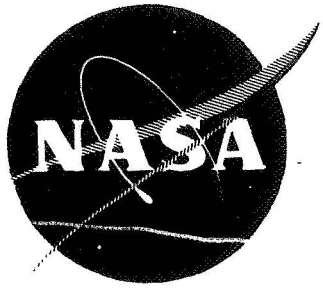
NASA CR 72861
NBS 4253439



DEVELOPMENT OF TEST FACILITIES
NOBLE METAL THERMOCOUPLE RESEARCH PROGRAM
1000 — 2000 C

by

P.Freeze, D.Thomas, S.Edelman, J.Stern

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

CONTRACT N A S A C - 46191 B

U.S. DEPARTMENT OF COMMERCE

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SUMMARY REPORT

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January 1971

CONTRACT NASA C-46191-B

Technical Management
NASA Lewis Research Center
Cleveland, Ohio
Chief, Instrument Research Branch
I. Warshawsky

U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards
Washington, D. C. 20234

FOREWORD

The research described herein, conducted at the National Bureau of Standards, Washington, D. C., is performed under NASA Contract C-46191-B, dated January 17, 1969, as NBS Project 4253439. The work is controlled by NASA Project Manager, I. Warshawsky, Chief of Instrument Research Branch, NASA Lewis Research Center and Technical Advisor, G.E. Glawe.

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ABSTRACT

This report describes test facilities developed and work performed to date on the Noble Metal Thermocouple Research program. The program involves three performance characteristics of noble metal thermocouples in oxidizing media, such as air and air containing various unburned gases.

Characteristic I, an investigation of the effects from catalysis on platinum wires in a low velocity facility, indicated significant effects from certain gas mixtures. The concentration of the unburned gases in a mixture, flow rate, and wire diameter contribute significantly to the size of the error. Preliminary results obtained in the high velocity test system show catalysis continues to be a source of error at increased velocities of 0.2×10^2 to 0.9×10^2 meters per second. Characteristic II is a thermoelectric stability study of the iridium-40% rhodium versus iridium thermocouple system at temperatures up to 2000 C in oxidizing media such as air. A test facility and associated equipment was constructed and tested. Characteristic III involves a study to determine the effects of large and small temperature gradients on the accuracy and stability of thermocouple measurements. A heat source to generate controlled temperature gradients was tested.

SUMMARY

The research program sponsored by NASA involves three performance characteristics of noble metal thermocouples in air and other oxidizing media containing a mixture of air and unburned combustibles, in the temperature range of 1000 to 1500 C. Characteristic I is concerned with the effect of catalysis on the temperature indication of noble metal temperature sensors. Measurements obtained from the Characteristic I study indicate that significant errors result in this temperature range when noble metal thermocouples are exposed to various unburned gas-air mixtures. Platinum thermocouple elements were exposed to gas mixtures of:

(a) air + 1% hydrogen

(b) air + 2% hydrogen

(c) air + 1% carbon monoxide + 0.01% hydrogen

(d) air + 2% carbon monoxide + 0.01% hydrogen

(e) air + 0.5% butane

(f) air + 2% butane

At a very low velocity of 2.2×10^{-3} meter per second, an air +2% butane mixture flowing over a 0.8 mm diameter platinum wire resulted in a temperature increase of approximately 100 C, at an initial wire temperature of 1000 C, when compared with the case for air flowing at the same velocity. This can be compared to an increase of 13 C in an air plus 2% hydrogen mixture and an increase of 7 C in an air plus 2% carbon monoxide mixture at the same gas velocity, wire diameter and initial wire temperature level. Data obtained in the velocity range of

2.2×10^{-3} to 8.8×10^{-3} meter per second show that the magnitude of the catalytic error for a particular wire sample is dependent on the concentration of the unburned gases, flow rate and wire diameter.

Preliminary measurements in a second test facility which operated in a higher velocity range showed that catalysis continues as a source of error over a velocity range of 0.3×10^2 to 0.9×10^2 meters per second. This facility has the capability of extending the velocity range to 1.8×10^2 meters per second.

Characteristic II is concerned with the thermoelectric stability of the iridium-40% rhodium versus iridium thermocouple system, for measuring temperatures up to 2000 C, in oxidizing media. A new test facility and associated equipment was constructed, installed and evaluated. This test facility is capable of subjecting two thermocouples to an atmosphere of nitrogen at 2000 C, and two other samples to an atmosphere of air simultaneously. Nitrogen is taken as the neutral reference environment. Behavior of a thermocouple in it is compared with the behavior of a test thermocouple in the oxidizing atmosphere, - air. The determination of any changes in the thermoelectric stability of the thermocouple can be accomplished without removing the operative portions of the thermocouples from the uniform hot temperature zone.

Characteristic III is an investigation of the effects of temperature gradient on the accuracy and stability of thermocouple measurements. A Nd:YAG laser was obtained as a heat source to raise the junction temperature of a water-cooled thermocouple probe to 2000 C. This

concentration of heat at the thermocouple junction will permit small and steep controlled gradients to be formed along the lead wires of the thermocouple.

1. INTRODUCTION

Along with the advances in the technologies of combustion and metallurgy have come increases in the temperature of gaseous products emanating from jet engines. The continuing effort to increase thrust and efficiencies of propulsion engines presages even higher temperatures of exhaust gas in the future. As a result of these higher temperatures a need has arisen for thermocouples capable of sustained operation in hot oxidizing gases. For systems operating in the temperature range of 1000 to 2000 C, it becomes necessary to make use of thermocouples made from noble metals, such as platinum, rhodium, iridium and their various alloys. These metals can be fabricated into thermocouples whose physical properties are generally known, but whose performance characteristics when subjected to high temperatures in oxidizing environments containing products of combustion are not known.

A research program sponsored by the National Aeronautics and Space Administration, Lewis Research Center, Cleveland, Ohio under NASA Contract C-46191-B, dated January 17, 1969, NBS Project 4253439, involves three areas in which the accuracy of noble metal thermocouples is affected.

Characteristic I is a study of the effect of catalysis on the temperature indication of noble metal thermoelements, particularly platinum-13% rhodium and platinum wires. There is a serious concern that these metals serve as catalysts in hot gaseous mixtures which are not in combustion equilibrium. There is good reason to believe that complete combustion is not attained for a considerable distance downstream from any combustion chamber. Hence, in the use of the platinum

metals as thermoelements, consideration must be given to the possibility that equilibrium will not exist at the point of use and further chemical reaction may take place on the element surface. Such catalytic action on the surface of an element of a small mass will be exothermic, and the heat liberated will tend to raise the temperature of a thermocouple to a value higher than its environment. Earlier investigators, Dahl and Fiock (ref. 1), Stanforth (ref. 2), Olsen (ref.3) and Stern, et al (ref. 4) observed this chemical effect.

The results of the Characteristic I studies conducted at NBS under this contract provide information on catalytic effects over the planned temperature range of 1000 to 1500 C, with various gas mixtures flowing at very low velocities. Preliminary results obtained with an improved facility and improved instrumentation give evidence that catalysis is present at higher velocities of 0.3×10^2 to 0.9×10^2 meters per second. The new facility can be evaluated over the same temperature range and with similar gas mixtures, as has been previously reported for low velocities.

