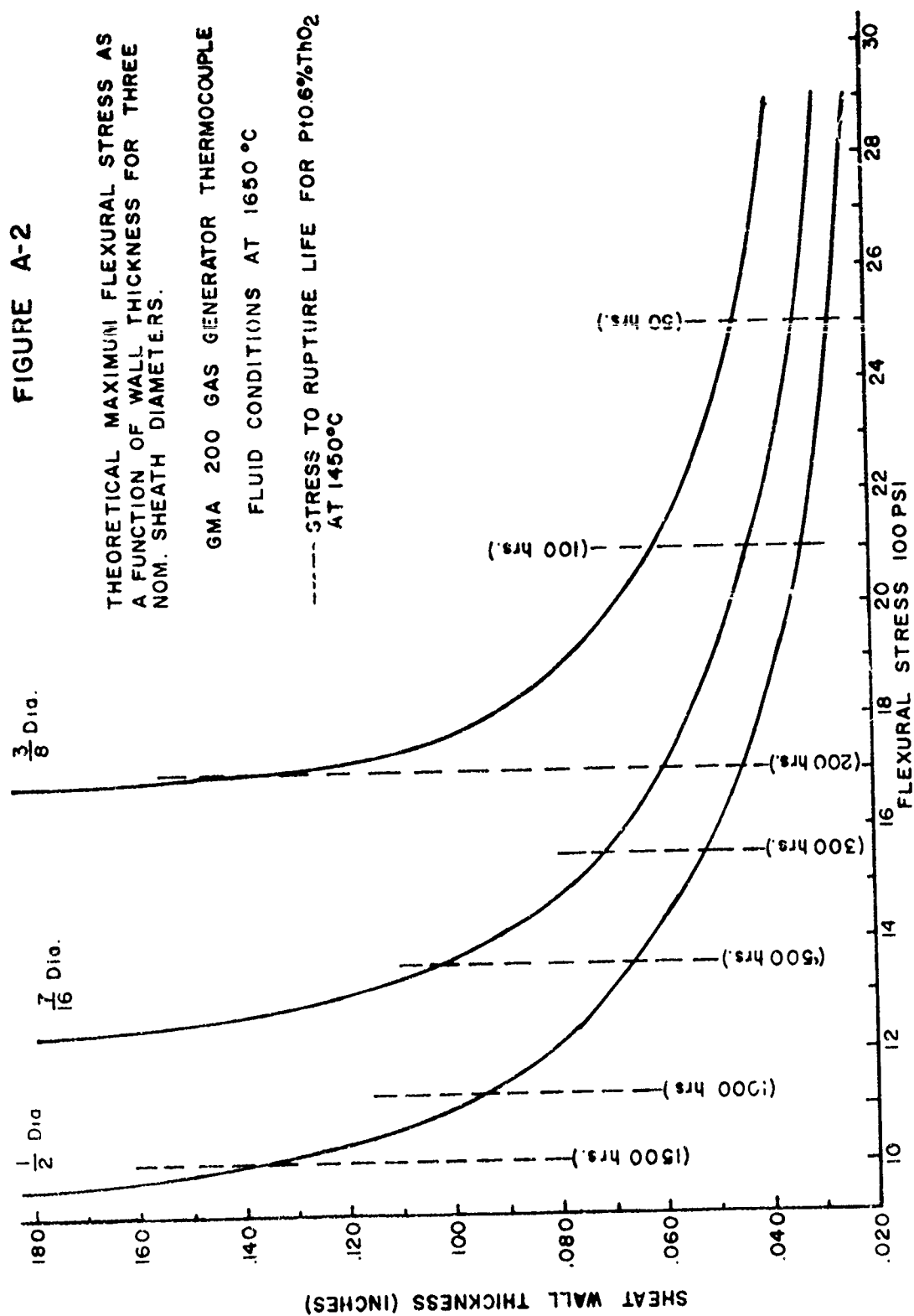
FIGURE A-2

THEORETICAL MAXIMUM FLEXURAL STRESS AS
A FUNCTION OF WALL THICKNESS FOR THREE
NOM. SHEATH DIAMETERS.

GMA 200 GAS GENERATOR THERMOCOUPLE

FLUID CONDITIONS AT 1650 °C

----- STRESS TO RUPTURE LIFE FOR P10.6%ThO₂
AT 1450°C



APPENDIX B
VIBRATION TESTING OF FINISHED PROBE

Specification for Thermocouple Vibration Testing

Experimental T/C

No. DDS 20200

For

Research & Development Department
Engelhard Industries Division
Engelhard Minerals & Chemicals Corporation
Menlo Park, Edison, New Jersey 08817

1. SCOPE

This specification covers the vibration test requirements for qualification testing of an experimental high temperature thermocouple for use in advanced designed gas turbine engines.

2. PURPOSE

The purpose of this investigation is to locate and identify any physical or structural weakness inherent in the thermocouple design or fabrication technique, under the specified test conditions, which would limit the device's usefulness or impair its functioning.

3. REFERENCE DOCUMENTS

3.1 Envelope Drawing T/C No. DDS 20200

4. VIBRATION FIXTURE

4.1 A vibration fixture shall be designed and provided such as to satisfy the following conditions.

4.1.1 A control accelerometer shall be placed on the fixture as close as possible to the test thermocouple mounting flange.

4.1.2 The fixture shall be capable of testing the thermocouple in all three major orthogonal axes at the indicated temperatures.

4.1.3 Sinusoidal transmissibility shall be such that vibration input in the axis of applied vibration at the thermocouple mounting point shall be within plus 3db of that specified over the entire frequency band.

4.1.4 Sinusoidal cross-talk shall not exceed the input.

5. FREQUENCY SPECTRUMS

5.1 The thermocouple shall be subjected to sinusoidal vibration, as indicated by the control accelerometer, as shown in Table 1.

TABLE 1

Sinusoidal Vibration Spectrum

<u>Frequency $\pm 2\% + 1/2$ Hz</u>	<u>Requirement</u>
10 - 2000 Hz	.50 inch constant double amplitude displacement, $\pm 10\%$ with automatic crossover at 7 "g" constant acceleration

- 5.2 Sweep rate shall be logarithmic and not to exceed one octave per minute. An upswing and downswing is required in each of the three major orthogonal axes.

6. RESONANT SEARCH6.1 Test Method

The thermocouple shall be subjected to the specified vibration spectrum, at ambient temperature, and a search made for major resonant points. A monitoring accelerometer shall be employed for this inspection, the mass of which is not to materially affect the recorded test results.

6.2 Thermocouple Monitoring

A suitable instrument (low voltage multi-test meter, e.g. "Simpson") shall be connected between the thermocouple lead wires and monitored for circuit continuity during the test. Similarly, the insulation resistance between any one lead wire and thermocouple ground shall be monitored. The insulation resistance measuring instrument shall be capable of measuring at least 1×10^5 ohm and not impress more than 50 v.d.c. on the thermocouple.

6.3 Test Report

A report shall be prepared and submitted upon completion of the test which will provide the following information for tests conducted on each of the three major orthogonal axes:

- A. Frequency, displacement (double amplitude) of the control accelerometer, and total time at test at loss of circuit continuity, if any.

- B. Frequency, displacement (double amplitude) of the control accelerometer, and total time at test for reduction of insulation to less than 1×10^4 ohm.
- C. Frequency, displacement (double amplitude) of the monitoring and control accelerometer, total time at test, and distance from thermocouple mounting flange for any resonant points with a Q greater than 1.5

7. HIGH TEMPERATURE VIBRATION TEST

- 7.1 The thermocouple shall be subjected to the specified vibration spectrum with the hot junction temperature maintained at $1450^\circ\text{C} \pm 25^\circ\text{C}$. Radiation or RF induction is the preferred heating method. Gas torches (oxygen-acetylene) may be used if sufficient quantity is employed to obtain uniform radial temperature along the thermocouples linear axis.

Secondary fine wire thermocouples (nominal .020 or less wire dia. ISA Type K) shall be attached to the transition joint and to the thermocouple at a point 1/4 inch distance from the test fixture. (Ref. Par. 3.1) The source of heat shall be adjusted such as to provide as uniform a temperature of $1450^\circ\text{C} \pm 25^\circ\text{C}$ along the axis of the thermocouple as possible, while restricting the transition temperature to $675^\circ\text{C} \pm 25^\circ\text{C}$.

Note: The thermocouple under test may be used as a means to measure the temperature. The electromotive force of the thermocouple at 1425°C and 1475°C is 21.52 mv and 22.58 mv respectively with the reference junction at 25°C .

7.2 Thermocouple Monitoring

A 50 mv maximum full scale recorder shall be employed to provide a permanent record of the test thermocouples output during the vibration test. Recorders or individual readings shall be taken of the secondary transition joint and cold end thermocouples.

Inspection for high temperature resonance, or other vibration induced occurrence may be observed aurally, visually, with strobe lights, microscopes, etc., or any other method deemed suitable or appropriate by a trained and experienced investigator.

7.3 Test Report

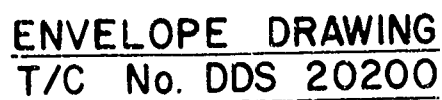
A report shall be prepared and submitted upon completion of the test which will provide the following information for tests conducted on each of the three major orthogonal axes:

- A. Copies of recorder trace showing thermocouple emf output during each vibration sweep. Notation shall be made of the chart calibration, start of upsweep, and start of downsweep. Any discontinuities, irregularities, or deviations in emf signal shall be noted as to vibration frequency at time of occurrence.
- B. Temperature readings recorded with the secondary fine wire thermocouples. If not continuously recorded, data points shall be taken at the start of the sweep, transition from upsweep to downsweep, and at the conclusion of the sweep.
- C. Any observed occurrence during the test which in the judgement of a trained and experienced investigator is abnormal.

