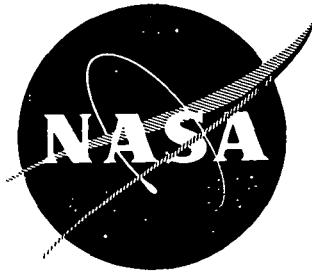NASA CR-120990
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STUDY OF PERFORMANCE CHARACTERISTICS
OF NOBLE METAL THERMOCOUPLE MATERIALS
TO 2000°C

BY

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16. Abstract

This report describes the work performed to date on the Noble Metal Thermocouple Research Program. The study involves three performance characteristics of noble metal thermocouples in oxidizing media, such as air and air containing certain incompletely-burned gases.

The study of Characteristic I, catalytic effects, indicated that significant errors can result when noble metal thermocouple materials are exposed to air containing unburned gases under laboratory conditions in the temperature range 25°C to 1500°C. Characteristic II is the thermoelectric stability of the iridium 40 rhodium to iridium thermocouple system at temperatures up to 2000°C in oxidizing media, such as air. Preliminary study results show a change in the system's thermoelectric output of less than 1% at a calibrating furnace temperature of 1700°C, after cumulative heating time of 359 hours at 1700°C. Characteristic III comprises the effects of large and small temperature gradients on the accuracy and stability of temperature measurements. A facility for study of this characteristic is under construction.

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METAL THERMOCOUPLE MATERIALS TO 2000°C

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National Bureau of Standards

SUMMARY

The research program sponsored by NASA involves investigation of three performance characteristics of noble metal thermocouples: 1) effects of catalysis; 2) effects of oxidizing atmospheres; 3) effects of severe and mild temperature gradients.

Characteristic I is the effect of catalysis on the temperature indication of noble metal temperature sensors. Measurements have been made of the effects of catalysis when platinum and platinum 13 rhodium thermoelements at temperatures ranging from 1000°C to 1500°C were exposed to a mixture of air and unburned gases flowing at gas velocities of 17 to 183 m/s. The thermoelements were exposed to the following mixtures by volume:

(a)   air + 1.0% carbon monoxide + 0.01% hydrogen

(b)   air + 2.0% carbon monoxide + 0.01% hydrogen

(c)   air + 1.0% hydrogen

(d)   air + 2.0% hydrogen

Measurements obtained with the gas mixtures indicate that the thermoelement temperature rise caused by catalytic heating increases with increasing gas velocity up to a velocity of 120 m/s. The temperature rise tends to decrease with further increase of velocity. At gas velocities ranging from 17 to 183 m/s, the rise in temperature (ΔT) resulting from catalysis ranged between 35°C and 380°C.

Experimental observations suggested that the effects of catalysis would reach a maximum value at some temperature below 1000°C. To evaluate the temperature of maximum effect, additional measurements were therefore made of the catalytic heating of various noble metal materials considered for use in high temperature thermocouple systems. The materials chosen were:

(a)   platinum

(b)   platinum 13 rhodium

(c)   iridium

(d)   iridium 40 rhodium

(e) Platinel 5335

(f) Platinel 7674

These thermoelements were exposed to mixtures of air + 0.5% butane flowing at 0.1 and 0.2 m/s. Measurements were made from 25°C to 1450°C to determine the maximum temperature rise caused by catalytic heating and the temperature at which the effect of catalysis becomes insignificant. The results show that platinum exhibits the largest temperature rise. The maximum rise in wire temperature ($\Delta T=165°C$) occurred upon exposure to a mixture of air + 0.5% butane flowing at 0.1 m/s of a sample wire that in air alone at the same velocity assumed a temperature of 240°C. The catalytic effect (temperature rise) dropped off rapidly below this level until at a wire temperature of 185°C it was found to be insignificant. The maximum rise in wire temperature occurred at different sample temperatures for the materials investigated. Platinum 13 rhodium exhibted the smallest increase ($\Delta T=85°C$) at a sample temperature of 1090°C in a similar gas mixture flowing at the same velocity.

Characteristic II is the thermoelectric stability of the iridium 40 rhodium to iridium thermocouple system for measuring temperature up to 2000°C in oxidizing media. Measurements were made at a furnace temperature of 1700°C. One thermocouple was heated in a nitrogen atmosphere and the other in an air atmosphere. In this investigation, nitrogen was the neutral reference environment. The behavior of a thermocouple in nitrogen was compared with the behavior of a similar thermocopule in an oxidizing atmosphere of air. The thermoelectric output of the thermocouple which was heated in air exceeded that of the sample in nitrogen. After a total heating time of 359 hours at 1700°C, the difference between the temperature indications of the two thermocouples at a calibrating temperature of 1700°C was equivalent to about 9.4°C which is less than 1% of the calibration temperature.

Characteristic III is the effect of steep and moderate temperature gradients on the accuracy and stability of thermocouple measurements. A test facility for this study is under construction. A Nd:YAG laser will be used to raise the thermocouple junction of a water-cooled probe to 2000°C. The concentration of heat applied in this manner to the thermocouple junction permits both gradual and steep gradients to be established along the thermocouple lead wires. A water-cooled test cell capable of systematically positioning the thermocouple is under construction. The necessary instrumentation has been acquired. The assembly of the necessary components is in progress.

INTRODUCTION

The importance of accurate temperature measurements in the development and operation of jet engines is well recognized. Such measurements determine the important operating characteristics of the engine and permit the designer to identify for special attention component changes that would most effectively improve engine performance. As engine temperatures have increased, a need has arisen for temperature sensors, such as thermocouples, capable of sustained operation in hot oxidizing gases. For systems operating in the temperature range 1000°C to 2000°C it becomes necessary to use noble metals such as platinum, rhodium, iridium and their various alloys for thermocouples. Thus, it is important to evaluate the performance characteristics of thermocouples fabricated from these materials which then are used at high temperatures in oxidizing environments containing products of combustion typical of those that exist in the engine. Such an evaluation will bring about a better understanding of the problems encountered in measuring temperatures under static and dynamic conditions and thus help to minimize temperature errors. Three performance characteristics have been selected for study.

Characteristic I is a study of the effect of catalysis on the temperature of noble metal thermoelements, particularly platinum and platinum 13 rhodium. Thermocouples made from noble metals and exposed to various incompletely-burned gases that exist in some engine exhaust will experience catalytic burning on the surface of the thermocouple. This catalytic reaction will raise the temperature of the thermocouple higher than the ambient gas temperature being measured. Various investigators, Dahl and Fiock [1]*, Stanforth [2], Olsen [3], Freeze, et al. [4] and R. L. Ash, et al. [5], have observed and reported this chemical reaction effect.

The results of the Characteristic I studies at NBS under this contract provide information over the originally planned temperature range of 1000°C to 1500°C, for various gas mixtures flowing at velocities between 17 and 183 m/s. The investigation suggested that catalysis error probably reaches a maximum at some temperature below 1000°C. Earlier studies reported by Olsen [3] showed that the temperature increase of a thermoelement reached a maximum at a temperature below 760°C and that the effect became negligible below 320°C. In view of these findings, and to augment the previous studies, the catalytic effects of various noble metal thermocouple materials were determined over the extended temperature range 25°C to 1450°C.

Characterisitic II is a study of the performance of a thermocouple system at temperatures up to 2000°C in oxidizing media. At present only thermocouples made of iridium and alloys of iridium appear suitable for use under these conditions.

Feussner [6] in 1933 proposed the use of iridium-rhodium to iridium thermocouples "for very high temperatures". Several alloys have been

[1]* Figures in brackets refer to literature references on page 20 of this paper.

proposed by various experimenters for the positive wire of the thermocouple, chief among them being iridium-rhodium alloys containing 40, 50 and 60% of rhodium. The thermal emf's of wires of these compositions are not widely different. The thermal emf of the iridium 50 rhodium wire is slightly higher than that of the other two compositions (iridium 60 rhodium or iridium 40 rhodium). Carter [7] postulated that at high temperatures, the iridium would volatilize preferentially from the iridium-rhodium alloy and the alloy would change to one enriched in rhodium. A change in emf resulting from the loss of iridium from the iridium 40 rhodium wire will alter the emf to a maximum at iridium 50 rhodium composition. Additional loss of iridium would slightly decrease the emf and thus the thermal output of this alloy would remain satisfactory for a longer time than either of the others. It is also expected that the high temperature limit will be somewhat greater for the alloy containing more iridium.

Blackburn and Caldwell [8] studied the various alloys of iridium-rhodium to iridium and developed comprehensive reference tables for use with thermocouples made of these materials. Their work did not yield results indicative of the thermoelectric stability of various combinations nor did they identify a preferred composition.

There is very little information available on the thermoelectric stability of iridium and alloys of iridium in oxidizing atmospheres. Rudnitskii and Tyurin [9] observed changes in thermal emf of the iridium 60 rhodium to iridium system after 10 hours at 1800°C. The total change did not exceed 0.8% of the measured temperature. Aleksaklin et al. [10] observed that "at 2000°C the iridium 60 rhodium to iridium thermocouple allows us with sufficient accuracy to measure the temperature, at least briefly, for a period of 10 - 20 hours."

The current study is concerned with the iridium 40 rhodium to iridium thermocouple. Its thermoelectric stability is being investigated at temperatures up to 2000°C in an oxidizing environment (air).

Characteristic III is a study of the effects of temperature gradients on the accuracy and stability of thermocouple indications. Practical temperature measurements within a stream of hot gas are often complicated by the presence of nonequilibrium conditions and of temperature gradients that may be as great as many hundreds of degrees per centimeter.

The thermal emf developed by a thermocouple of homogeneous thermoelements is a function of the junction temperature alone. However, if a thermoelement is not homogeneous, the emf developed is a function not only of the temperature of the junction, but also of the temperature distribution along the element.

In the current investigation, thermocouple junctions protruding beyond water-cooled sheaths will be exposed to controlled temperature gradients at various temperatures from 1500°C to 2000°C. The effects of severe (1500°C/cm) and moderate (400°C/cm) gradients on themocouple performance will be determined and evaluated.

The test facility for this study is under construction.

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APPARATUS

The study of catalytic effects received first priority. It was conducted in two facilities, one designed for high velocity studies and the other for low velocity investigations. Figure 1 is a cross-section drawing of the high velocity test facility used to determine the catalytic heating effects on test samples at gas velocities between 17 and 183 m/s.

The apparatus is designed to enable pressurized gas to enter a plenum chamber and then flow into a constricted test section in which a 7.6-cm length of the sample is mounted parallel to the gas flow.

Since the test sample in the high velocity facility was mounted between an upper and a lower test sample holder, it was necessary to design a flexible electrical contact that would allow the test sample to expand and contract without flexing, as it changed temperature. To achieve this, the lower end of the test sample was attached to a small molybdenum weight immersed in a metal to which the external electrical contact was made. The metal, was a eutectic mixture, 24.5% indium -75.5% gallium, with a melting point of 16°C.