Characteristic II is concerned with a study of a thermocouple system considered for use up to 2000 C in oxidizing media. Only thermocouples of iridium and alloys based on iridium are suitable in such conditions. Aleksakhin, et al (ref. 5) studied thermocouples made from iridium alloys and reported certain combinations were suitable for use up to 2100 C, in an oxidizing atmosphere. Blackburn and Caldwell (ref. 6) covered selected alloys from the standpoint of usefulness as thermocouple elements such as 40, 50 and 60 percent rhodium. Their study did not yield results on which a definite preference could be based or

indicate the thermoelectric stability of the various combinations. This investigation is concerned with the iridium-40% rhodium versus iridium system. Its thermoelectric stability at temperatures up to 2000 C, in an oxidizing medium is being investigated.

The furnace required for this study was built and evaluation tests begun. This furnace is capable of maintaining an oxidizing, reducing, neutral, or inert atmosphere inside any of four zirconia test cells for extended heating periods.

Characteristic III is the development of a method to determine the effects of temperature gradients on thermocouple indications. In the use of thermocouples, it is desirable to maintain the reference junction at a constant temperature, so that the Thermal Electromotive Force developed by a thermocouple of homogeneous elements immersed in a constant temperature sink or a medium with severe temperature gradients depends only on the junction temperature. However, in practical applications in both the laboratory and industrial usage, the handling and cold working of thermocouple wires results in the developing of inhomogeneous segments along the wires. The emf developed by an inhomogeneous thermocouple depends not only on the temperature of the junction, but on the temperature distribution along the elements. Thermocouples are to be exposed to controlled gradients in gas mixtures at temperatures up to 2000 C, and the effects on their performance analyzed and evaluated.

A test facility for this study was designed. A laser is to be used as a concentrated heat source that heats only a discrete portion of the thermocouple to 2000 C.

2. APPARATUS AND TEST PROCEDURE

I. The study to determine the catalytic effects received first priority. The studies were conducted in two facilities. The first, described in a report by Stern, et al (ref. 4), is a low velocity test facility. The velocities were in the range of 2×10^{-3} to 9×10^{-3} meter per second or quite small as compared to those encountered in aircraft propulsion research. The second facility was designed for higher velocities in the range of 0.1×10^{2} to 1.8×10^{2} meters per second. In both facilities, the effect of catalysis on thermocouples is evaluated by using single wires of the positive and negative elements. The elements are resistance heated to achieve exposure temperature. The thermoelements specified for the test are platinum-13% rhodium and platinum.

Figure 1 is a general schematic diagram of the low velocity test apparatus with the test element in position. The test element is formed into a hair-pin configuration as shown in Figure 2. Various wires of 0.3, 0.5, and 0.8 mm diameter were tested. Platinum wires serving as voltage leads were welded to the test element. The sample was electrically heated to various temperatures between 1000 and 1500 C by use of a current regulated dc power supply. Three parameters were measured after the temperature of the test element had reached stability. The three parameters were:

1. The direct current flowing through the sample.
2. The voltage drop (IR) across the 150 mm test length of the sample.
3. The brightness temperature of the test element as determined

with a calibrated optical pyrometer. The position on the wires on which the optical pyrometer was sighted is point A, Figure 2.

The above parameters were first measured at each temperature with only air flowing through the test chamber, then with the mixture of air plus a known amount of a combustible gas. The mixtures used were air-hydrogen, air-carbon monoxide, air-carbon monoxide with a trace of hydrogen and air-butane. Measurements were made at velocities of 2.2 x 10^{-3}, 4.4 x 10^{-3}, 6.6 x 10^{-3}, and 8.8 x 10^{-3} meter per second.

Preliminary studies were also conducted in a high velocity test section shown in Figure 3. The apparatus was designed to allow the test gases to enter a plenum chamber at low velocities and then increase to higher velocities in a constricted test section. The inside diameters of the test section and plenum chamber were 0.64 cm and 6.4 cm respectively. The test sample was mounted along the vertical axis of the test section and was held in this position by upper and lower water-cooled sample holders. The length of the test sample exposed to the gas flow was 8.3 cm. The velocity of the gases flowing in the test section was determined by measurement of total pressure, static pressure, and temperature of the fluid. The static pressure in the test section was determined through the static pressure tap in the center of the test section and the total pressure in the system was determined with a plenum chamber pitot tube. The temperature of the test sample was measured using an optical pyrometer with appropriate corrections for the spectral emissivity of the test sample and transmission losses of the viewing window. To eliminate undesirable bending of the sample by thermal expansion at high temperatures, electrical contact to the lower end of

the sample was provided through an indium-gallium eutectic. A small molybdenum weight attached to the test sample was suspended in the eutectic which is liquid at room temperature. Electrical power was brought to the eutectic by a molybdenum-clad copper wire extending to the outside of the apparatus.

During an actual test run, pressurized dry air was released into the calming chamber, Figure 4, where it would strike a diffusing plate and flow through a series of diffusing screens to reduce flow turbulence. With the dry air flowing over the test sample at a predetermined velocity, the electrical power to the test sample was increased until it reached a selected test temperature between 1000 and 1500 C. The sample temperature was then accurately determined using the optical pyrometer. This procedure was repeated with a test gas mixture (unburned gas) flowing over the sample at the same velocity as the dry air and with the same electrical power to the test sample. The difference between the two measured temperatures was attributed to the sample temperature increase due to catalysis on the surface of the sample. To verify that this increase in sample temperature resulted from catalysis, a sample was prepared from a non-catalytic material, - gold. Although the wire failed at the higher velocities, the measurements obtained indicated the gold sample to be free of catalytic effects.

II. A high temperature furnace shown in Figure 5 was designed and built for the Characteristic II study to determine the thermoelectric stability of the iridium-40% rhodium versus iridium thermocouple system in oxidizing media, at temperatures up to 2000 C. In the construction of the furnace considerable emphasis was placed on the following

features:

1. Four zirconia test cells were located inside a tantalum heating element for comparing two thermocouples in a nitrogen atmosphere (neutral reference environment) with two thermocouples in air.
2. During operation of the furnace at temperatures between 1500 and 2000 C, a hot zone uniform in temperature to within ±1% is maintained inside the heating element over approximately 460 mm of its length.
3. The thermocouple pairs are first calibrated by immersing them in a portion of an isothermal zone to a position where their junctions are located at the center of the upper sighting window. Then, for the prolonged heating phase of the test, the thermocouple pairs are fully immersed to a position at the center of the lower sighting window. For recalibration the pairs are again withdrawn to their original position. In this way the segment of thermocouple that had been exposed to the temperature gradient during aging will not contribute to the thermoelectric output, so that it is assured that observed changes in thermal output are due to aging at the test temperature.
4. The thermocouples are freely suspended in the hot zone of the test cells.
5. At temperatures above 1600 C, the thermocouple is so suspended as to be free from contact with any electrically conductive materials.

The heater is a split tantalum tube 10.2 cm in diameter and 80 cm in length maintained in a helium atmosphere. Located within this test section are four zirconia tubes and a tantalum well containing a reference thermocouple. Each zirconia tube is approximately 2.5 cm in diameter and 80 cm long. The tantalum well is 80 cm long closed at one end and has an outside diameter of 1.3 cm and a wall thickness of 1.5 mm.

The temperature of the hot zone will be accurately determined using a calibrated optical pyrometer sighted on the blackbody located at the end of the reference thermocouple well. A second optical pyrometer will be sighted through the upper window with the tantalum well as the target. Corrections for emissivity and window transmission loss will be made to provide the true temperature of the hot zone.