8. DOCUMENTATION

8.1 Three copies of a final report shall be prepared, one of which must be a reproducible master, which will include the following information:

- A. Make, model and serial number of vibration shaker and power console.
- B. Make, model, serial number, and date of last calibration of control and monitoring accelerometers.
- C. Test report as per Par. 6.3
- D. Test report as per Par. 7.3
- E. Photograph and negative of high temperature test setup.



GENERAL ELECTRIC

SPACE DIVISION

GENERAL ELECTRIC COMPANY VALLEY FORGE SPACE CENTER
(MAIL P. O. BOX 8555 PHILADELPHIA PENNSYLVANIA 19101) Phone (215) 962-2000

August 5, 1975

Engelhard Industries Division
Engelhard Minerals Chemicals Corporation
Menco Park
Edison, New Jersey 06817

Attention: D. A. Toenshoff

Subject: Thermocouple Vibration Testing Quotation
Reference C-75189

Gentlemen:

Testing of one (1) thermocouple, No. DDS 20200, per the specification furnished with your 20 May 1975 Request for Quotation can be accomplished for the sum of \$1300.

The price quoted is based on the following clarifications and/or exceptions to your specification.

Page 1, Paragraph 5.1

Sinusoidal vibration will be limited to 7 G's or .50 inch double amplitude displacement $\pm 10\%$ in low frequency range.

Page 2, Paragraph 6.3

In lieu of plotting displacement, acceleration will be plotted/monitored and included in the Final Test Report. Displacement will be calculated from the acceleration frequency data.

Page 3, Paragraph 7.1

An oxygen-acetylene torch will be used to heat the thermal-couple during high temperature vibration test.

Note: The secondary fine wire thermocouples are to be provided and attached to the test specimen by Engelhard Industries.

GENERAL ELECTRIC

D. A. Toenshoff

- 2 -

August 5, 1975

Page 4, Paragraph 8

Four (4) copies of the Final Report will be provided (original plots included) in lieu of three (3) copies and one (1) reproducible master.

Polaroid photos will be provided in lieu of negatives.

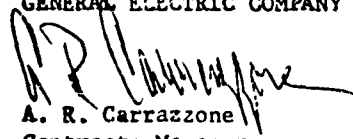
Testing can be accomplished within three (3) weeks after receipt of order and test sample.

This quotation will remain valid for sixty (60) days from date of issue and is subject to mutually acceptable terms and conditions being included in any resultant order.

If we can be of any further service, please contact the undersigned.

Very truly yours,

GENERAL ELECTRIC COMPANY



A. R. Carrazzone
Contracts Manager
Room U4018; A/C 215 962-6652

/shj

TEST REPORT CT-5-K8C

TEST REPORT

EXPERIMENTAL THERMOCOUPLE

VIBRATION TEST

FOR

ENGELHARD INDUSTRIES DIVISION

RESEARCH & DEVELOPMENT DEPARTMENT

BY

GENERAL ELECTRIC COMPANY

SPACE SYSTEMS ORGANIZATION

TEST OPERATIONS SUBSECTION

SEPTEMBER 10, 1975

PREPARED BY:

R.G. Shoulberg 9/16/75
R.G. SHOULBERG, TEST PROJECT ENGINEER

APPROVED BY:

A.C. Hibbets
A.C. HIBBETS, MANAGER
DYNAMIC TEST & ENVIRONMENTAL ENGINEERING

1.0 OBJECTIVE

To subject an experimental high temperature thermocouple to the Vibration Test Procedures specified by Engelhard Industries DDS 20200.

2.0 SUMMARY

The experimental thermocouple successfully passed the vibration tests per the requirements of Engelhard Industries.

3.0 CUSTOMER

Engelhard Industries Division
Engelhard Minerals & Chemicals Corp.
Menlo Park, Edison, New Jersey 08817

4.0 REQUIREMENT DOCUMENT

Engelhard Industries Specification for Thermocouple Vibration Testing No. DDS 20200.

5.0 SECURITY CLASSIFICATION

Unclassified

6.0 TEST SPECIMEN DESCRIPTION

Experimental Thermocouple S/N 102 DDS 20200

7.0 TEST EQUIPMENT DESCRIPTION

The test equipment provided by the General Electric Company in support of this program is presented in Table 1.

8.0 TEST FACILITY

The General Electric Company Space Systems Organization's Component Test Laboratory was used to support testing.

9.0 TEST PROCEDURE

The sinusoidal vibration test was conducted in accordance with Engelhard test requirements. The unit was subjected to sinusoidal vibration test as follows:

<u>Frequency (Hz)</u>	<u>Applied Vibration Level</u>
10 - 2000 - 10	7g (0-peak) limited to 0.5" D.A.
Sweep Rate	1 octave/min.
Time per Axis	8.65 minutes

10.0 TEST FIXTURE

The vibration test fixture was provided by GE (47D231045 plus adapter).

11.0 TEST CONDITIONS

The thermocouple was mounted in the vibration fixture and on the C-150 shaker system.

Insulation resistance checks were performed before and after each vibration at ambient temperature.

During ambient temperature vibration tests, continuity of the experimental thermocouple was observed. Response measurements to determine significant resonant frequencies were made with an accelerometer sensing the axial acceleration of the mounting fixture. This method, while not giving quantitative amplification factors will aid in determining resonant frequencies of the total system. Control accelerometer acceleration versus frequency plots and response accelerometer plots are included in this report.

High temperature during hot vibration testing was achieved with an oxy-acetylene torch. Temperatures were measured by monitoring and recording T/C output in millivolts. Test accelerometers were wrapped with asbestos cloth which was kept water-soaked during application of the torch.

After completion of the ambient and high temperature tests, additional vibration was performed for lead and transverse axes. During these tests a small accelerometer (1.5 gram) was bonded to the T/C tip. Sensing direction was in the same direction as the input vibration enabling the measurement of response versus input. Plots of these responses are included in this report.

12.0 DISCUSSION OF RESULTS

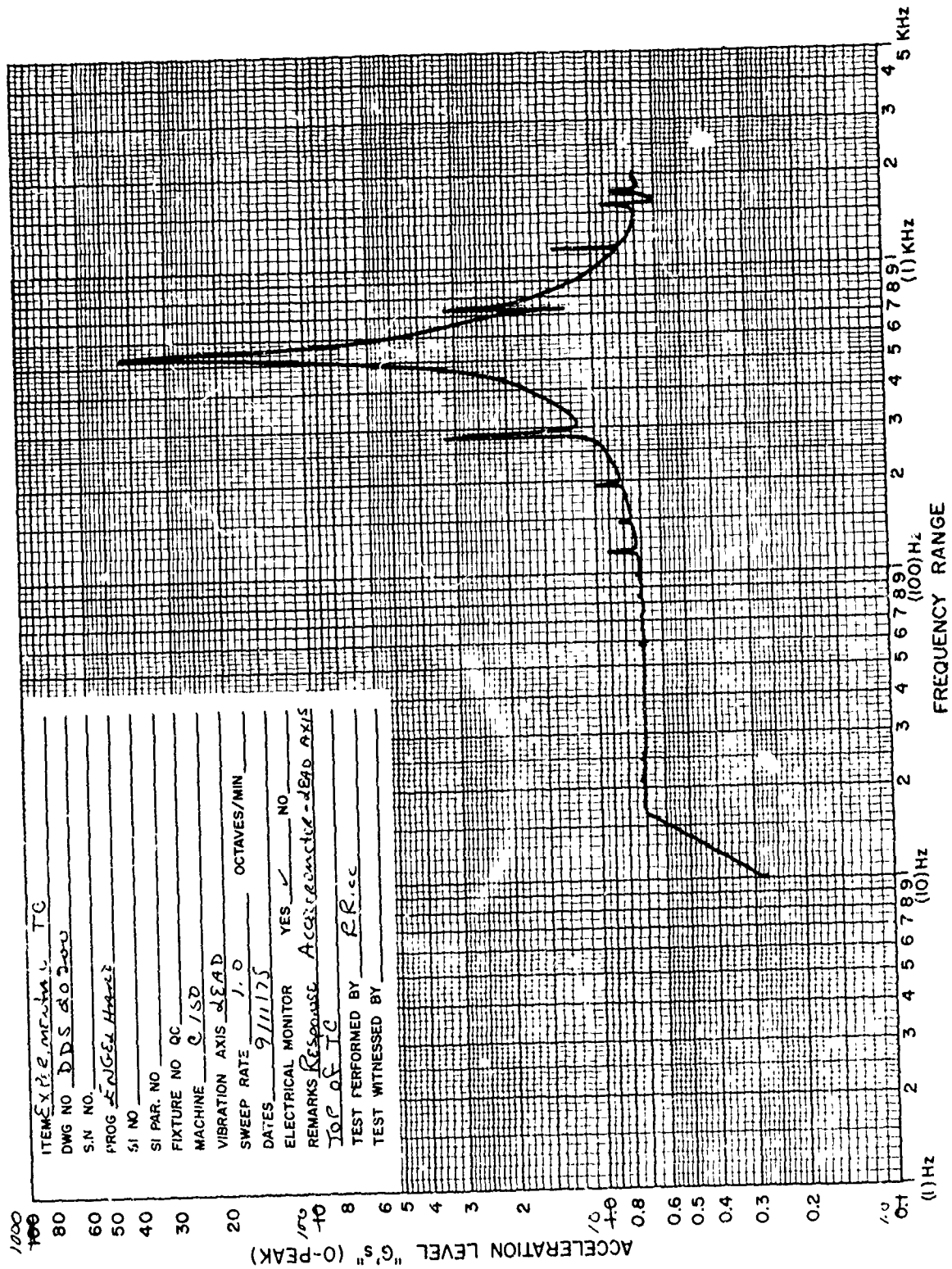
The thermocouple survived all testing as demonstrated by continuity, insulation resistance and visual inspection. Lead and transverse response plots shown major resonances at 530 Hz and 730 Hz which are the classic first cantilever modes. The difference is attributed to the asymmetry of the mounting. The tip deflection ratio at 530 Hz relative to the base displacement is $440 \pm 7 = 62.86$. The input displacement at 7g (0-peak) is