The dry air and test gas mixtures used in the experiments were obtained from commercial compressed gas cylinders. The nominal content of the minor gas components was specified to be accurate within ±10%. All gas mixtures of air plus carbon monoxide contained 0.01% hydrogen to stabilize the catalytic burning.

All gases were initially passed through a calming chamber consisting of a diffusing plate and a series of six diffusing screens.

The sample temperature was determined from measurements made with a calibrated optical pyrometer.

The catalytic studies over the extended temperature range, 25°C to 1450°C, were conducted at two velocities 0.1 and 0.2 m/s. Figure 2 shows the facility. The apparatus consists of a water-cooled chamber with the test sample mounted in the central region of the chamber and suspended between two electrodes. Located in the side of the chamber is a Pyrex window through which the test sample can be viewed with an optical pyrometer.

A non-turbulent flow was maintained over the test sample by placing in the bottom of the test chamber a silver[1] foil diffuser through which the test gases flowed before passing over the test sample. Again gas mixtures from compressed gas cylinders supplied by a commercial supplier were used.

The test samples were fabricated into a "U" shape with a 1.9-cm length of the sample mounted perpendicular to the gas flow. All test samples

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[1]According to Olsen [3] oxidized silver is non-catalytic.

were 0.5-mm in diameter and 0.25-mm diameter voltage lead wires were
welded to the extreme ends of the 1.9-cm section of each sample. These
lead wires were of the same materials as the respective test samples.

The apparatus used to study the effects of an oxidizing medium on
the thermoelectric stability of the iridium 40 rhodium to iridium
thermocouple system is described by Freeze et al. [4]. Figure 3 shows
the thermocouple facility used for these studies. Figure 4 is a schematic
diagram of the test furnace and its associated equipment. Briefly, the
apparatus consists of a vertical furnace heated by a split tantalum tube
heating element, 10-cm in diameter and 80-cm long, in a helium atmosphere.
During operation of the furnace at temperatures between 1500°C and
2000°C, a hot zone uniform in temperature to within ±1% is maintained
inside the tantalum heating element over approximately 46-cm of its
length. Four zirconia test cells can be located inside the tantalum
element test zone to permit the comparison of two thermocouples in an
atmosphere of nitrogen (neutral reference environment) with two
thermocouples in air.

The furnace is provided with a tantalum well centrally located between
the zirconia test cells. It contains a calibrated tungsten 3 rhenium
to tungsten 25 rhenium thermocouple in a helium atmosphere for
measuring the nominal furnace temperature and provides a reference
signal for the furnace control during the prolonged heating periods.

The temperature of the hot zone is accurately determined by using a
calibrated optical pyrometer sighted on a small tantalum tube black-
body located at the end of the reference thermocouple well. A second
optical pyrometer can be sighted through the upper furnace window with
the tantalum thermocouple well as its target.

Radiation losses from the hot zone are reduced by surrounding the
tantalum heater with six layers of tantalum shield assemblies (top, bottom
and side). The furnace is enclosed in a dual-wall stainless steel water-
cooled jacket.

The furnace can be maintained at any temperature between 200°C and 2300°C.
The instrumentation for controlling the furnace includes a recorder
control system having a combination of manual and automatic modes of
operation. In the automatic mode, the controller and recorder system
utilizes the thermal output of the control thermocouple in the tantalum
well assembly as a reference to maintain a predetermined furnace
temperature within ±1/2%.

Provision is made to circulate either oxidizing or inert gases through
the zirconia test cells containing the sample thermocouples under test.

During the calibration periods the thermal emf's of the sample thermo-couples can be switched to the desired read-out equipment.

Characteristic III Study of the effect of temperature gradient on the accuracy and stability of thermocouple measurements up to 2000°C in oxidizing, neutral and reducing atmospheres received third priority. The apparatus for this investigation is being designed and constructed.

A Nd: YAG laser capable of heating iridium and iridium-rhodium alloys to 2000°C is being installed in the facility. The test section is being designed and constructed. It will consist of a water-cooled chamber so designed that a water-cooled temperature probe having an exposed iridium-rhodium to iridium thermocouple junction can be mounted in the central region of the chamber. Proper alignment assemblies are designed into the facility for locating the thermocouple junction relative to the laser heat source. A window is located in the side of the chamber through which the thermocouple junction and segments of lead wires can be viewed with an optical pyrometer.

Characteristic I Measurements to determine the temperature rise caused by catalytic heating were made in the high velocity facility over the planned temperature range 1000°C to 1500°C. The velocity range was from 17 to 183 m/s. Flow velocity in the test section was determined by measuring the total pressure, static pressure, and temperature of the gas.

The test samples selected for the study were 0.5-mm diameter platinum and platinum 13 rhodium. These noble metal elements were exposed to the following gas mixtures by volume:
(a) air + 1.0% carbon monoxide + 0.01% hydrogen
(b) air + 2.0% carbon monoxide + 0.01% hydrogen
(c) air + 1.0% hydrogen
(d) air + 2.0% hydrogen
(e) air

During a test, pressurized dry air is released into the calming chamber where it strikes a diffusing plate and flows through a series of diffusing screens which reduce the turbulence. With the dry air flowing at a predetermined velocity over the thermoelement sample located in the nozzle section, the electric current is increased until the sample reaches a selected temperature in the desired range. The thermoelement temperature is accurately determined using an optical pyrometer. This procedure is repeated with a test mixture containing unburned gas flowing over the sample at the same velocity as the dry air and with the same electric current through the test sample. The difference between the two measured temperatures is attributed mainly to catalytic combustion on the surface of the sample. A small part of the temperature increase is attributed to increased I^2R heating resulting from the increase of sample resistance with temperature (about 5% of the temperature indication in the case of the highest observed catalytic effect of a platinum wire exposed to air +2.0% hydrogen at 1380°C and a flow of 183 m/s.)

To verify that the increase in sample temperature of the platinum and the platinum-rhodium alloy resulted from catalysis, a sample was prepared of gold wire known to be non-catalytic. Although the wire failed mechanically at a velocity of 31 m/s, measurements obtained at lower velocities indicated no temperature rise in air + CO mixture at temperatures up to the melting point of the gold sample.

Catalytic effects in the extended range from 25°C to 1450°C were studied in the low velocity facility with test-gas velocities of 0.1 and 0.2 m/s. The general experimental procedure for these studies was similar to that described above for the high velocity facility. In the range 950°C to 1450°C the temperature of the test sample was measured with an optical pyrometer. Lower temperatures, below the range of the optical pyrometer, were measured by making use of the relationship between the sample's electrical resistance and its temperature. This relationship was derived for each test sample from measurements of electrical resistance at 25°C and in the range 950°C to 1450°C.

The catalytic effects were evaluated for six noble metal thermocouple materials: Platinum, platinum 13 rhodium, iridium, iridium 40 rhodium, Platinel 5355[2], and Platinel 7674[2]. (The composition of Platinel 5355 is stated by the manufacturer to be 55% palladium, 31% platinum and 14% gold, and of Platinel 7674 to be 65% gold and 35% palladium.)

As previously discussed, the difference between the temperature of the sample exposed to a gas mixture and its temperature in air when carrying the same electric current is attributed largely to catalytic combustion at the sample surface. A small fraction of the temperature difference is attributable to increased electrical power dissipation in the sample at the higher temperature as a result of the increased sample resistance with temperature. This increased power dissipation is significant at the lower test temperatures and corrections were applied to the observed sample temperatures in the gas mixture to permit a closer estimate of the temperature rise attributable to catalysis.

<u>Characteristic II</u> The thermoelectric stability of the iridium 40 rhodium to iridium thermocouple system is being studied by comparing in the high temperature furnace facility the thermal emf of a thermocouple immersed in a test cell containing an atmosphere of nitrogen (reference neutral atmosphere) with that of a thermocouple in a test cell containing an oxidizing environment such as air. The thermocouples are first calibrated by immersing them in the isothermal zone so that their junctions are located in the plane of the center of the upper window of the furnace. Then, for the prolonged heating phase of the test, the aging period, the thermocouples are fully immersed in their respective zirconia test cells to a position in the plane of the center of the lower sight window. For recalibration the thermocouples are withdrawn to their previous positions. Thus, the wire segments of each thermocouple that are exposed to any temperature gradient during aging can not contribute to the thermoelectric output so that the observed changes of thermal output can be unambiguously attributed to aging at the desired test temperature.

An optical pyrometer is sighted through each sight window to determine the temperatures at the calibrating and aging positions of the test samples. Proper corrections are applied for window transmission losses and for emissivity of the tantalum tube at the upper window.

Measurements have been obtained on thermocouples aged at 1700°C. Two thermocouples were prepared from continuous segments of iridium 40 rhodium and iridium wires, 0.8-mm in diameter. Each thermoelement was first throughly cleaned using a soapy water rinse and finally a second rinse with ethyl alcohol. The wires were then annealed in air at 1800°C for five minutes. Thermocouples were formed by welding with an air-hydrogen flame. The lead wires of the thermocouples were threaded through spiders fabricated of iridium disks and beryllia spacers. Since the

[2]Registered trade name of Engelhard Minerals and Chemical Corporation. Platinel 5355 is thermoelectrically positive and Platinel 7674 is thermoelectrically negative relative to platinum.

axes of the thermocouples are vertical the mass of these spiders provide tension in the thermocouple wires. The geometry of the spiders center the wires within each zirconia test cell, thereby preventing contact between the wires and the inner surface of the cells when the thermocouples are moved from the calibrating to their aging positions.

Two zirconia test cells were cleaned by heating the furnace to 1600°C and then cooling in their respective atmospheres of air or nitrogen. A thermocouple was then located 32.0-cm from the bottom of each test cell, the calibrating station for the test. To insure the desired atmosphere for each cell a positive flow of gas, 1 cm^3/s was maintained at all times.

The furnace was then heated to a temperature of 1200°C. Measurements were made to determine the temperature-emf relationship of the two thermocouples, one in the oxidizing atmosphere and the other in the nitrogen atmosphere. Observations were made at 50°C increments as the temperature was increased over the range 1200°C to 1700°C.

The thermocouples were then immersed 30.5-cm deeper into their respective test cells for the initial prolonged heating period. The temperature during the aging period was maintained within ±4°C at 1700°C.

After the initial aging period was completed, the thermocouples were returned to their calibration position for a second determination of their temperature-emf relationships to ascertain whether any drift of the thermoelectric output of the thermocouples had resulted from the heating time. Twelve successive calibrations followed, bringing each total exposure time at 1700°C to 33.0, 49.5, 71.5, 76.5, 82.0, 98.9, 115.6, 116.0, 211.0, 259.0, 309.0, and 359.0 hours, respectively.

After being heated for 359 hours, both thermocouples failed near their junctions while being relocated in their respective aging positions.

RESULTS AND DISCUSSION

<u>Characteristic I</u> The experimental study of catalysis by noble metal thermocouple materials in the high velocity test facility has been completed.