The tantalum heater is surrounded by tantalum heat shields. These consist of three separate components (top, side, and bottom). Each component is an assembly of six layers of tantalum sheet. They are enclosed in a dual-wall stainless steel water cooled jacket.

The water cooling for the furnace is supplied from a manifold system which is connected to two independent water supplies. The system is provided with appropriate instrumentation so that either supply can be operated manually or in an automatic mode.

Figure 6 is a schematic arrangement of the furnace and its associated equipment. A controller is capable of maintaining the furnace at any temperature within the range of 200 to 2300 C. The controller is a silicon controller rectifier type with an input of 480 volts, 3 phase and 60 hertz. The output is supplied to a combination Scott connected transformer and step-down transformer.

The controller includes an adjustable current limiting device which can be set for a pre-determined maximum current supplied to the furnace. If a maximum current is reached during operation in either the manual or automatic furnace control mode, the limiting device does not cut the power off but instead maintains this maximum current until the furnace controls are reset.

The control instrumentation for the furnace includes a recorder and a C.A.T. control unit with a combination manual and automatic mode of operation. On the automatic mode, the controller and recorder system utilizes the thermal output of the control thermocouple in the tantalum well assembly to maintain a predetermined furnace temperature within ±1/2%.

A vacuum system consisting of a mechanical pump in series with a diffusion pump and a liquid nitrogen trap are required for purging the system. A helium atmosphere is then maintained over the tantalum components in the furnace hot zone during operation of the facility.

Provisions are made to circulate oxidizing, inert, or neutral gases through the zirconia test cells containing the test thermocouples. During the calibration periods the thermal emf's of the test thermocouples can be switched to the desired read-out equipment.

III. Characteristic III involves a study of the effects of temperagure gradient on the accuracy and stability of thermocouple measurements up to 2000 C. This investigation is third in priority. Experiments indicated that gradients could be formed without affecting the flow of gas over the thermocouple if a laser was used to heat the junction of the thermocouple. A Nd:YAG laser was acquired and tested.

The initial test of the laser was to determine if it could produce 60 watts of beam power continuously for a period of one and one half hours. A broad-band cw laser power meter was used to measure directly the radiated power.

A test was then conducted to determine that divergence of the laser beam was within the angle of 2.5 degrees, as required, in the specification.

3. DISCUSSION OF RESULTS

I. The results of the experiments on catalysis by noble metal thermocouple materials were obtained in the low velocity test facility. The high velocity system was designed and constructed during this contract period. The facility was nearly completed except for the required accurate pressure measuring instrumentation. However, preliminary measurements were made with less sophisticated instrumentation.

The work consisted of studying the effects of catalysis on platinum thermocouple wires as a function of the following parameters:

1. Test gas composition
2. Test gas concentration
3. Flow rate and temperature, separately or synergistically.
4. Wire size (diameter).

The measurements show a temperature increase due to catalysis over the entire temperature range of 1000 to 1500 C, for the three gas mixtures studied. Figure 7 is a plot showing catalytic error for 0.8 mm diameter platinum wire samples in gas mixtures of air + 2% butane, air + 2% hydrogen, and air + 2% carbon monoxide at a very low velocity of 2.2 x 10^{-3} meter per second, all at a pressure level of approximately one

atmosphere. The increase in wire temperature as a result of catalysis was obtained for the three mixtures, but the increase from the air + 2% butane mixture was considerably larger than the increases in either air + 2% hydrogen or air + 2% carbon monoxide. At a temperature of 1025 C, an increase in temperature of approximately 95 C was obtained with a mixture of air + 2% butane. This can compare to an increase of 12 C in air + 2% hydrogen and an increase of 7 C in air + 2% carbon monoxide. A similar comparison can be made at 1500 C. The increases in wire temperature are 36 C for air + 2% butane, 2 C for air + 2% carbon monoxide and 1 C for air + 2% hydrogen.

Figure 8 presents the results of additional measurements on an 0.8 mm diameter platinum sample in a gas mixture of air + 2% butane and in a mixture of air + 0.5% butane, at a gas velocity of 4.4×10^{-3} meter per second. The concentration of a combustible in a given mixture affects the size of the error due to catalysis. At 1200 C the error obtained with an air + 0.5% mixture of butane is 34 C compared with 134 C for air + 2% butane. This indicates an approximately linear relationship between the concentration of combustibles of a given composition and the resulting catalysis error.

It is to be noted the error due to catalysis decreases as the temperature of the wire increases.

The effect of flow rate on catalysis error is presented in Figure 9. The temperature rise caused by catalysis is nearly proportional to the flow rate at the velocities covered in the low velocity facility. The greater flow of combustible gases over the platinum element produces more combustion, transferring more heat to the wire while thermal

loss by radiation is increased only slightly by the higher temperature of the wire. The magnitude of the effect at lower temperature is what might be expected from a heat balance between the rate of combustion and the rate of heat transfer.

Figure 10 shows the effects of catalysis as a function of wire diameter. For the three sizes of platinum wires used in the test, it was found that the catalytic temperature rise becomes greater with a decrease in wire diameters. These results are qualitative , as expected, however the deviation from a hyperbolic relation remains to be explained.

Figure 11 presents some preliminary results obtained in the new high velocity test system. Although the facility can reach velocities of 1.8×10^2 meters per second or greater, the figure presents data obtained to date showing temperature increases due to catalysis as a function of various velocities of 0.3×10^2, 0.6×10^2 and 0.9×10^2 meters per second. Included in the figure for comparison are the results obtained in the low velocity test apparatus on 0.5 mm diameter platinum samples in a gas mixture of air + 2% carbon monoxide + .01% hydrogen.

The preliminary results obtained in the high velocity apparatus indicate that the effects of catalysis continue to increase with increasing velocities, up to 0.9×10^2 meters per second.

It should be pointed out that during the normal operation of a modern jet engine, the concentrations of carbon monoxide, hydrogen, and hydrocarbons are considerably smaller than those used in the gas mixtures in this study. Thus the catalytic heating of normally operated engines would be proportionally less. The concentrations selected were

such that the catalysis contribution to the sample temperature could be determined over the entire range of temperatures and velocities selected for the investigation.

II. The high temperature furnace for use in determining the thermoelectric stability study of the iridium-40% rhodium versus iridium thermocouple system in oxidizing media was placed in operation and various tests begun. A series of tests was conducted to determine the temperature gradient along approximately 50 cm of the hot zone with one zirconia test cell in place. The observed maximum temperature difference along this entire hot zone at 1600 C was 11.6 C, and at 2000 C was about 25 to 30 C. This difference was within the desired ± 1% at 1600 C, but exceeded the tolerance desired at the upper limit of 2000 C.

Difficulties were encountered in the use of calcia stabilized zirconia tubes as test cells for aging the thermocouples. The rate at which these tubes are heated and cooled is important, especially at their critical temperature range of 700 to 800 C. Often during the cooling phase of a heating cycle, tube failure occurs if the rate of cooling of 150 C per hour in the critical temperature range is exceeded. New thin-wall magnesia stabilized zirconia tubes have been obtained and appear to be considerably more resistant to thermal shock.