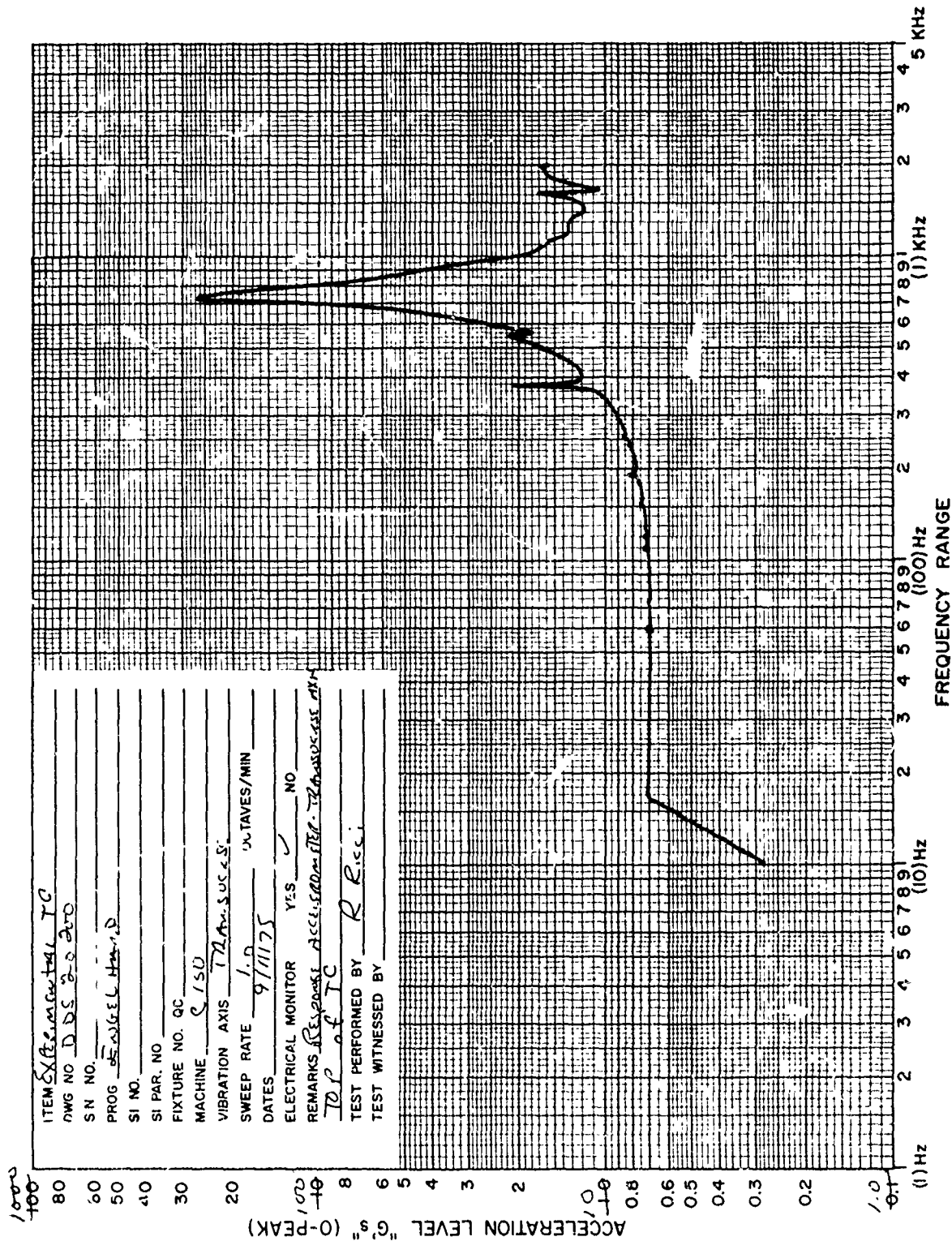
$$d = \frac{7}{0.102 (530)^2} = 0.000244, \text{ therefore, the tip displacement} \\ (\Delta) = 0.015 \text{ inch.}$$

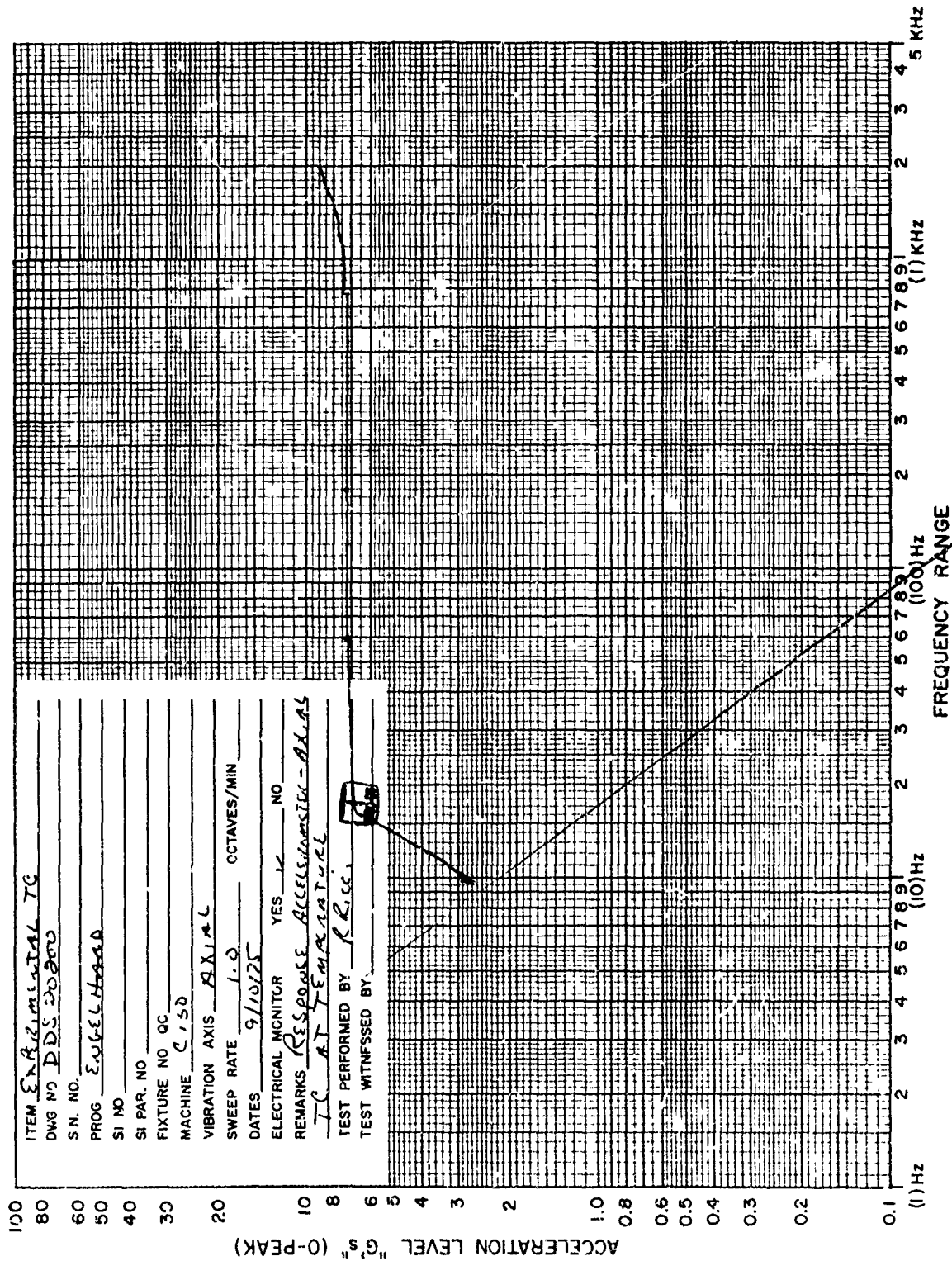
Further analysis of the support in actual service should be made to evaluate how realistic this mode of vibration will be.