The temperature rise due to catalytic heating was measured for 0.5-mm diameter platinum samples in four gas mixtures, air + 1.0% CO + 0.01% H_2; air +2.0% CO + 0.01% H_2; air + 1.0% H_2; and air + 2.0% H_2, flowing at gas velocities between 17 and 183 m/s and at exposure temperatures from 1000°C to 1400°C.

Figure 5 is a plot showing results typical of those obtained from the experiments. This plot shows the variation of catalytic error for a platinum sample exposed to a mixture of air + 2.0% hydrogen, at various velocities. It shows that the sample temperature rise at a particular gas velocity decreased with increasing sample temperature. Also, the catalytic heating values increased with increasing gas velocity up to approximately 120 m/s and then decreased slightly with further increase of velocity. The possiblity that some of this decrease could be the result of poisoning of the test sample surface during testing has not been investigated.

Figure 6 presents the results of measurements on platinum samples in a gas mixture of air + 1.0% CO +0.01% H_2 and in a mixture of air + 2.0% CO +0.01% H_2, at a gas velocity of 153 m/s. The concentration of a combustible in a given mixture affected the magnitude of the temperature rise due to catalysis. At 1200°C the temperature rise obtained with the 1.0% CO-air mixture was about 85°C compared with 172°C for a 2.0% CO-air mixture, suggesting an approximately linear relationship between the concentration of combustibles and the resulting error.

The catalytic temperature rise was determined for the platinum 13 rhodium wires of 0.51-mm diameter in two gas mixtures (air + 1.0% CO + 0.01% H_2 and air + 1.0% H_2) at velocities between 17 and 183 m/s. Figure 7 shows the variation in catalytic rise for a sample exposed to a gas mixture of air + 1.0% H_2 for the range of velocities investigated. The data obtained for this alloy are quite similar to those for platinum under similar conditions.

Figure 8 presents the catalytic temperature rise for platinum and for platinum 13 rhodium wires of equal diameter exposed to a mixture of air + 1.0% CO + 0.01% H_2 flowing at a velocity 183 m/s. The plot indicates that at a wire temperature of 1200°C there was a temperature rise of about 64°C for the alloy wire and 81°C for the platinum wire. The catalytic error values for platinum 13 rhodium samples are between 80% and 90% of those obtained for the platinum samples.

These studies indicated that the temperature increases of the wire samples tested increased with decreasing element temperature for a given gas mixture flowing at a constant velocity. The results suggested

that the catalytic error values reach a maximum at some temperature below 1000°C. In view of this, studies were conducted to determine the maximum catalytic temperature increases for six thermocouple materials: platinum, platinum 13 rhodium, iridium, iridium 40 rhodium, Platinel 5355 and Platinel 7674. The materials were tested at temperatures over an extended range of 25°C to 1450°C in air, and air + 0.5% butane flowing at velocities of 0.1 and 0.2 m/s. A platinum sample was tested in air, and air + 2.0% hydrogen, at the same velocities.

Figure 9 shows the catalytic temperature rise for the platinum sample in air + 0.5% butane as the temperature is decreased from 1200°C. At a gas velocity of 0.1 m/s, the catalytic rise reached a maximum of 165°C at a sample temperature of 240°C. At 0.2 m/s the maximum rise, 175°C, was reached at 250°C. After reaching a maximum, the temperature increases dropped very sharply to zero at 185°C and 170°C, respectively. For the platinum 13 rhodium sample the temperature rise reached a maximum at relatively high sample temperatures, about 1090°C and 1075°C, and then decreased, but not monotonically, to zero at 360°C. The temperature increases for platinum and the platinum 13 rhodium sample are almost the same in the temperature range of 1100°C to 1200°C although the trends of the curves differ.

Figure 10 is a plot of measurements obtained with a platinum sample exposed to air + 2.0% hydrogen flowing at the same two velocities. In this case, the temperature rise continues to increase with decreasing temperatures, at both gas velocities, to the lowest wire temperature observed, 25°C.

Catalytic rises were determined over the entire temperature range for all six materials in an air + 0.5% butane mixture at the two velocities. Table 1 summarizes the highlights of the test results; (a) the sample temperature where the temperature rise, ΔT, is a maximum, (b) the maximum ΔT and (c) the sample temperature where ΔT is zero. Also included in Table 1 are the results with a sample of platinum exposed to a mixture of 2.0% hydrogen in air.

It is noted that during the efficient operation of modern jet engines, the concentrations of carbon monoxide, hydrogen and hydrocarbons in the exhaust gas are a hundred times less than the concentrations used in this study and thus, the catalytic heating of the thermoelements in efficiently operated jet engines will be lower. In research on jet engine combustors, however, high concentrations of incompletely-burned gases may be encountered. In spark-ignition internal combustion engines, for very rich mixtures the hydrogen and carbon monoxide concentration may rise to the order of 10%, while higher-hydrocarbon content may rise to 1%.

Characteristic II Observations to determine the thermoelectric stability of the iridium 40 rhodium thermocouple system were begun. Preliminary measurements were made with thermocouples made of 0.8-mm diameter wires. One thermocouple was immersed in a nitrogen reference environment and

TEST SAMPLE IN + AIR + 0.5% BUTANE * AIR +2% H_2	GAS VELOCITY, 0.1 m/s			GAS VELOCITY, 0.2 m/s		
	SAMPLE TEMPERATURE WHERE ΔT IS MAXIMUM (°C)	MAXIMUM ΔT (°C)	SAMPLE TEMPERATURE WHERE ΔT = 0 (°C)	SAMPLE TEMPERATURE WHERE ΔT IS MAXIMUM (°C)	MAXIMUM ΔT (°C)	SAMPLE TEMPERATURE WHERE ΔT = 0 (°C)
+ PLATINUM	240	165	185	250	175	170
+ Pt - 13% Rh	1090	85	360	1075	95	360
+ IRIDIUM	1280	105	525	1280	116	480
+ Ir - 40% Rh	595	100	350	585	110	315
+ PLATINEL 5355	480	145	290	480	145	210
+ PLATINEL 7674	735	100	515	760	100	515
* PLATINUM	25	360	---	25	360	---

TABLE 1 - SELECTED TEST RESULTS OF CATALYTIC ERRORS OF VARIOUS NOBLE METAL MATERIALS OVER AN EXTENDED TEMPERATURE RANGE

the other in an oxidizing medium. Both were aged in an isothermal zone for a cumulative heating time of 359 hours at 1700°C. Each of the two thermocouples were initially calibrated using an optical pyrometer to obtain the true temperature. The temperature-emf relations derived from this initial calibration were used as a reference for all subsequent measurements.

Figure 11 presents a summary of the cumulative changes in thermoelectric output at a check temperature of 1700°C resulting from aging exposures of the thermocouples at 1700°C. The changes represent the differences between the thermocouple in a nitrogen environment and the thermocouple located in air, the oxidizing environment. According to the convention adopted here the negative sign indicates that the thermocouple in air has the larger thermoelectric output at the check temperature. The calibration of the thermocouple in the reference nitrogen atmosphere remained stable throughout the successive aging periods. The changes in thermal emf was never greater than 7 microvolts from the initial calibration of the thermocouple. From the plot, it is evident that the thermal emf of the thermocouple in the oxidizing environment continued to increase for about the first 70 hours of heating time. During the next 45 hours there was no significant change in thermocouple emf. Additional heating time resulted in a continuing decrease in emf output of the thermocouple, suggesting that the thermoelectric output is maximum from 70 to 115 hours.

These changes in the temperature-emf relation of the thermocouple resulting from the cumulative heating time at 1700°C suggest some instability in the iridium 40 rhodium to iridium thermocouple system. The change in thermoelectric output, corresponding to a change in temperature indication that is less than 1% of the aging temperature, may be caused by a change in composition of one of the components of the alloy element resulting from selective oxidation and evaporation.

After 359 hours of cumulative heating, the wires of both thermocouples had undergone structural changes making them extremely brittle and causing them to fail. Sample lengths of wire were cut from each element of the two thermocouples for microstructure examinations. Segments were obtained from each of four elements about 0.3-mm from each junction extending in length to about 2.4-cm from the junction. Such samples are referred to as "near junction". Also the alloy leg exposed to the air atmosphere showed oxidation and some melting had occurred along the wire surface adjacent to the junction. About 7.5-cm from the junction a reddish-brown coating was observed on this alloy element. A segment was cut about 7.5 to 9.5 cm from the junction "in uniform zone" for examination and comparison. Macrographs and photomicrographs were made of the elements in their post anneal condition and again following prolonged heating at 1700°C for 359 hours. Figure 12 presents three pictures of wire segments cut from each annealed thermoelement prior to their being formed into the two test thermocouples. The top sample in the macrograph, 12a, shows the surface of the pure iridium element and the bottom sample the iridium 40 rhodium wire. The regions shown were not exposed to high temperatures after being annealed.

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Figures 12b and 12c are longitudinal sections of the iridium and the alloy wires showing microstructure in regions not exposed to high temperatures.

Figure 13 shows photographs of the iridium 40 rhodium wire surface after the 359 hours in air. The changes in structure appear greater than experienced by the other elements in either medium and suggest the possibility that this alloy may have experienced a change in composition as well as structural deterioration. Figure 13a shows the surface of the alloy wire that was exposed to the high temperature in an air atmosphere. The specimen shown in the top photograph had a reddish-brown coating. This section was obtained from a region farther from the thermocouple junction than the lower sample, which was directly adjacent to the junction. Figure 13b is a longitudinal section of the alloy wire that was exposed to high temperatures in an air atmosphere. The microstructure reveals a grain boundary phase and large smooth cornered grains which indicate that melting had occurred. Figure 13c shows the grain boundary phase in the photomicrograph, 13b. The evidence of melting suggests that an oxide of rhodium was formed. Figure 13d is a longitudinal section of the same wire farther from the thermocouple junction showing erosion of the surface and the voids near the wire surface. Considerable grain growth is also shown. Figure 13e presents an area having voids along the edge shown in Figure 13d. These voids indicate diffusion of the iridium into the atmosphere faster than it could be replenished by diffusion of the metal behind it. The red-brown color of the coating indicates that it is a rhodium oxide. Such an oxide would be expected to form if the surface is rhodium rich. This would explain the increase in emf of the thermocouple heated in air for the first 70 hours. The maximum emf was reached as the composition presumably approached a 50-50 ratio. If the loss of iridium continued during additional heating time, the rhodium content would eventually become greater in the alloy leg of the thermocouple and its thermal output would slowly begin to decrease, as observed.

In addition to the examinations of the microstructure of the wires, an analysis of composition by the electron microprobe technique was made to compare the compositions of the heated and unheated alloy wires and to examine the edges of the wires heated in air for composition changes. The purity of the iridium sample was also investigated.

Table 2 presents the results of the analysis of the alloy sample. The weight fractions of iridium and rhodium in the unheated wire that served as a reference was taken as 60 iridium 40 rhodium for comparison with the heated wires, but the true composition has not been determined.