III. The effort on the Characteristic III study was directed toward developing the necessary test facility. The laser heat source was acquired and tested. It produced a constant power output of 61 watts for a period of 1 1/2 hours. This output was greater than the 43 watts required to heat a thermocouple of iridium-40% rhodium versus iridium to the desired temperature of 2000 C. The beam divergence was found

satisfactory.

4. CONCLUSIONS

The results of the studies indicate temperature sensing elements made of platinum materials are subject to errors due to catalysis. The figures present data indicating that the magnitude of catalysis error is dependent upon the type of combustible in the air mixture, the concentration of the combustible in the mixture, the flow rate of the gas medium, and the diameter of the platinum wire.

The results of the preliminary studies in the high velocity facility show that catalysis continues to be a source of error in the velocity range of 0.3×10^2 to 0.9×10^2 meters per second. It will be necessary to go to higher flow rates to ascertain if there is a velocity at which this heating effect reaches a limit or no longer exists. (The present apparatus is expected to permit extension of this study to velocities of 1.8×10^2 meters per second.)

The evaluation of the furnace for the thermoelectric stability study of the iridium-40% rhodium versus iridium system indicates the furnace is suitable for use at temperatures up to 2000 C, for extended periods of heating. The temperature gradient in the furnace hot zone is satisfactory for the planned method of aging of the thermocouples and checking for any changes incurred from their exposure to an oxidizing atmosphere.