TABLE 1 VIBRATION TEST EQUIPMENT

ITEM	MANUFACTURER	MODEL NO.	IC NO.	CALIBRATION DATE	CALIBRATION DUE DATE
Vibration System consisting of:					
Vibration Exciter	MB Electronics Corp.	C-150		N/A	N/A
Cooling Unit		PHM 11		N/A	N/A
Power Amplifier		4700	NW2141	N/A	N/A
Accelerometer	Endevco	2273	VD 0221	8/18/75	8/18/76
Accelerometer	Endevco	2273A	G40111	8/18/75	8/18/76
Logarithmic Converter	Hewlett-Packard	7562A	NW6193	4/8/75	10/10/75
Logarithmic Converter	MB Electronics	N165	NW2096	5/27/75	11/26/75
Hydrostatic Bearing Table	Team Corporation	1830PB	NW3283	N/A	N/A
Spectral Density Voltmeter	MB Electronics	N122	NW2097	12/13/74	9/12/75
Oscilloscope	Hewlett-Packard	122AR	NW2098	10/4/74	10/3/75
Sweep Oscillator	MB Electronics	N752-5	NW2103	7/15/75	10/15/75
Amplitude Servo Monitor	MB Electronics	N753A	NW2102	7/15/75	10/15/75
Zero Drive Charge Amplifier	MB Electronics	N400 N400	NW2133 NW2100	8/12/75 7/16/75	11/12/75 10/16/75
Millivoltmeter	Doric	210	NW6130	2/6/75	11/6/75
Megger	Electrospace Corp.	650	G24703	7/1/75	7/1/76