The changes in composition of the wire in the nitrogen test environment were negligible, but the alloy in the oxidizing medium appears to have changed appreciably. The grain boundaries near the thermocouple junction of the sample heated in air, Figures 13b and 13c, contained a large concentration of silicon. For the sample taken farther from the junction of the wire heated in air, Figures 13d and 13e, voids were

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observed near the surface of the sample. There was also iridium
enrichment near the surface of the sample shown in **Figure 13e**.

A possible source of silicon is the magnesia stabilized zirconia test
cell. A spectrochemical analysis permits an estimate of silicon content
of 0.1 to 10 percent by weight in our test cells.

TABLE 2. - ANALYSIS OF COMPOSITION OF ALLOY SAMPLE

Speciman	Wt. Fraction Ir	Wt. Fraction Rh
12c (unheated reference)	0.60	0.40
-- (heated in N_2- near junction)	0.61	0.39
-- (heated in N_2- uniform temperature zone)	0.60	0.40
13b (heated in air - near junction)	0.59	0.39
13c (heated in air - grain boundary)	0.20	0.75
13d (heated in air - uniform temperature zone)	0.58	0.38
13e (heated in air near surface)	0.68	0.32
Sample without numbers are not shown in photographs.		

Table 3 presents the weight fractions of iridium found in the iridium
wires. The pure iridium wire not exposed to high temperatures (Figure
12b) was used as a reference for unity concentration of iridium in
an ion microprobe analysis.

Some small composition changes were found. The possibility of con-
tamination of the iridium thermoelement by rhodium vaporized from the
alloy thermoelement was considered, but no rhodium was detected in any
of the wires by the ion microprobe analysis which could detect a rhodium
content as small as 0.1%.

The preliminary data presented in figure 11 suggest the possibility that
the changes in thermal emf with the continued heating periods might

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result from a change in composition in the alloy element of the iridium 40 rhodium to iridium thermocouple. These results could be consistent with the plot shown in figure 14 from a paper by Blackburn and Caldwell [11] which presents the change in thermal emf of iridium-rhodium to iridium thermocouples with alloy composition at a temperature of 1316°C. It is apparent that a loss of iridium from the alloy results in an increase in thermal emf; reaching a maximum at the iridium 50 rhodium composition. Thereafter, as the alloy becomes more rhodium enriched, the emf decreases.

However, the results of the examinations of the microstructure of the wires and the analysis of their compositions did not conclusively support this hypothesis.

TABLE 3. - ANALYSIS OF COMPOSITION OF IRIDIUM SAMPLE

Speciman	Wt. fraction Ir.
12b (unheated reference)	1.00 (by definition)
-- (heated in N_2 - uniform temperature zone)	0.93
-- (heated in N_2 - near junction)	1.00
-- (heated in air - uniform temperature zone)	0.98
-- (heated in air - near junction)	0.98
Samples without numbers are not shown in photographs.	

Characteristic III The design and construction of the test facility for the study of gradient effects is underway. Adapters were constructed to incorporate the heat source into the test rig. The heat source, a Nd:YAG laser, is capable of heating the test thermocouple junction to 2000°C. A 10-cm focal length objective lens is used to focus the laser beam on the thermocouple junction.

A water-cooled test section is being constructed to house the test probe which is also water-cooled. The noble metal thermocouple elements extend about 2.5-cm beyond the end of the water-cooled probe where the wires are welded into a junction. Means were provided so that the test probe can be accurately aligned with the concentrated beam from the laser

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source. A viewing window in the test facility permits optical pyrometer measurements. This window permits temperature determinations to be made at the thermocouple junction and at desired positions along the lead wires of the thermocouple to observe the gradients, mild and severe, that have been established. Gradients along the test thermocouple elements ranging from 400°C/cm to 1500°C/cm can be achieved by varying the length of exposed thermocouple elements protruding from the water-cooled probe.

CONCLUDING REMARKS

1. The results of these studies indicate that the accuracy of thermo-
   couples made from noble metal materials (platinum, platinum 13
   rhodium, iridium, iridium 40 rhodium, Platinel 5355 and Platinel
   7674) used under similar conditions can be appreciably affected by
   catalytic heating errors. The data indicate that the magnitude of
   catalytic error is dependent on (a) velocity of the gas medium (b)
   concentration of the combustible in the air-gas mixture (c) type
   of combustible in the mixture, and (d) temperature of the noble metal
   thermocouple.

2. Preliminary observations indicate that the iridium 40 rhodium to
   iridium thermocouple combination can maintain its thermoelectric
   stability within 1% of the test furnace temperature, 1700°C, for a
   cumulative heating time of 359 hours, at one atmosphere pressure
   of essentially still air as the oxidizing medium.

   The wires of both the reference thermocouple in a nitrogen environ-
   ment and the test thermocouple in air suffered structural changes
   associated with enlarged grain growth. These changes resulted in
   wire failure after 359 hours. This may have been caused by contamina-
   tion from impurities, such as silicon, or from phase changes in the
   wire structure due to the cumulative heating time.

   The data on the instability effect and structural deterioration of the
   thermocouple wires presented in this report are not complete. More
   data will be obtained at higher temperatures to determine the
   usefulness of the iridium 40 rhodium to iridium thermocouple system
   under oxidizing conditions in the temperature range 1700°C to 2000°C.
   These additional data will permit more generalized conclusions to be
   drawn on the identity of the sources of error which affect thermo-
   electric stability and life expectancy of this thermocouple system
   and to suggest means of reducing them.

3. Evaluation of the effect of mild and severe temperature gradients
   on the indications of noble metal thermocouples will be continued.

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     40 Percent Iridium-60 Percent Rhodium Versus Iridium
     Thermocouples", J. Res. Nat. Bur. Stand. - Engineering
     and Instrumentation, 66C, No. 1 (January - March, 1962).