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LIST OF FIGURES

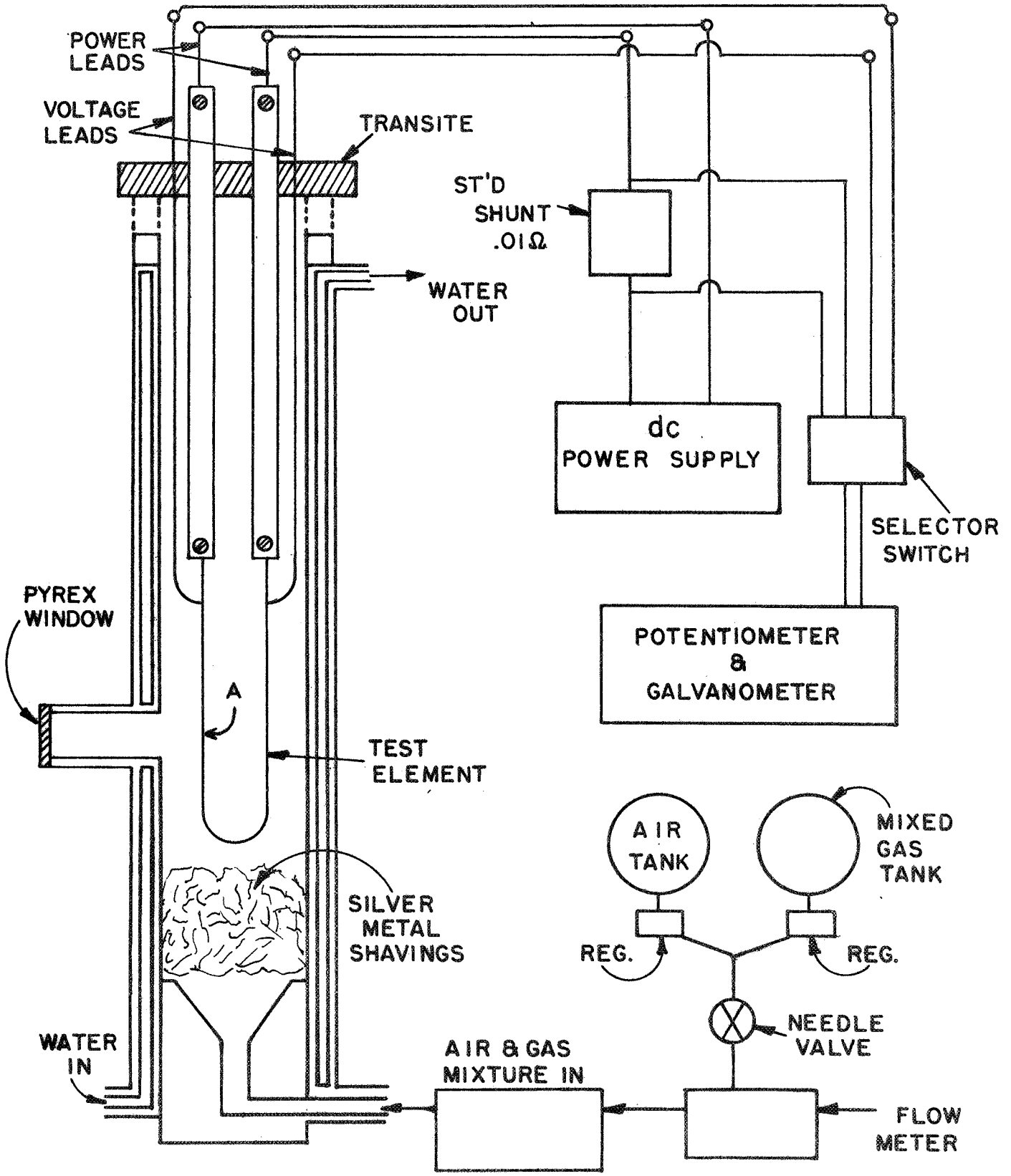

FIGURE 1.– SCHEMATIC DIAGRAM OF LOW VELOCITY FACILITY WITH TEST ELEMENT IN POSITION

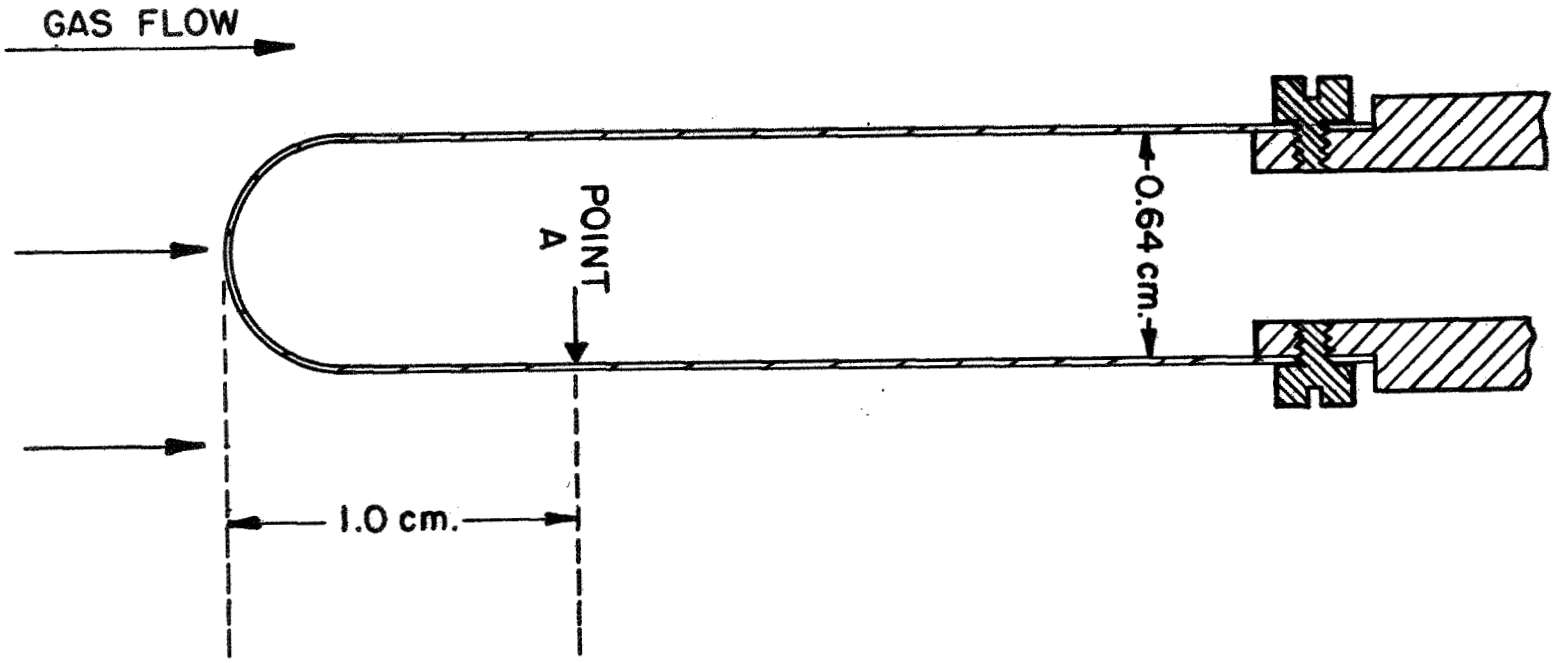

FIGURE 2.- SAMPLE TEST ELEMENT