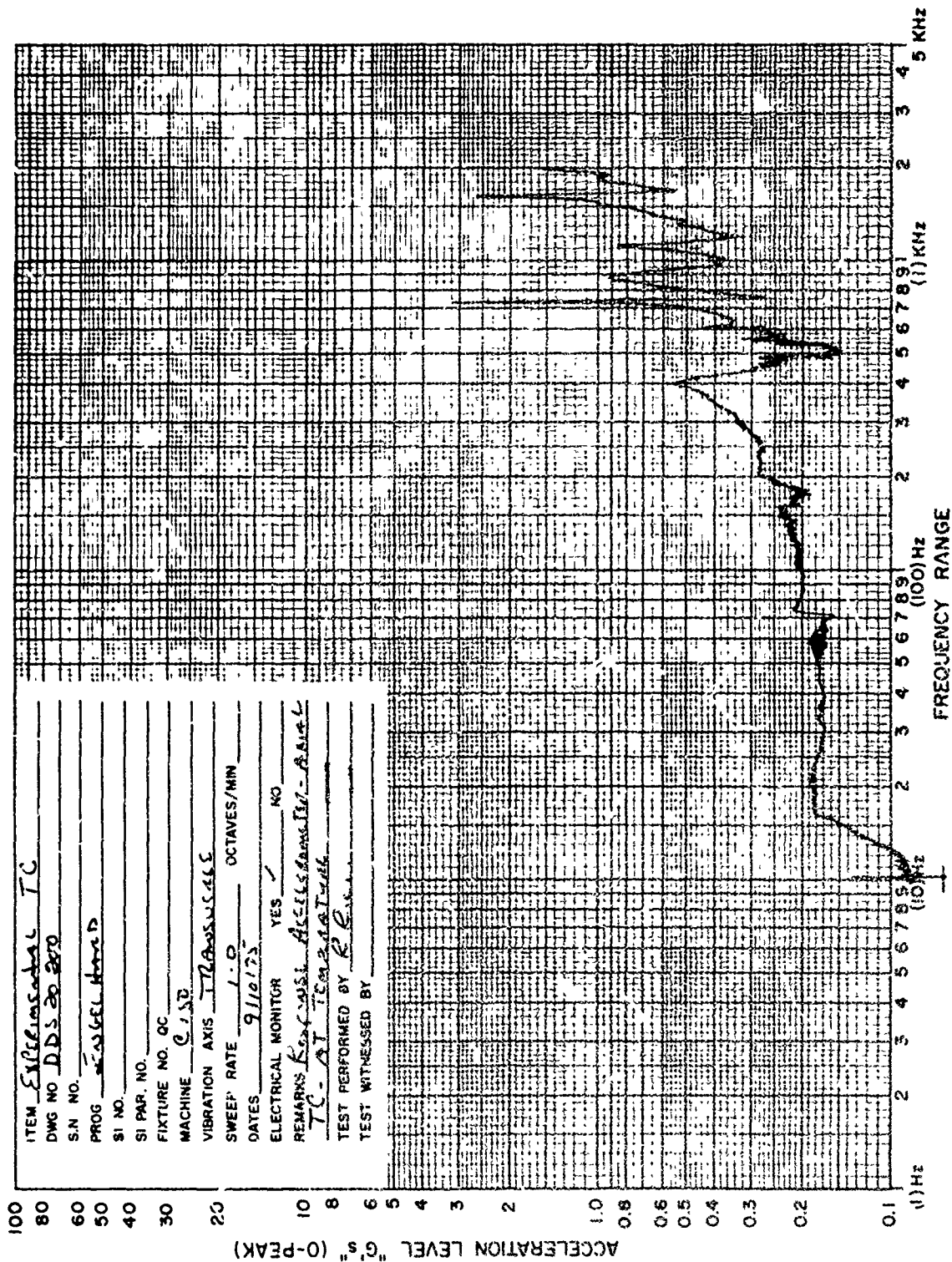




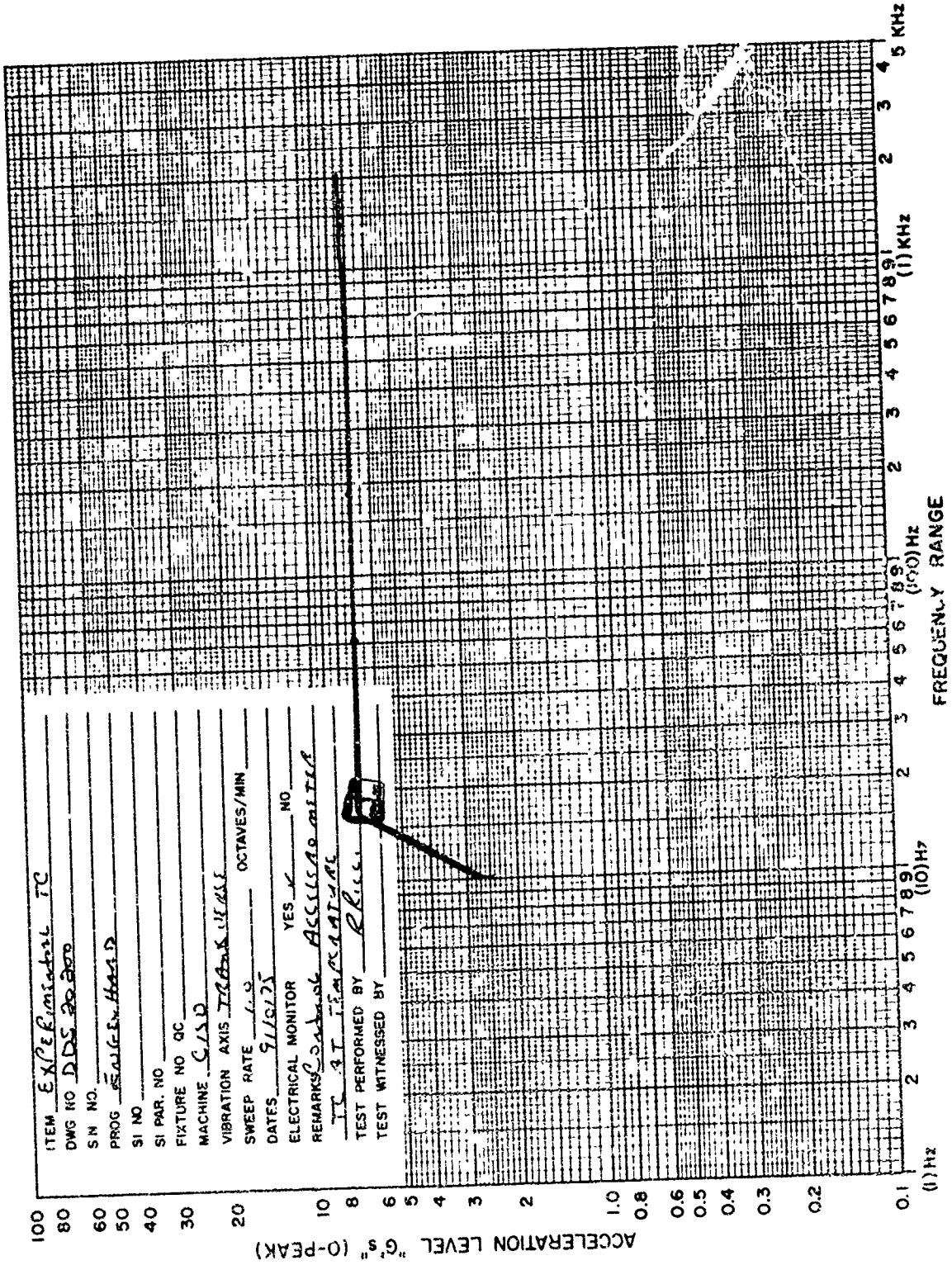


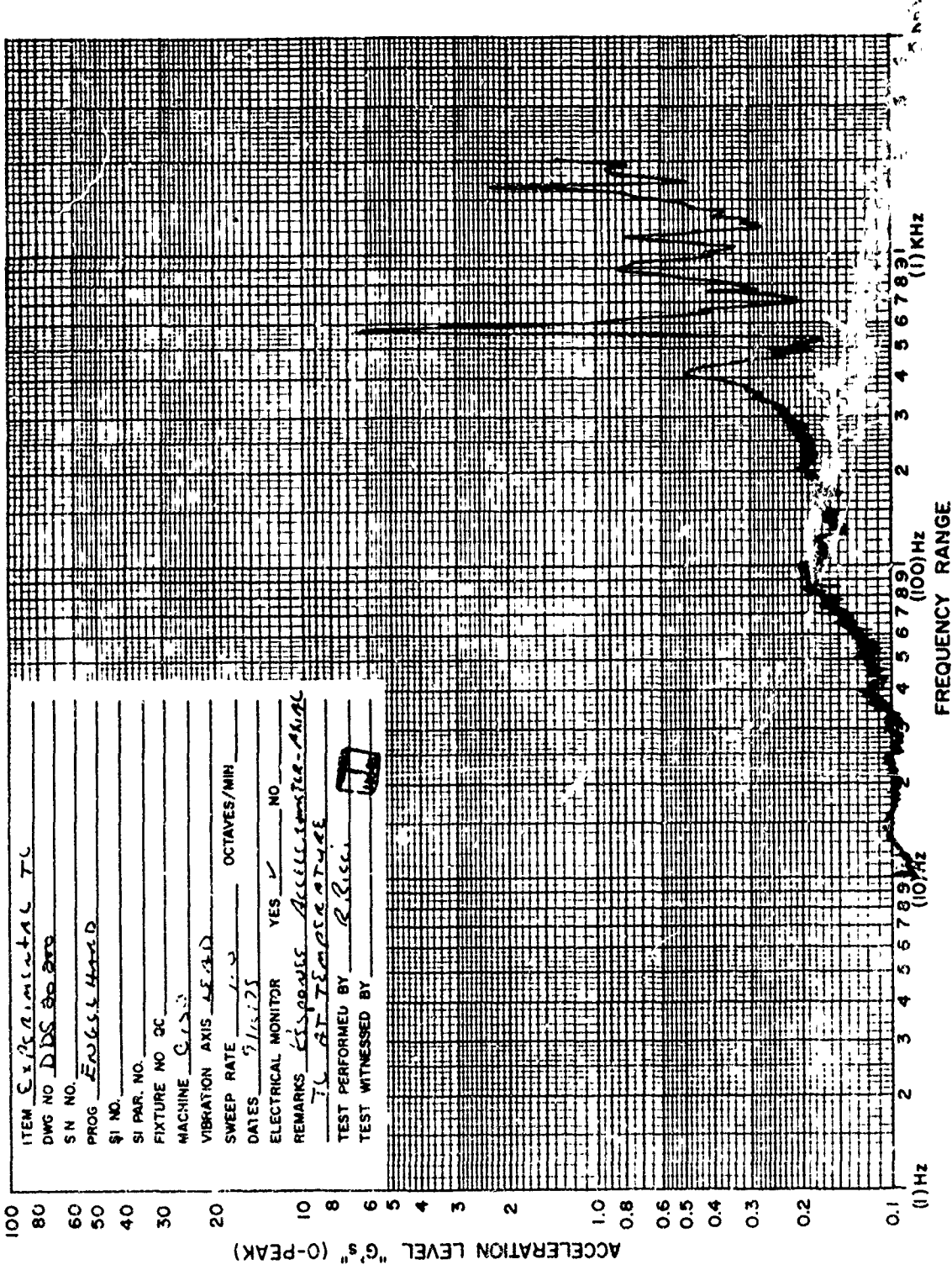
ITEM EXPERIMENTAL TC
DWG NO DDS-3090D
S.N. NO.
PROG. SAMPLE HAND
SI NO.
SI PAR. NO.
FIXTURE NO. OC
MACHINE CUSO
VIBRATION AXIS Axial
SWEEP RATE 1.0 OCTAVES/MIN
DATES 9/16/78
ELECTRICAL MONITOR YES ☒ NO
REMARKS Control Accelerometer TC AT Tuckman Inc.
TEST PERFORMED BY R.H.C.
TEST WITNESSED BY _____

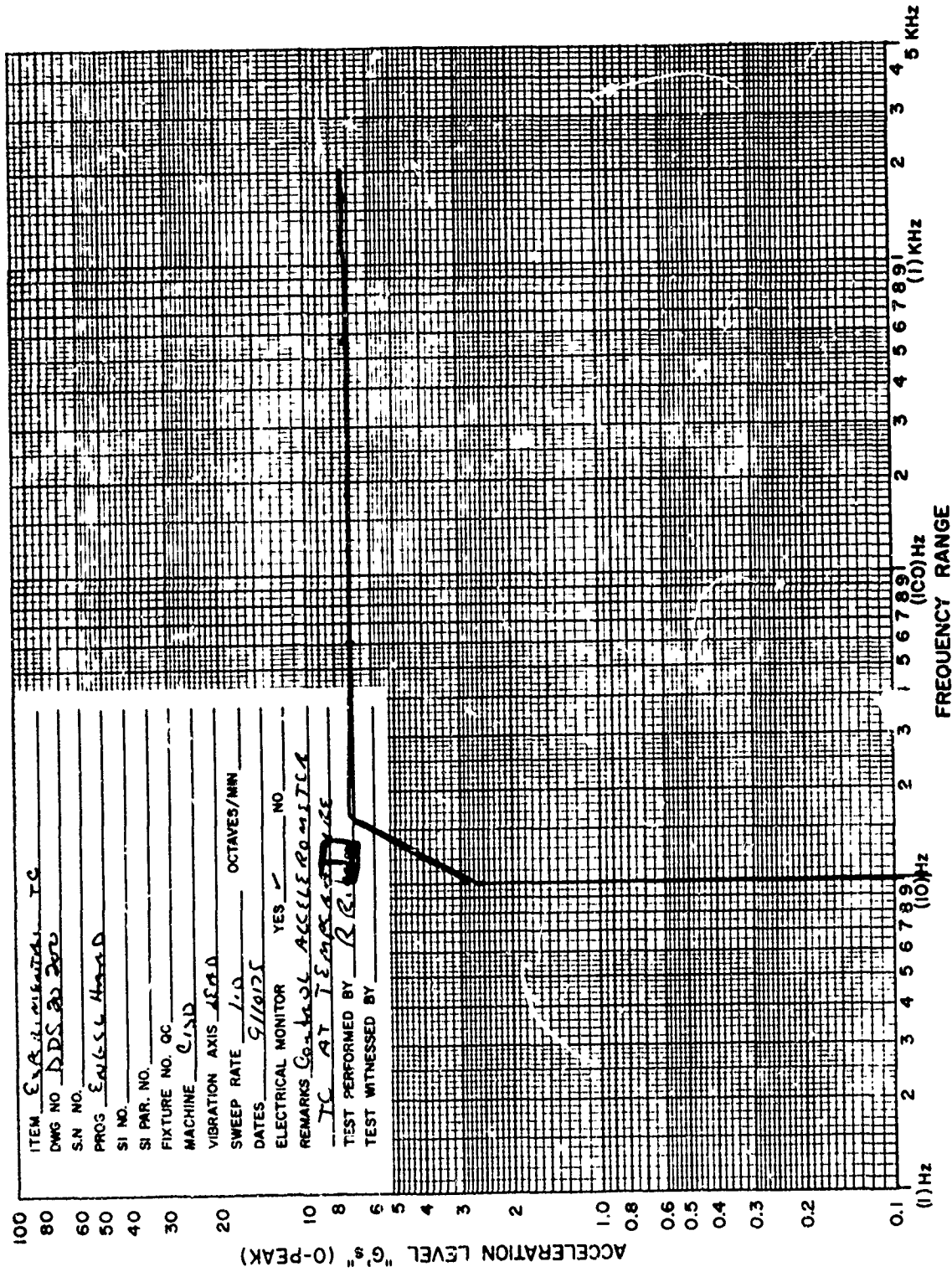
ACCELERATION LEVEL "g"_{rms} (0-Peak)
FREQUENCY RANGE (Hz)