LIST OF FIGURES

21

POWER LEAD

SCALE 1 cm = 2.3 cm

0 1 2 3

WATER COOLED
SUPPORTS

WATER
PORT

STATIC
PRESSURE TAP

VIEWING WINDOW

THROAT SECTION

TEST SAMPLE

WATER
PORTS

POWER LEAD

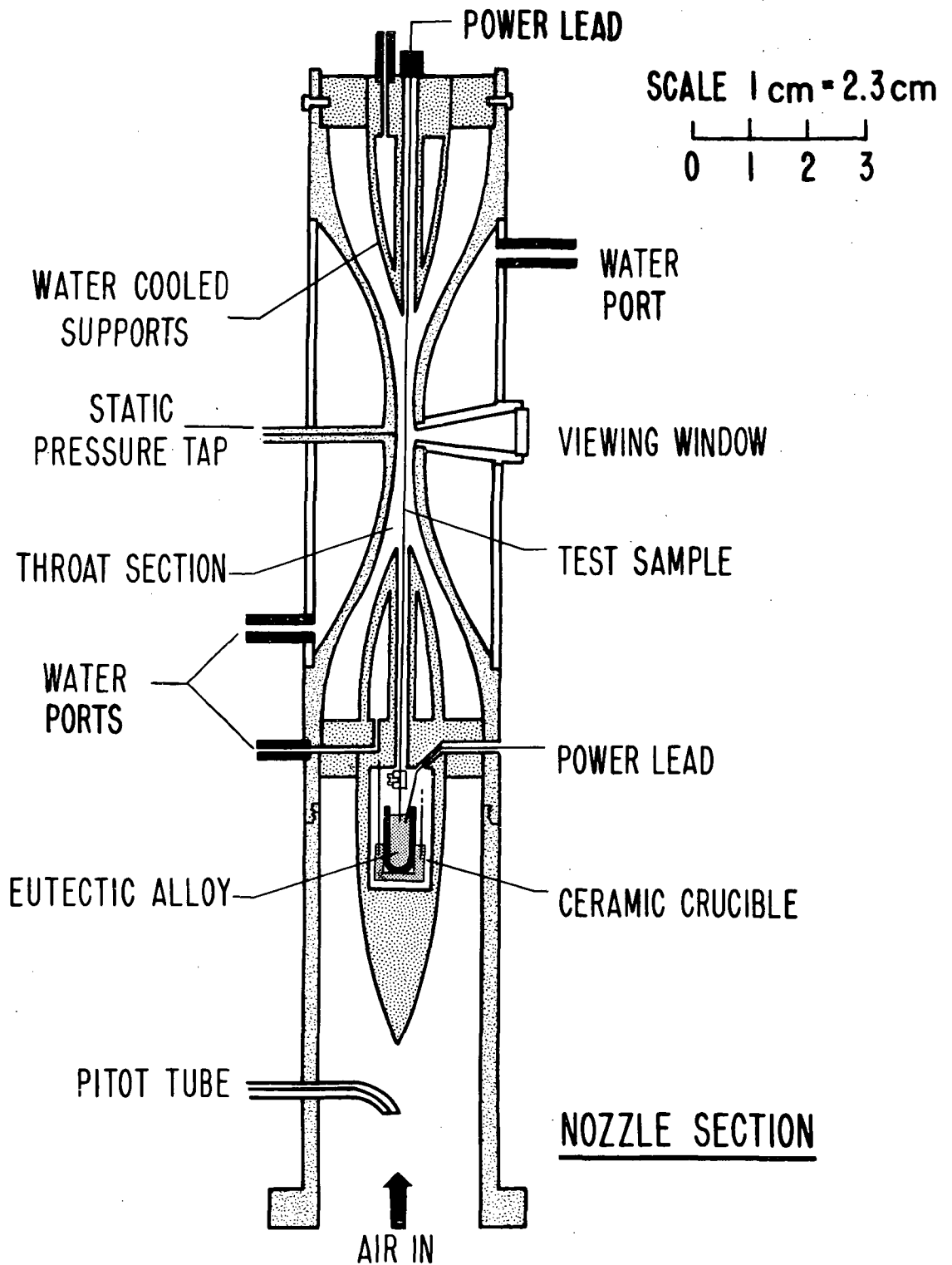EUTECTIC ALLOY

CERAMIC CRUCIBLE

PITOT TUBE

NOZZLE SECTION

AIR IN

FIGURE 1. – TEST SECTION OF HIGH VELOCITY FACILITY

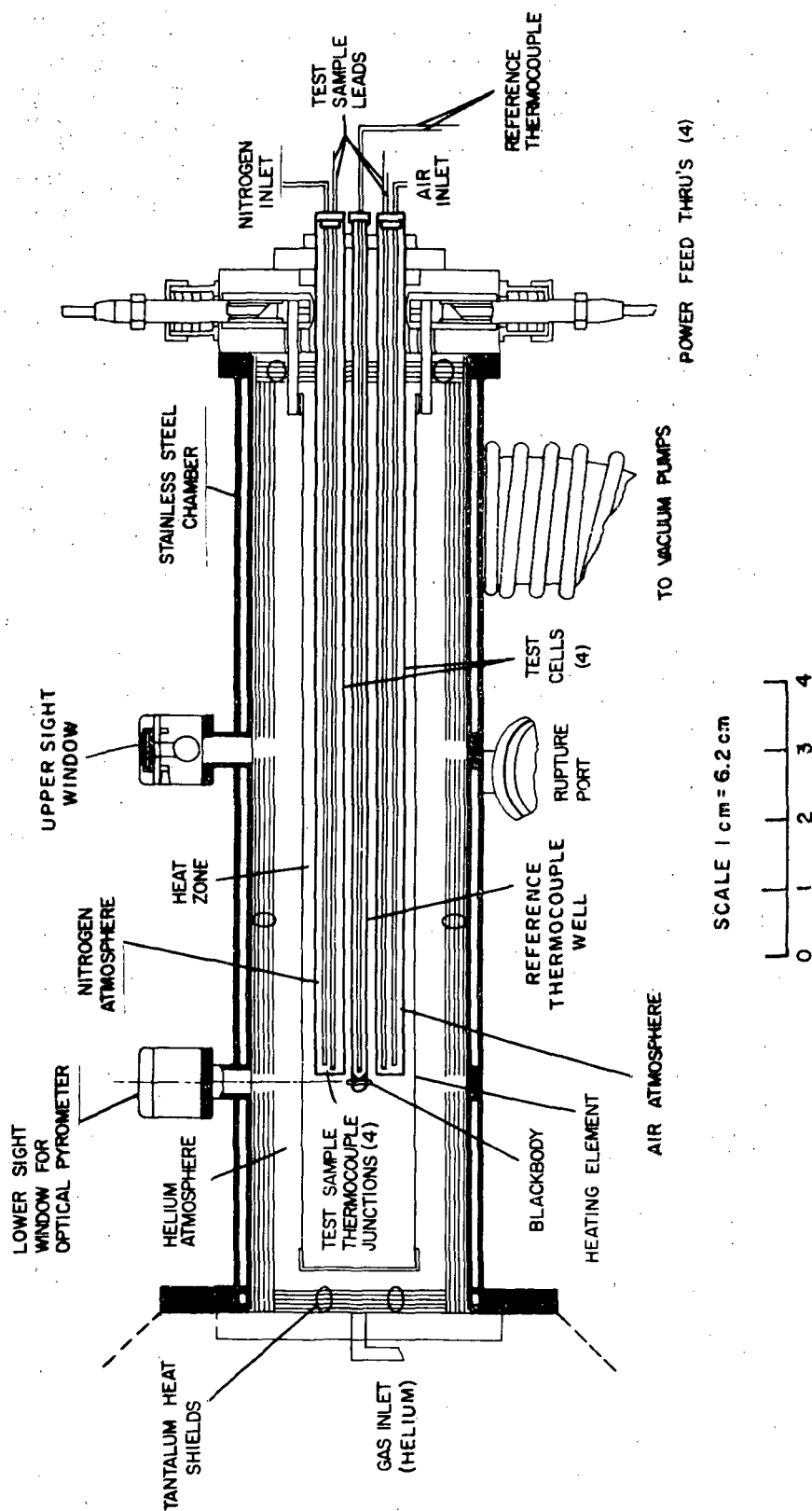FIGURE 2.- SCHEMATIC DIAGRAM OF LOW VELOCITY FACILITY
WITH TEST ELEMENT IN POSITION

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NITROGEN INLET

TEST SAMPLE LEADS

REFERENCE THERMOCOUPLE

AIR INLET

POWER FEED THRU'S (4)

STAINLESS STEEL CHAMBER

UPPER SIGHT WINDOW

TEST CELLS (4)

RUPTURE PORT

TO VACUUM PUMPS

HEAT ZONE

NITROGEN ATMOSPHERE

REFERENCE THERMOCOUPLE WELL

AIR ATMOSPHERE

SCALE I cm = 6.2 cm

0 1 2 3 4

LOWER SIGHT WINDOW FOR OPTICAL PYROMETER

HELIUM ATMOSPHERE

TEST SAMPLE THERMOCOUPLE JUNCTIONS (4)

BLACKBODY

HEATING ELEMENT

TANTALUM HEAT SHIELDS

GAS INLET (HELIUM)

FIGURE 3.–THERMOCOUPLE FACILITY FOR STABILITY STUDIES

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FIGURE 4.– SCHEMATIC OF FURNACE AND ASSOCIATED EQUIPMENT

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FIGURE 5.- VARIATION IN CATALYTIC EFFECTS FOR 0.5-mm DIAMETER PLATINUM WIRE EXPOSED TO AIR+2.0 % HYDROGEN MIXTURE FOR VARIOUS FLOW VELOCITIES

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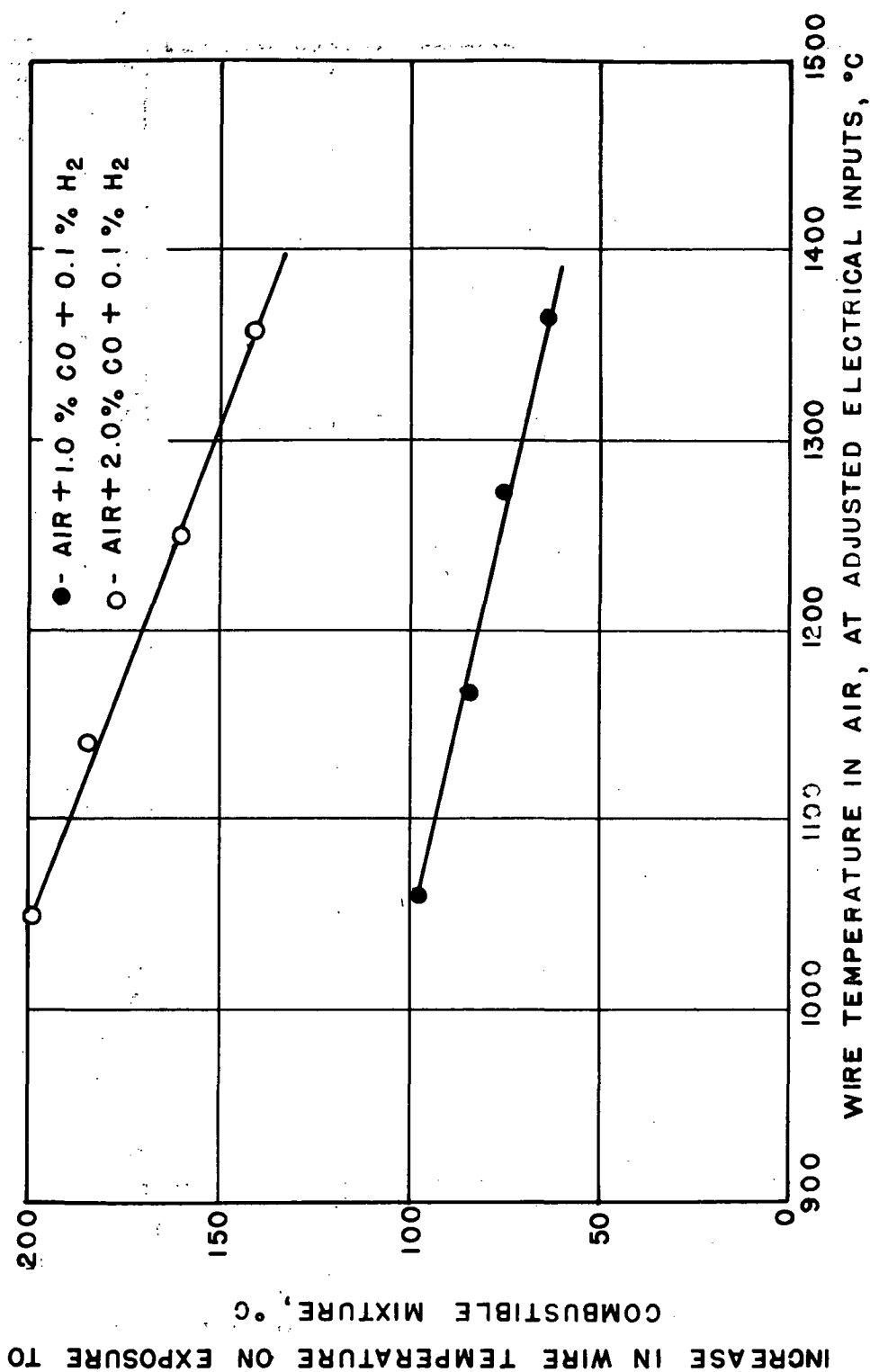
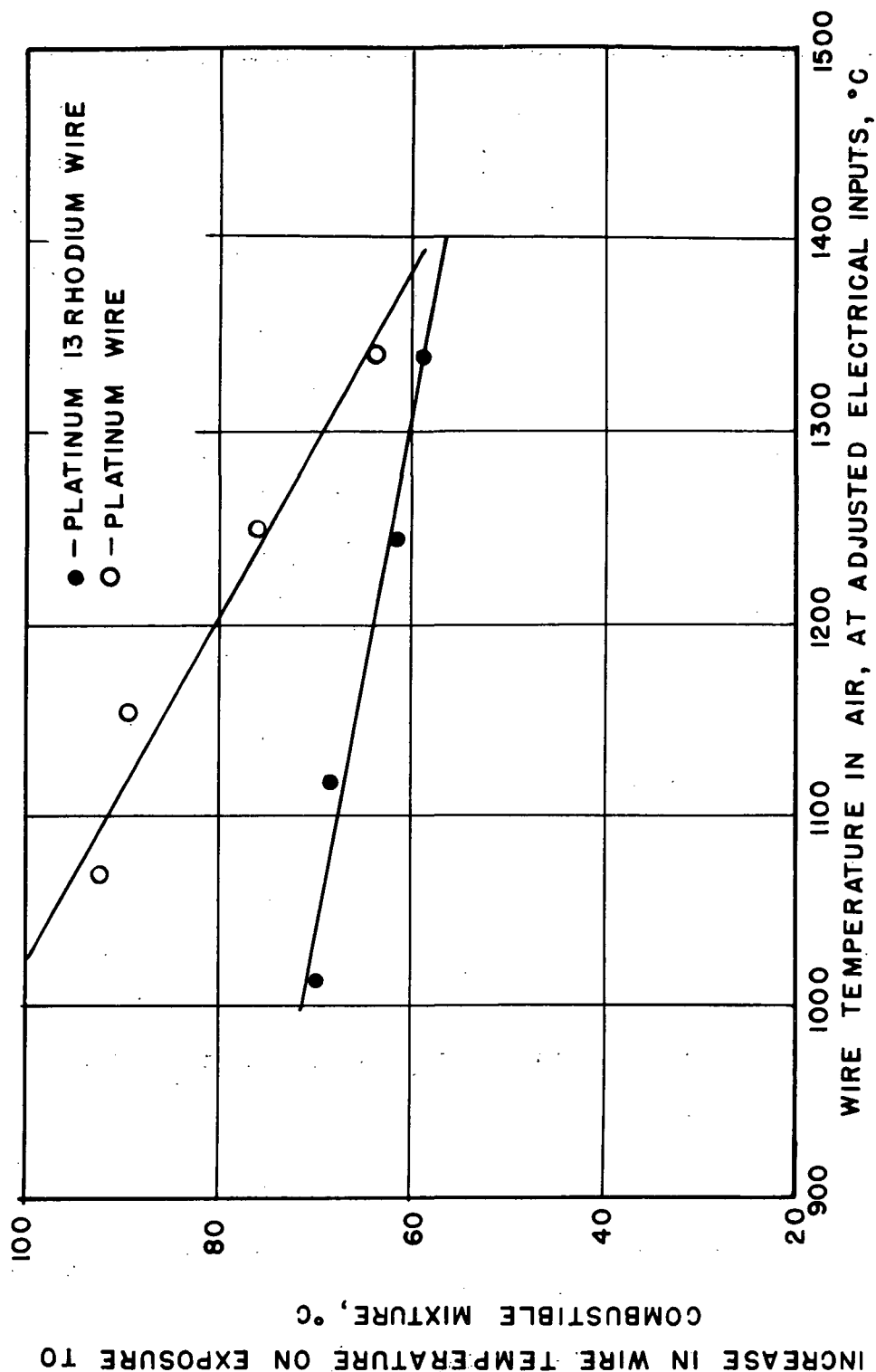FIGURE 6.- CATALYTIC EFFECTS FOR 0.5-mm DIAMETER PLATINUM WIRES IN AIR-CARBON MONOXIDE MIXTURES FLOWING AT 153 m/s
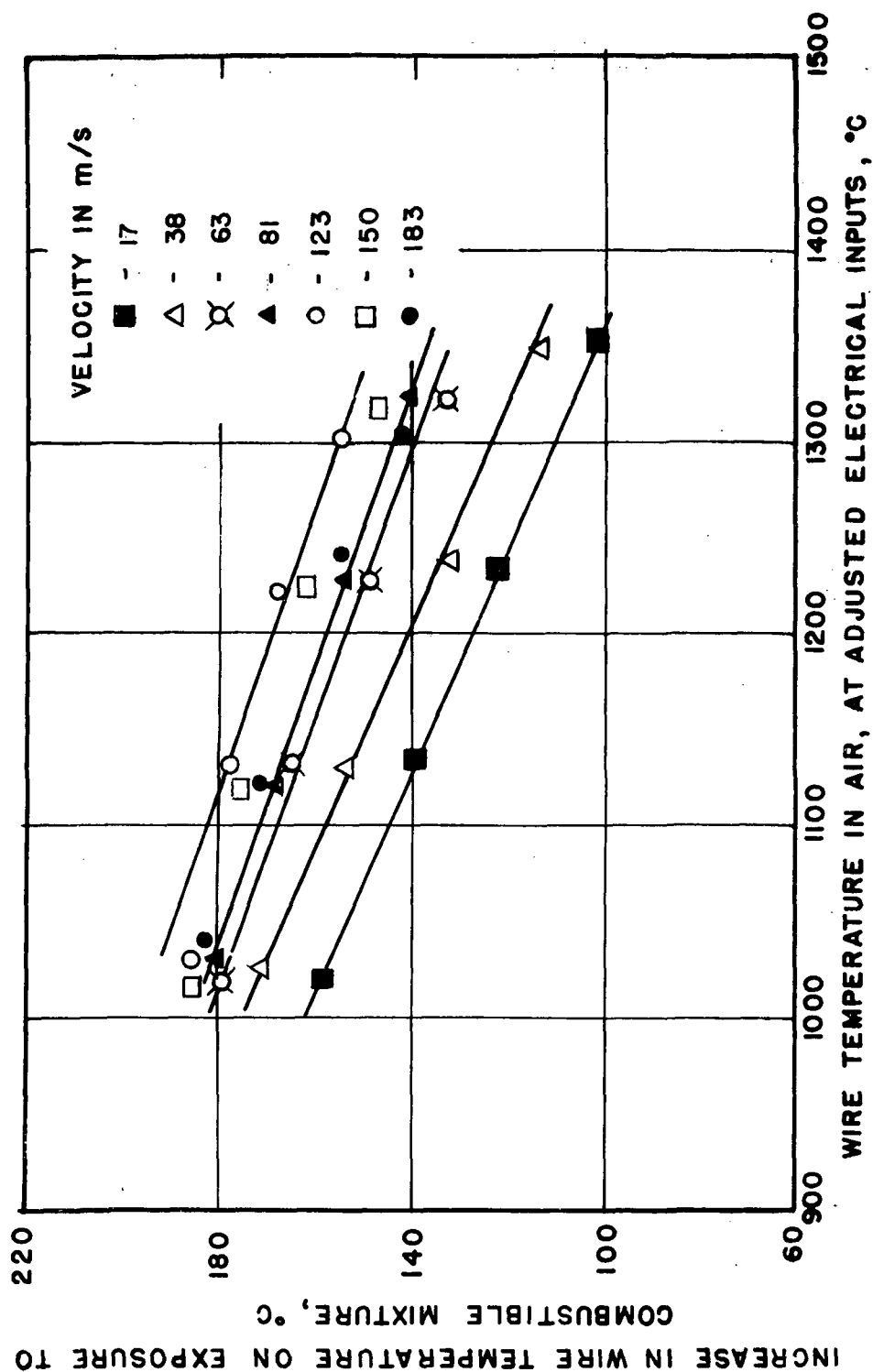
FIGURE 7.- VARIATION IN CATALYTIC EFFECTS FOR 0.5-mm DIAMETER PLATINUM 13 RHODIUM WIRE EXPOSED TO AIR+1.0 % HYDROGEN MIXTURE FOR VARIOUS FLOW VELOCITIES