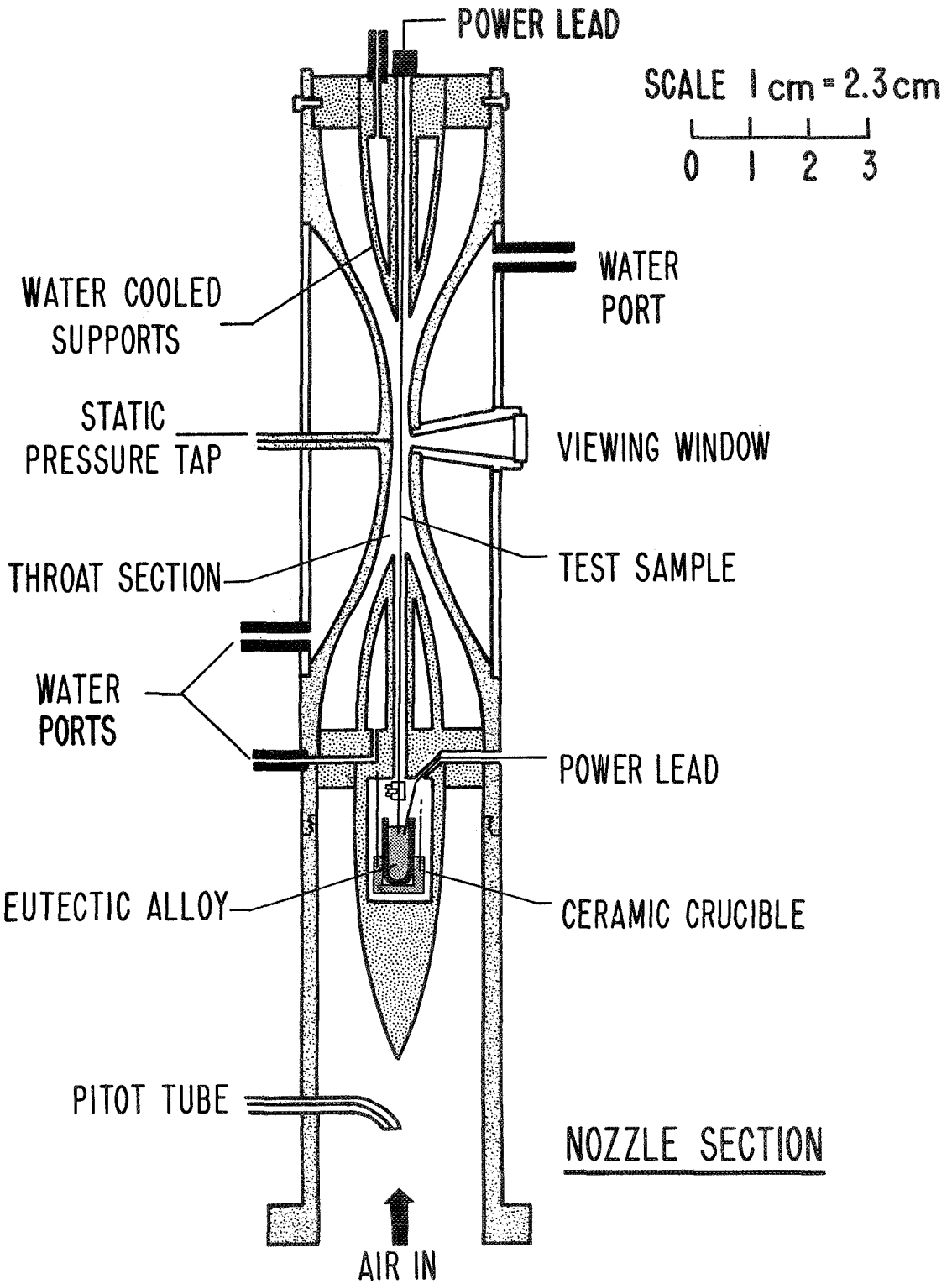

FIGURE 3.- TEST SECTION OF HIGH VELOCITY FACILITY

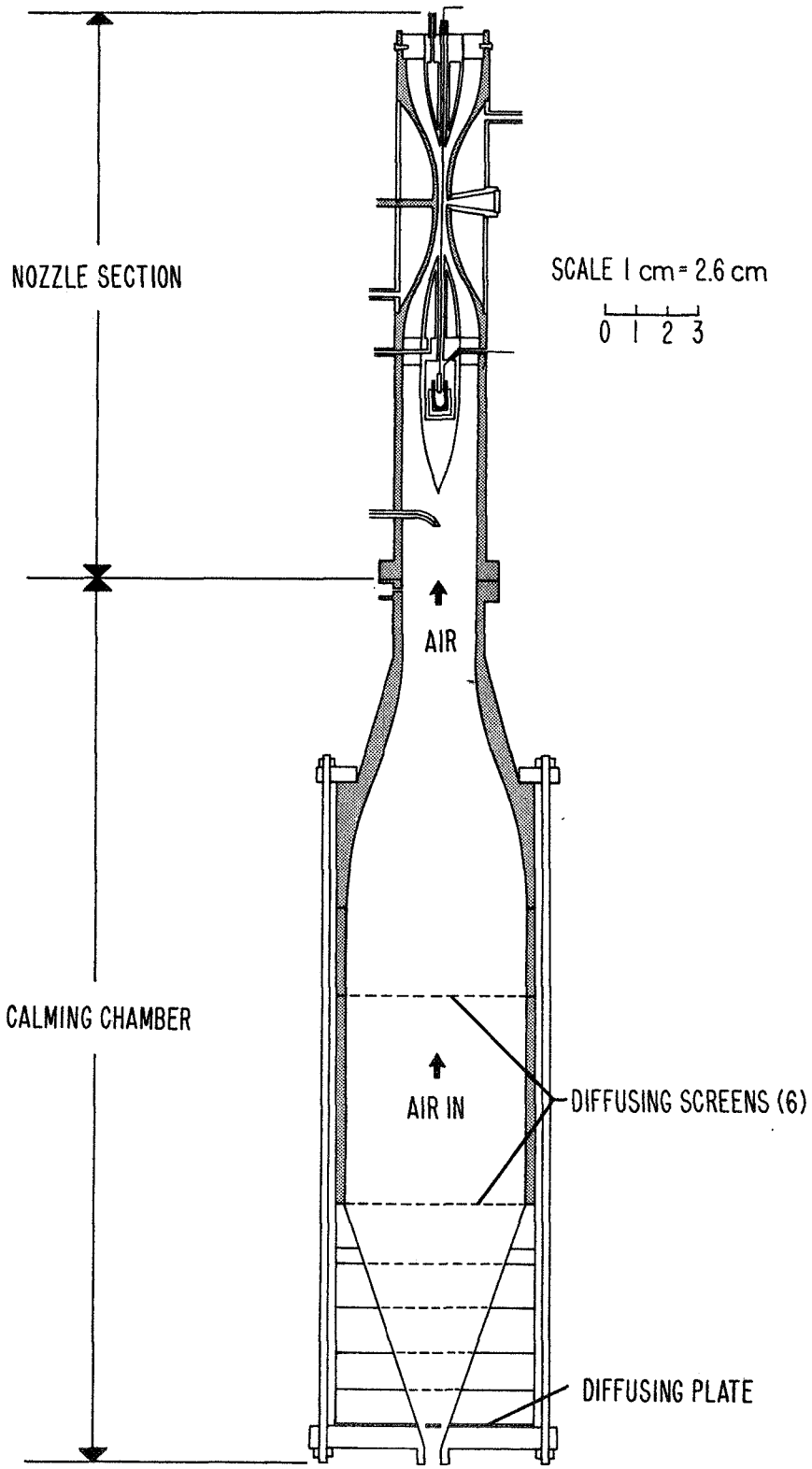

FIGURE 4 – HIGH VELOCITY CATALYSIS TEST FACILITY

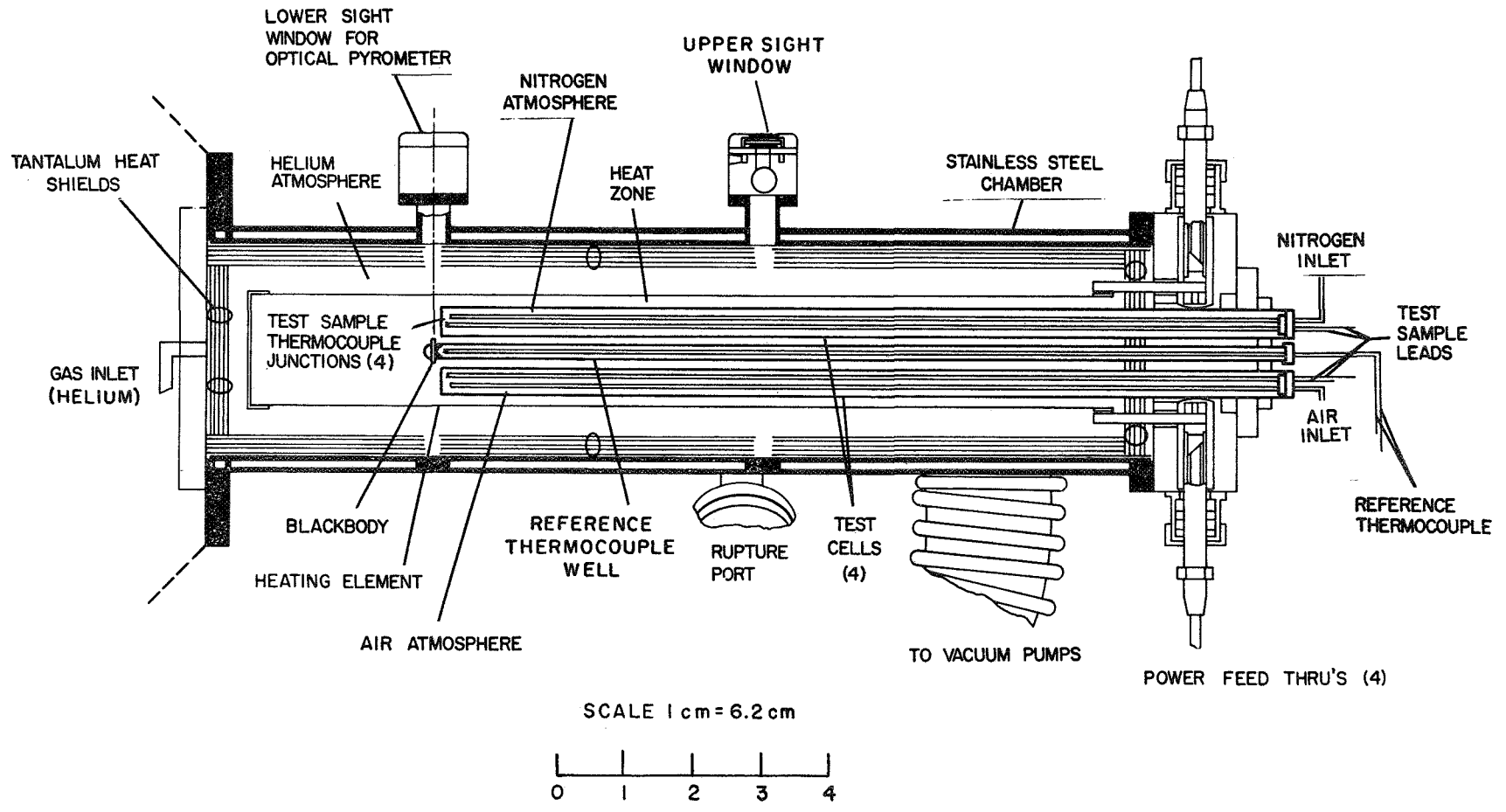

FIGURE 5 - THERMOCOUPLE FACILITY FOR STABILITY STUDIES