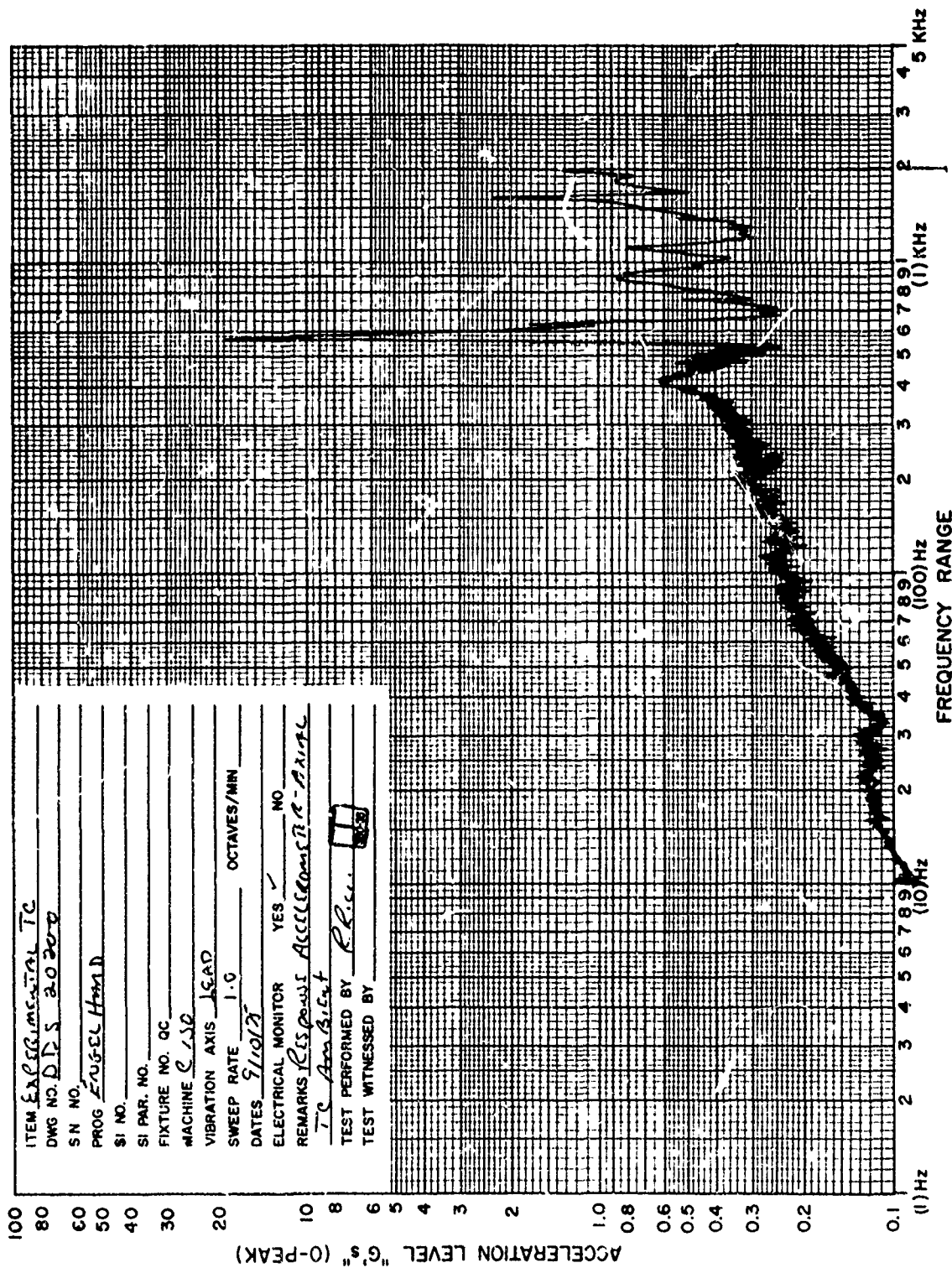
ITEM EXPERIMENTAL TC
 DWG NO DDS 20200
 SN NO. _____
 PROG ENGINE HARD
 SI NO. _____
 SI PAR. NO. _____
 FIXTURE NO. QC _____
 MACHINE C-150
 VIBRATION AXIS TRANSLVERSE
 SWEEP RATE 1.0 OCTAVES/MIN
 DATES 9/18/75
 ELECTRICAL MONITOR YES X NO _____
 REMARKS Control Accelerometer
TC AT TEMPERATURE
 TEST PERFORMED BY R. B. C.
 TEST WITNESSED BY _____