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FIGURE 8.— CATALYTIC EFFECTS FOR 0.5-mm DIAMETER NOBLE METAL WIRES IN AIR + 1.0 % CO + 0.1 % H_2 MIXTURE FLOWING AT 183 m/s

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PLATINUM WIRE

----- PLATINUM 13 RHODIUM WIRE

● - VELOCITY 0.1 m/s

○ - VELOCITY 0.2 m/s

WIRE TEMPERATURE IN AIR, AT ADJUSTED ELECTRICAL INPUTS, °C

200

160

120

80

40

0

0   200   400   600   800   1000   1200

INCREASE IN WIRE TEMPERATURE ON EXPOSURE TO COMBUSTIBLE MIXTURE, °C
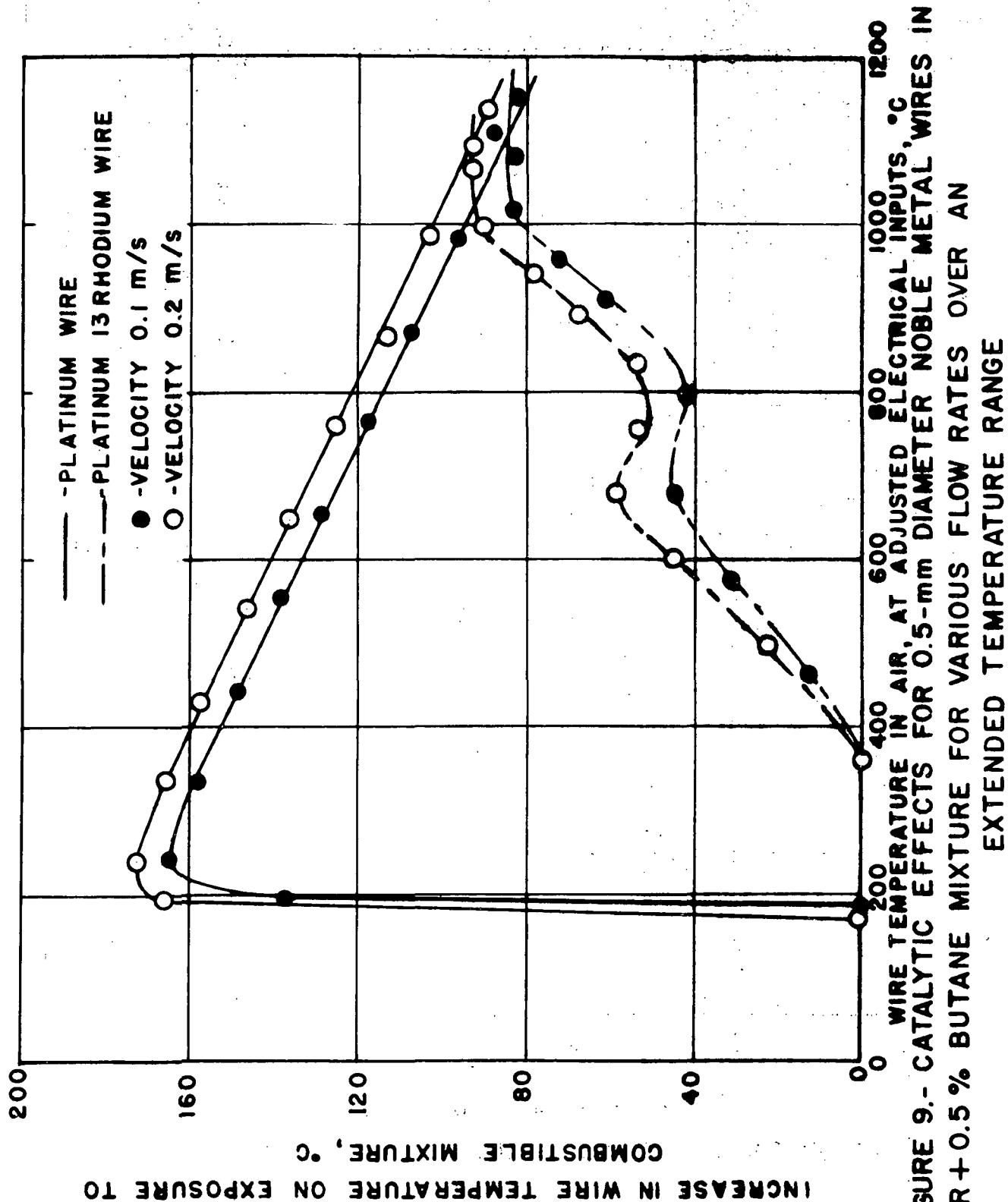
FIGURE 9.- CATALYTIC EFFECTS FOR 0.5-mm DIAMETER NOBLE METAL WIRES IN AIR + 0.5 % BUTANE MIXTURE FOR VARIOUS FLOW RATES OVER AN EXTENDED TEMPERATURE RANGE