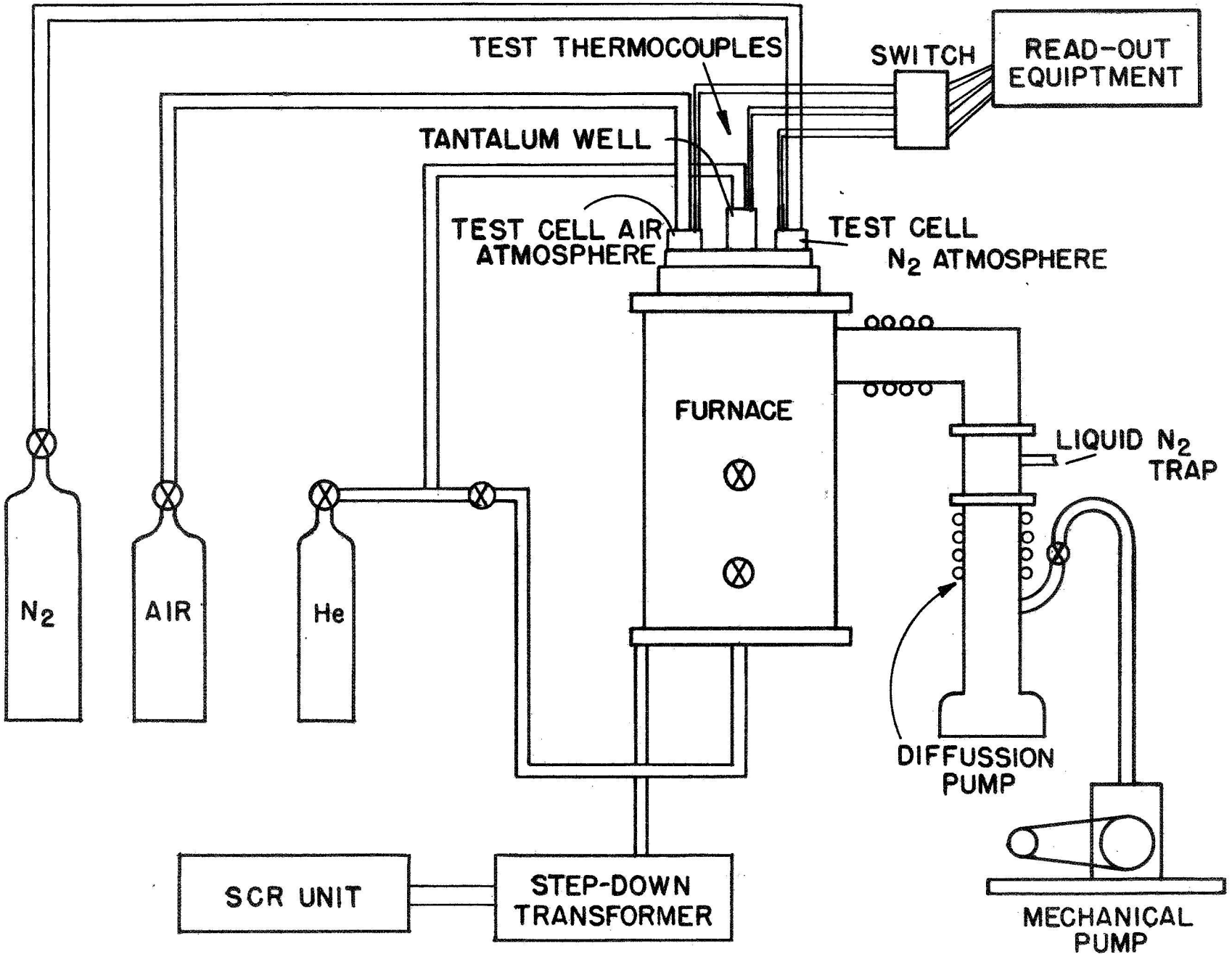

FIGURE 6.–SCHEMATIC OF FURNACE AND ASSOCIATED EQUIPMENT

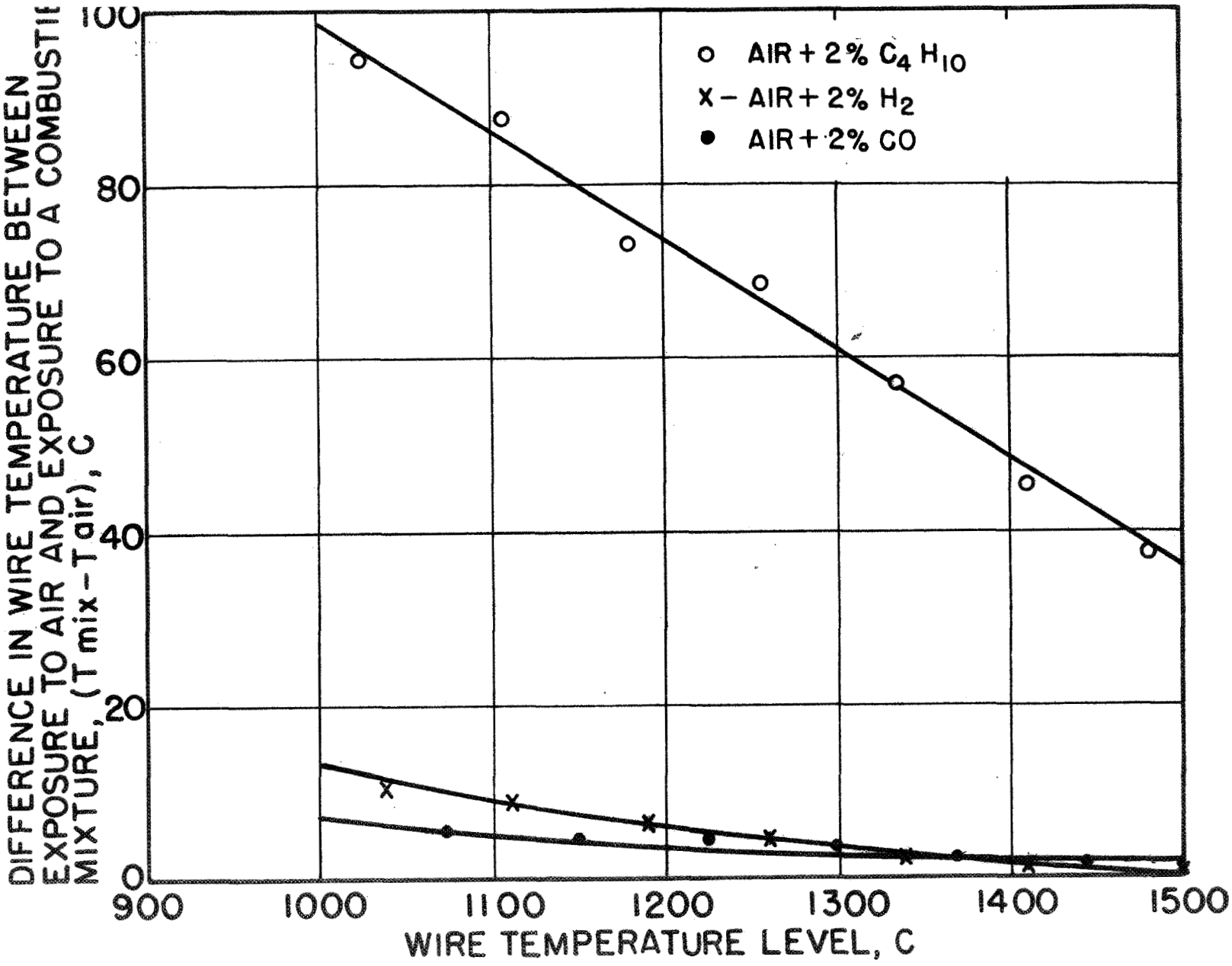

FIGURE 7.- CATALYTIC ERROR FOR 0.8 mm DIAMETER PLATINUM WIRES FOR THREE COMBUSTIBLE MIXTURES FLOWING AT 2.2×10^{-3} METER PER SECOND