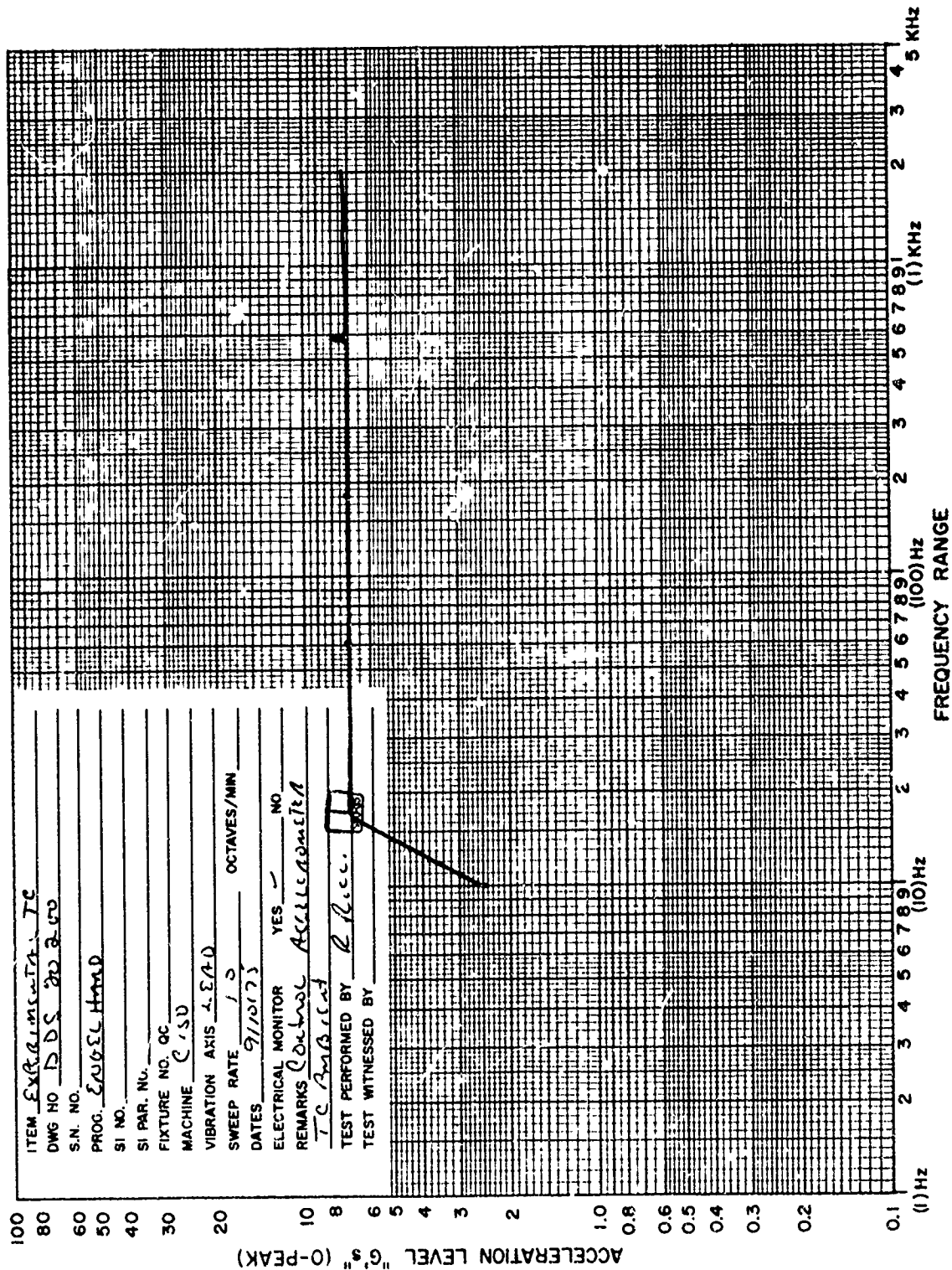


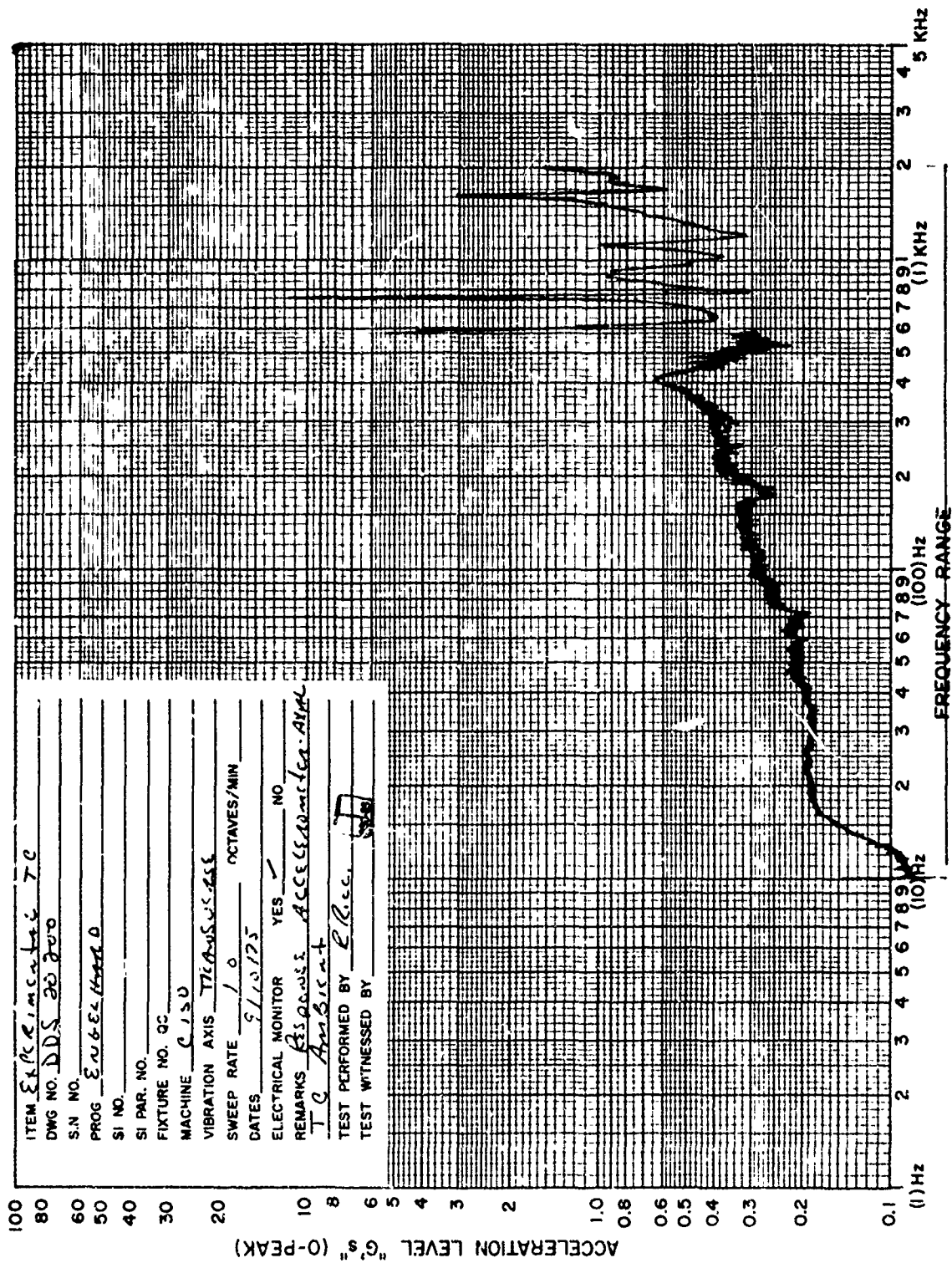


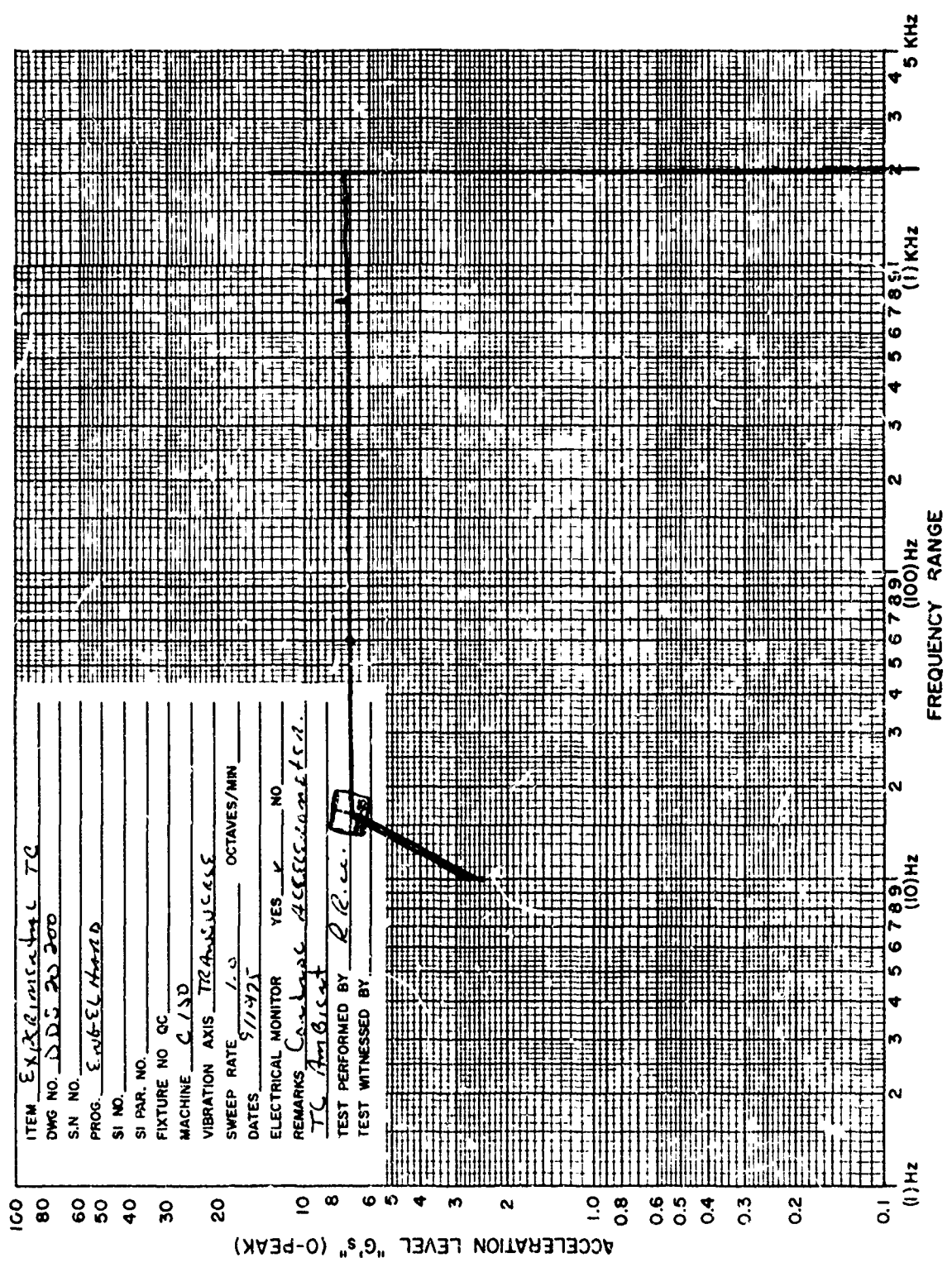
ITEM ENG. 1000000000 TC
 DWG NO 1000000000
 S.N. NO. 1000000000
 PROG ENG. 1000000000
 SI NO. 1000000000
 SI PAIR. NO. 1000000000
 FIXTURE NO. QC 1000000000
 MACHINE 1000000000
 VIBRATION AXIS 1000000000
 SWEEP RATE 1000000000 OCTAVES/MIN
 DATES 1000000000
 ELECTRICAL MONITOR YES ☒ NO
 REMARKS 1000000000
1000000000
 TEST PERFORMED BY 1000000000
 TEST WITNESSED BY 1000000000



ITEM EXPERIMENTAL TC
 DWG NO. DD5 20200
 SN NO. _____
 PROG ENGELHARD
 SI NO. _____
 SI PAR. NO. _____
 FIXTURE NO. QC _____
 MACHINE C 130
 VIBRATION AXIS HEAD
 SWEEP RATE 1.0 OCTAVES/MIN _____
 DATES 9/10/73
 ELECTRICAL MONITOR YES ☒ NO ☐
 REMARKS RESPONSE ACCELEROMETER - ANALOG
TC Ambient
 TEST PERFORMED BY R. Lee
 TEST WITNESSED BY _____