30
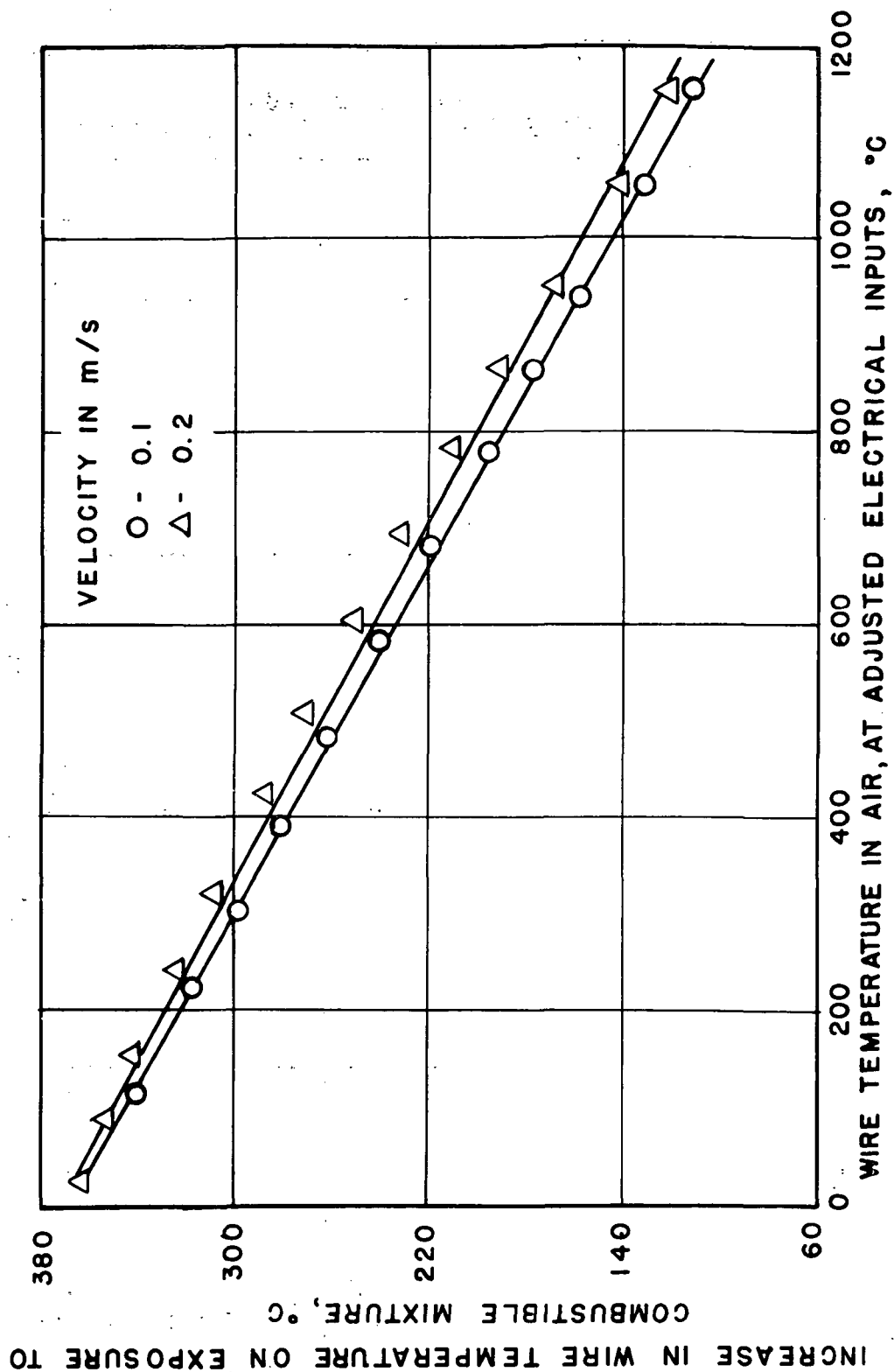
FIGURE 10.- CATALYTIC EFFECTS FOR 0.5-mm DIAMETER PLATINUM WIRE IN AIR+2.0% HYDROGEN MIXTURE FOR VARIOUS FLOW RATES OVER AN EXTENDED TEMPERATURE RANGE
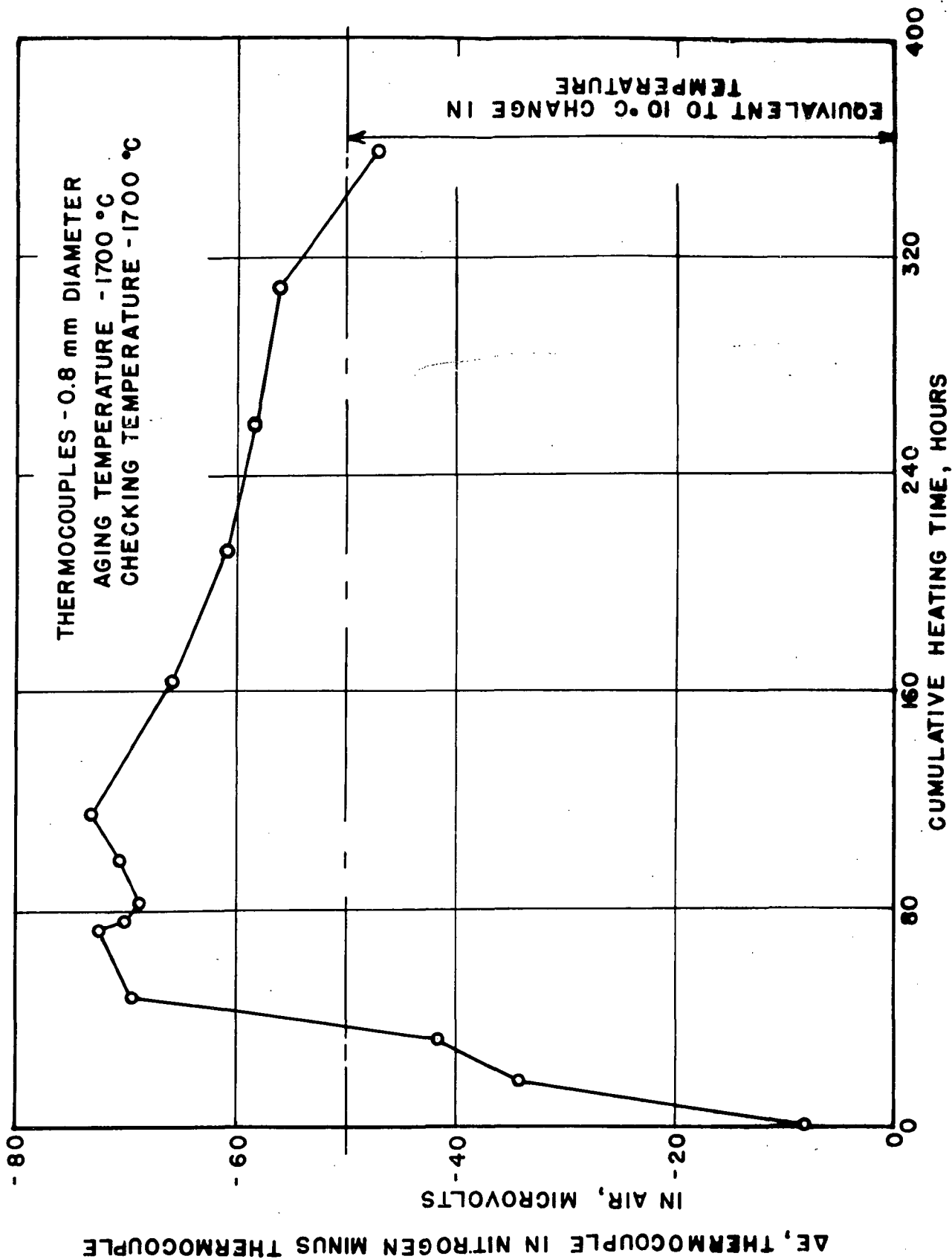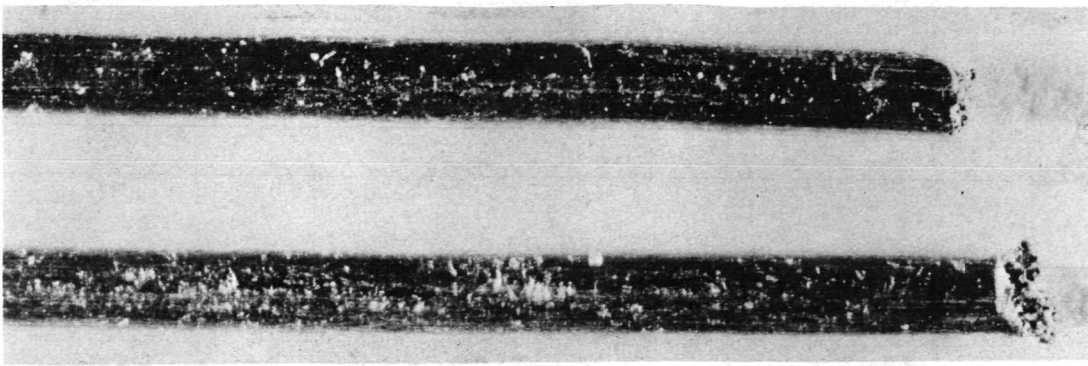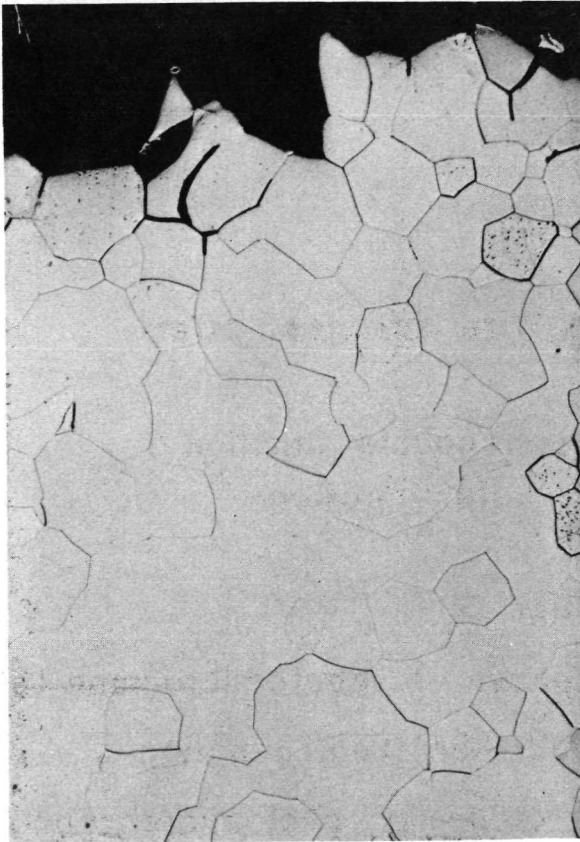
FIGURE 11.- SUMMARY OF CUMULATIVE CHANGES IN THERMAL OUTPUT RESULTING FROM LONG TERM HEATING OF A THERMOCOUPLE IN AN OXIDIZING MEDIUM