ITEM EXPERIMENTAL TC

DWG NO. DDJ 20200

S.N. NO. _____

PROG. EMBEL HARD

SI NO. _____

SI PAR. NO. _____

FIXTURE NO. QC

MACHINE C130

VIBRATION AXIS TRANSLUCSE

SWEEP RATE 1.0 OCTAVES/MIN

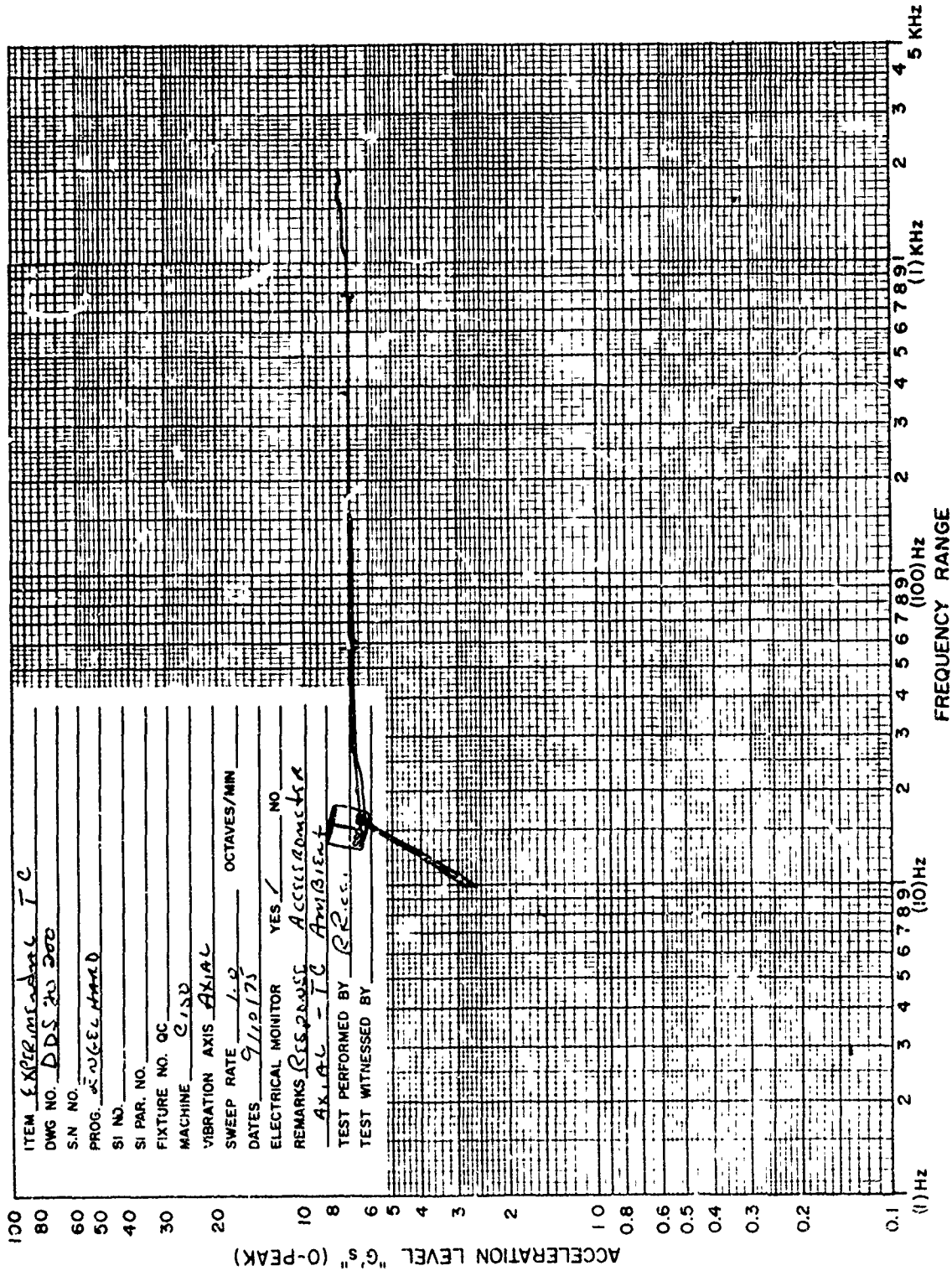
DATES 9/1/75

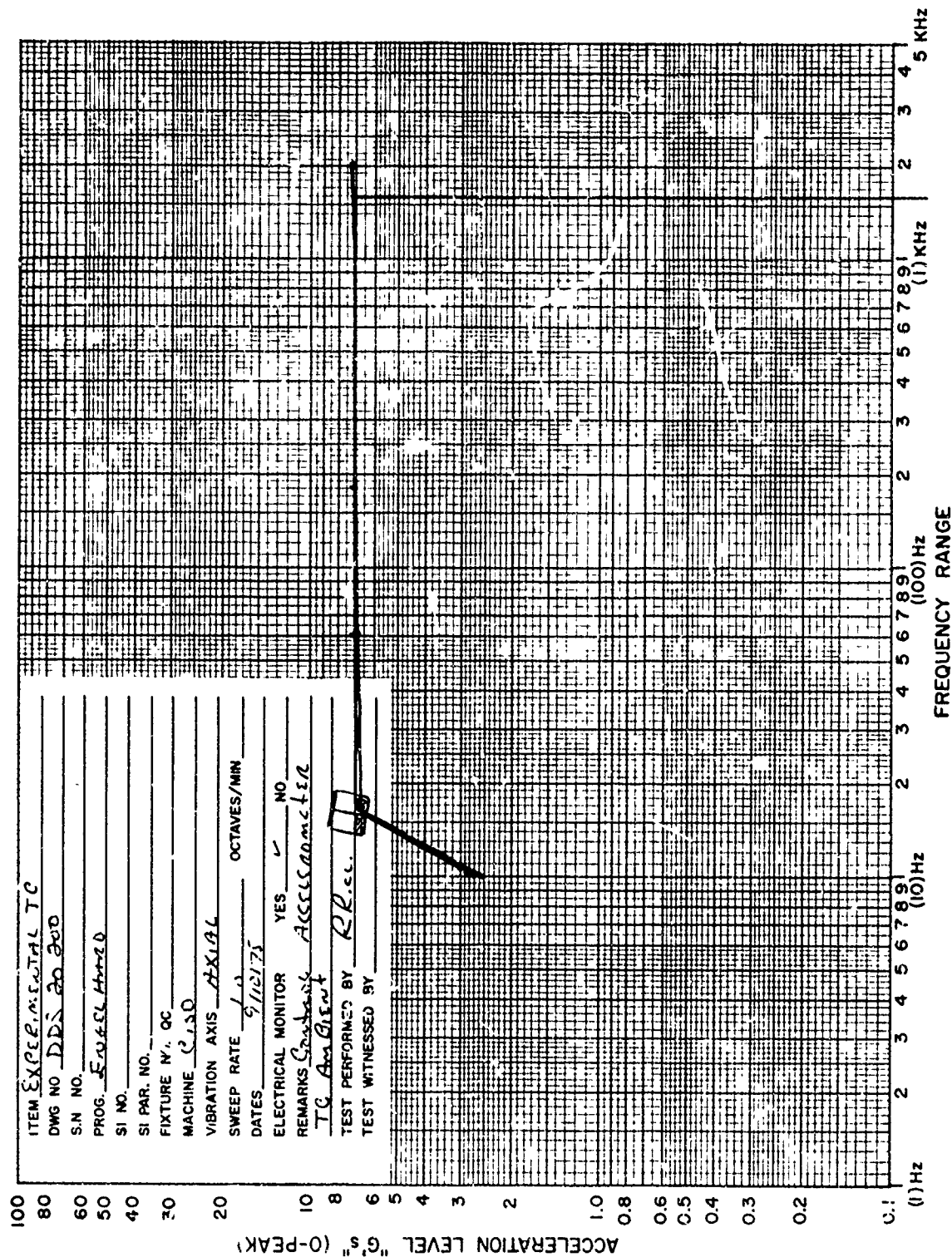
ELECTRICAL MONITOR YES X NO _____

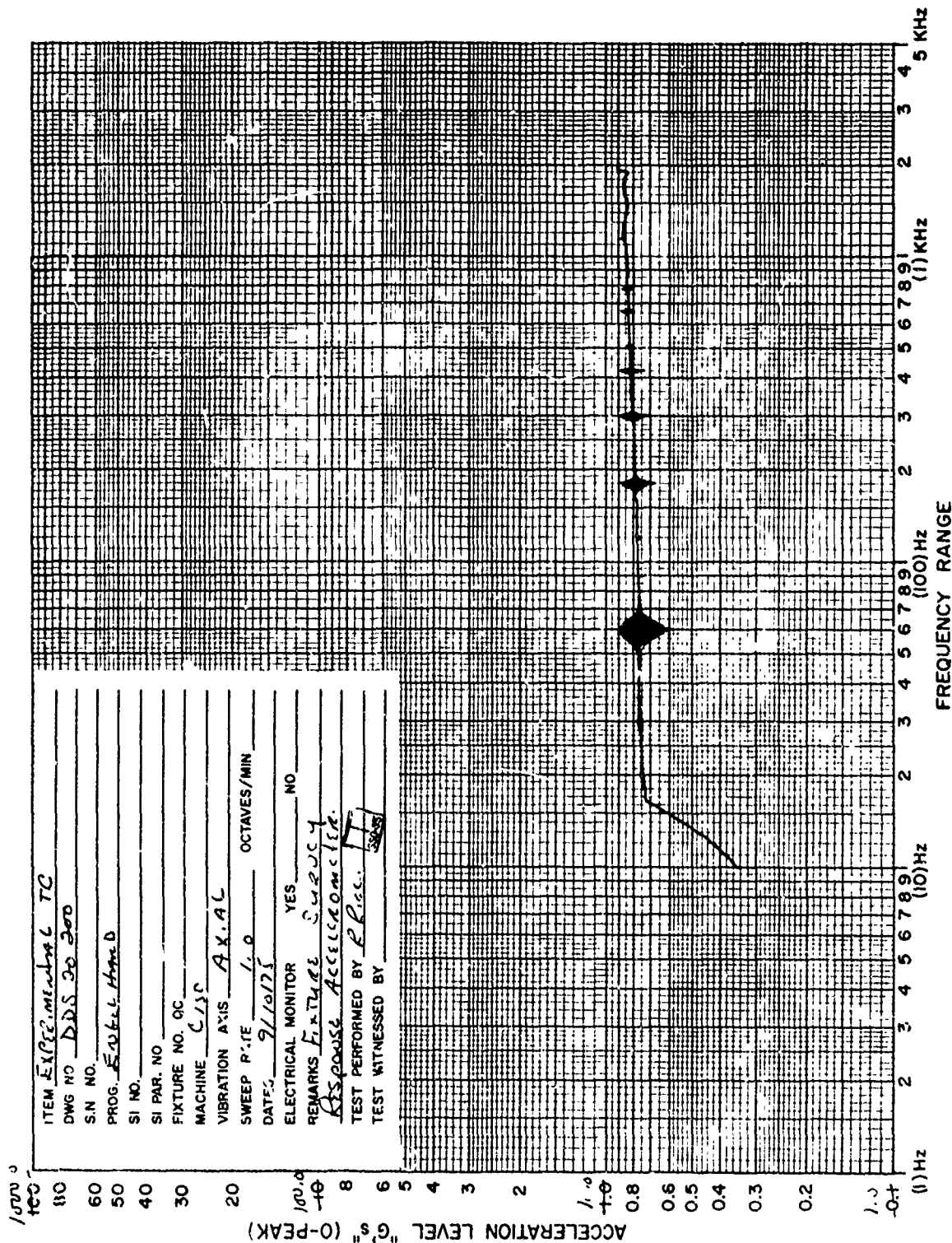
REMARKS TC Ambient Acceleration

TEST PERFORMED BY R.R. W.

TEST WITNESSED BY _____







ITEM Experimental TC
DWG NO. DD50000
S/N NO. _____
PROG Engcl Hand
SI NO. _____
SI PAR. NO. _____
FIXTURE NO. 00
MACHINE C150
VIBRATION AXIS AXIAL
SWEEP RATE 1.0 OCTAVES/MIN
DATES 9/10/73
ELECTRICAL MONITOR YES ☐ NO ☐
REMARKS Final Acceptance
TEST PERFORMED BY R. C. C. H.
TEST WITNESSED BY _____

ACCELERATION LEVEL "G's" (0-PEAK)

FREQUENCY RANGE

(1) Hz (10) Hz (100) Hz (1) KHz 5 KHz