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12a.- Magnification - X 16

b. Magnification - X 100   c.
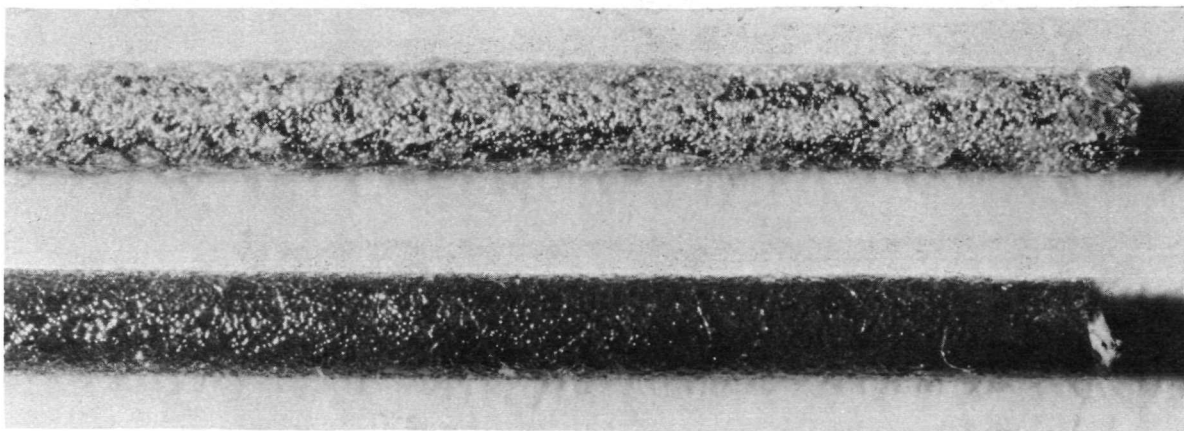
12a. Macrographs of wire samples (Unheated)
    Top- Iridium sample
    Bottom- Iridium 40 Rhodium sample

b. Longitudinal section - Iridium sample

c. Longitudinal section - Iridium 40 Rhodium sample

FIGURE 12.- WIRE STRUCTURE OF THERMOELEMENTS FOLLOWING
    AN ANNEALING AT 1800 °C, FOR FIVE MINUTES

13 a.- Magnification -X 16

13 a. Macrograph - Iridium 40 Rhodium Wire samples, after
    359 hours at 1700 °C, in air.

        Top - Region farther from thermocouple junction
   Bottom - Region near thermocouple junction

    b. Longitudinal section near junction

    c. Area showing grain boundary phase in photomicrograph (b)

    d. Longitudinal section, segment farther from junction

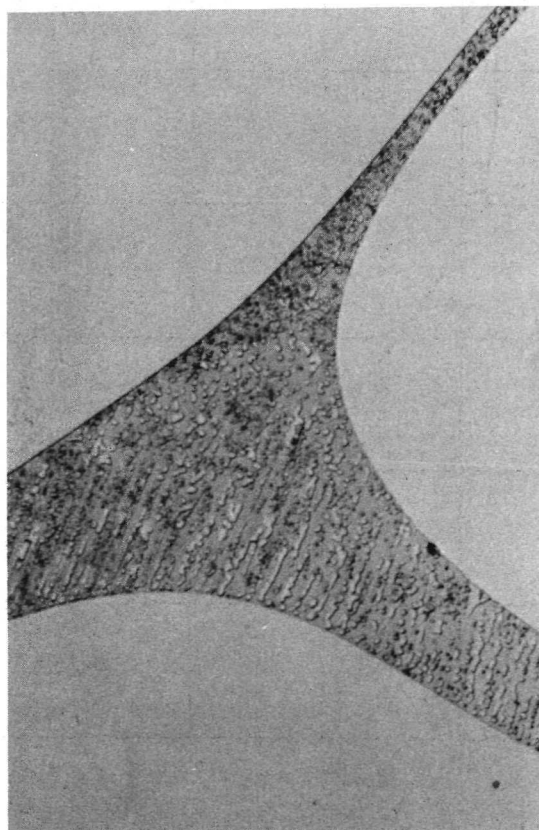   e. Area showing voids along the edge in photomicrograph (d)

FIGURE 13.- STRUCTURE OF IRIDIUM 40 RHODIUM WIRE
        EXPOSED TO HIGH TEMPERATURES, IN AIR
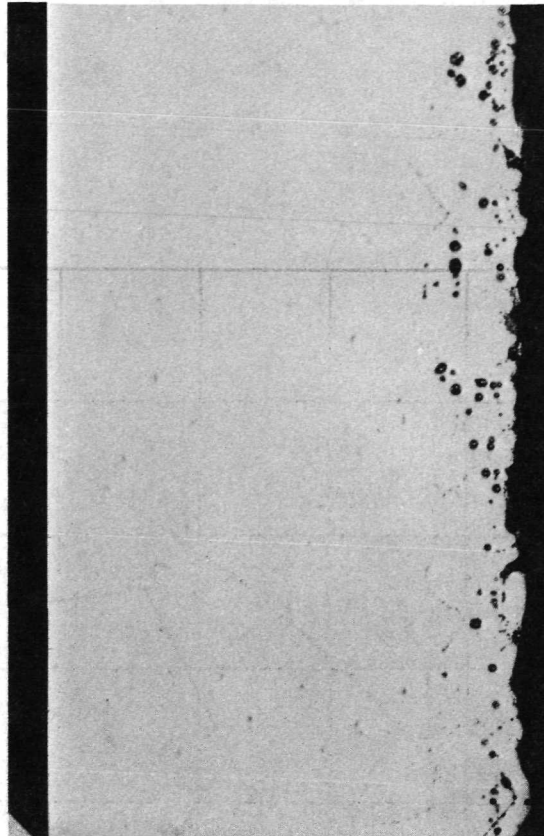
13 d.- Magnification – X 100

13 e.- Magnification- X 250
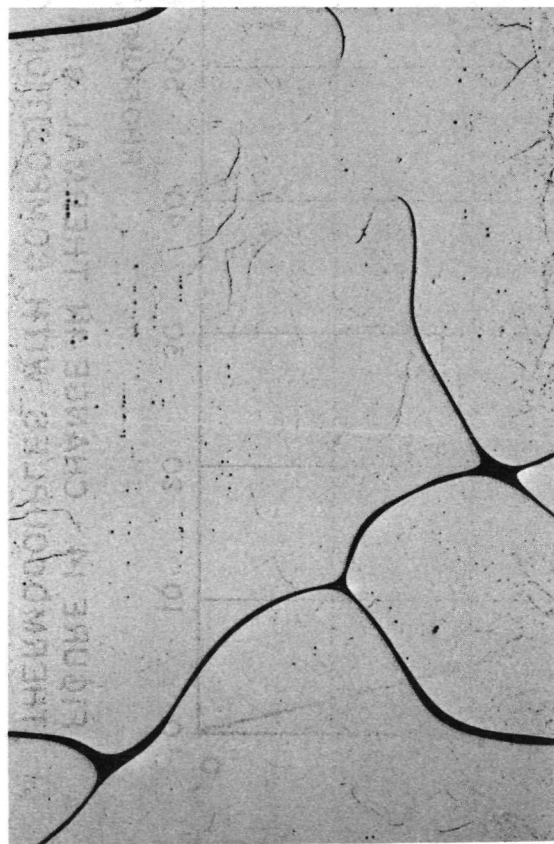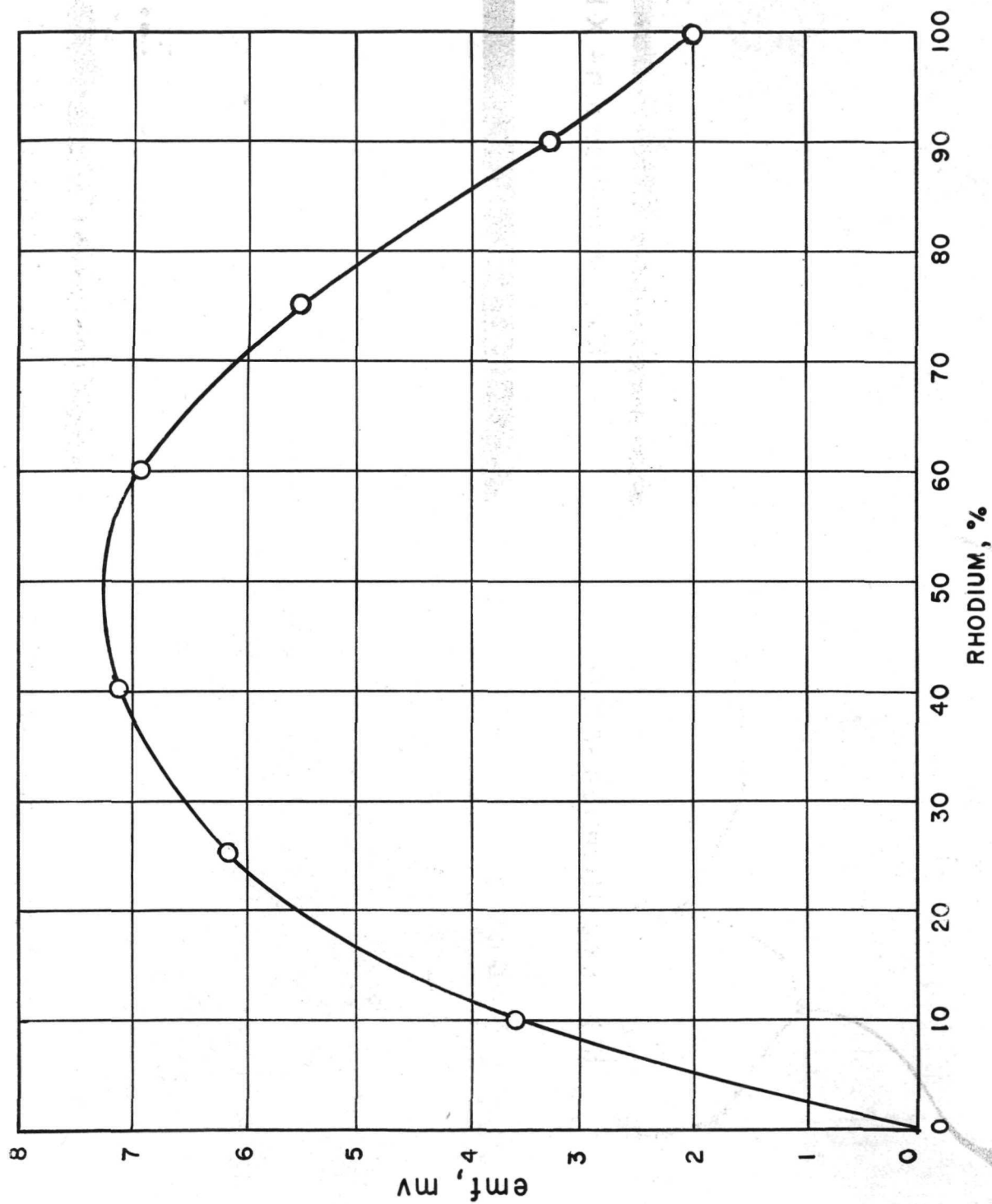

13 b.- Magnification -X100

13 c.- Magnification- X 1200

FIGURE 14.- CHANGE IN THERMAL emf OF IRIDIUM-RHODIUM TO IRIDIUM THERMOCOUPLES WITH COMPOSITION OF THE ALLOY, AT 1316 °C

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