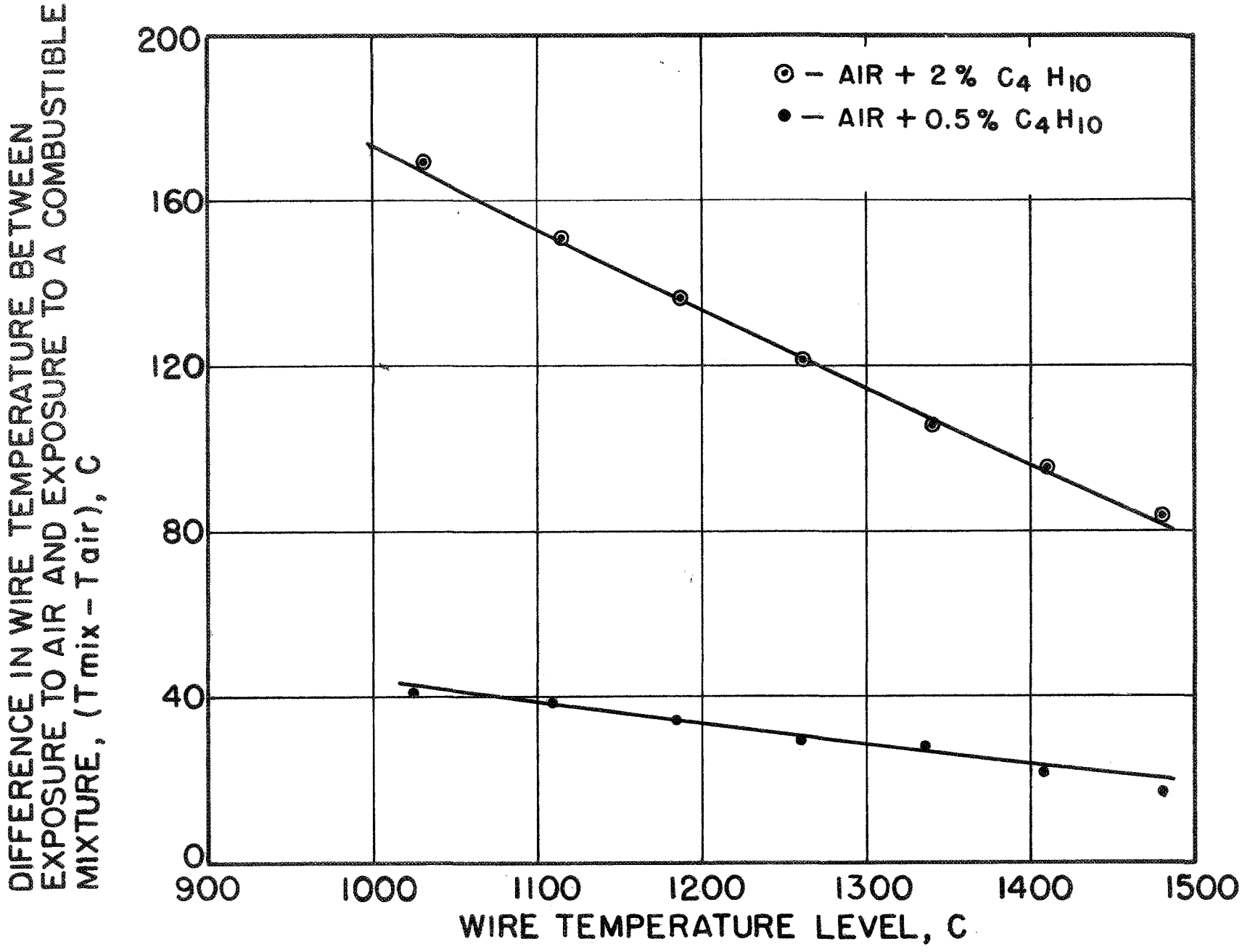

FIGURE 8.– CATALYTIC ERROR FOR 0.8mm DIAMETER PLATINUM WIRES ON BUTANE-AIR MIXTURES FLOWING AT 4.4×10^{-3} METER PER SECOND

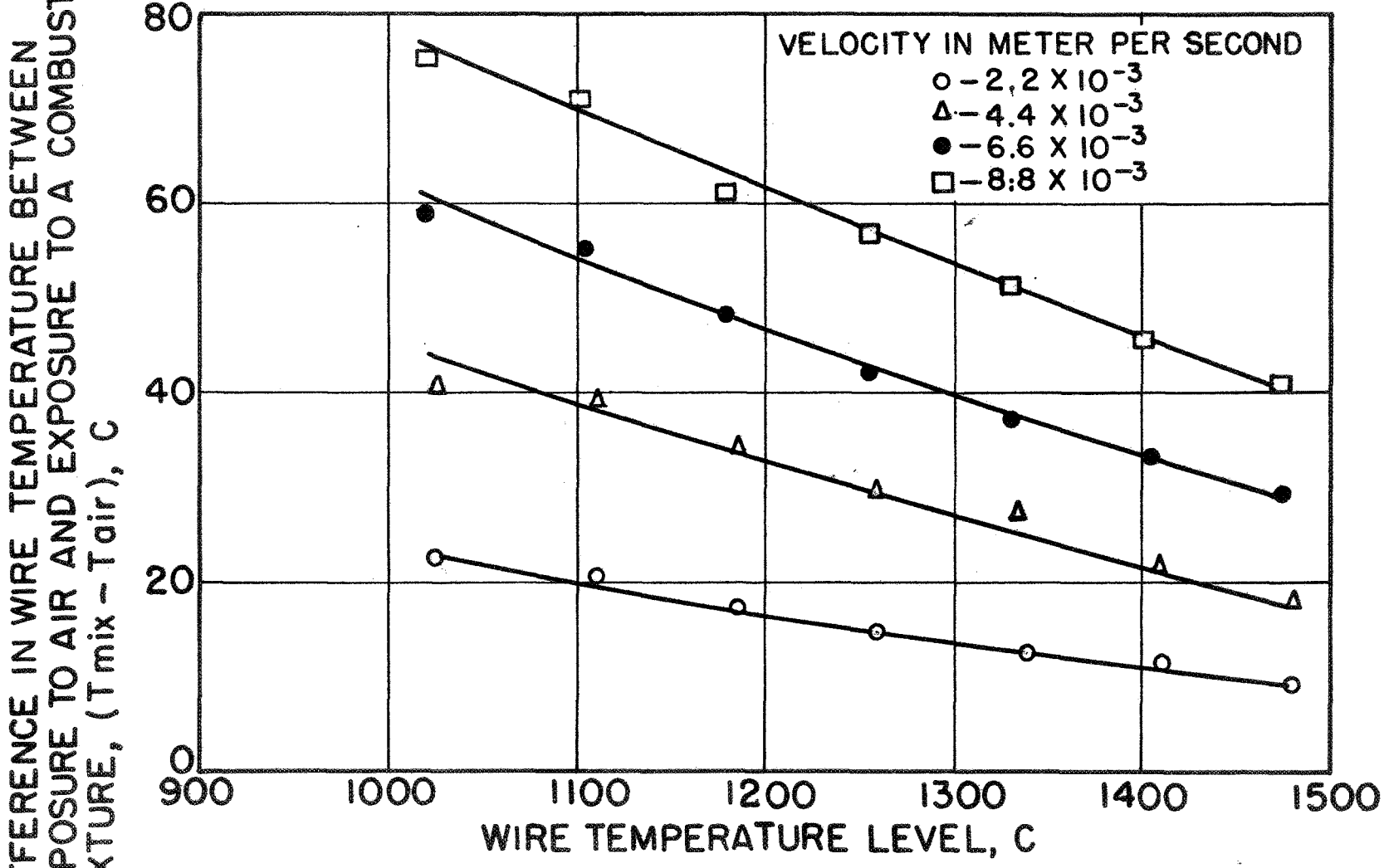

FIGURE 9. - VARIATION IN CATALYTIC ERROR FOR 0.8 mm DIAMETER PLATINUM WIRE EXPOSED TO (AIR + 0.5 BUTANE) MIXTURE FOR VARIOUS FLOW VELOCITES.

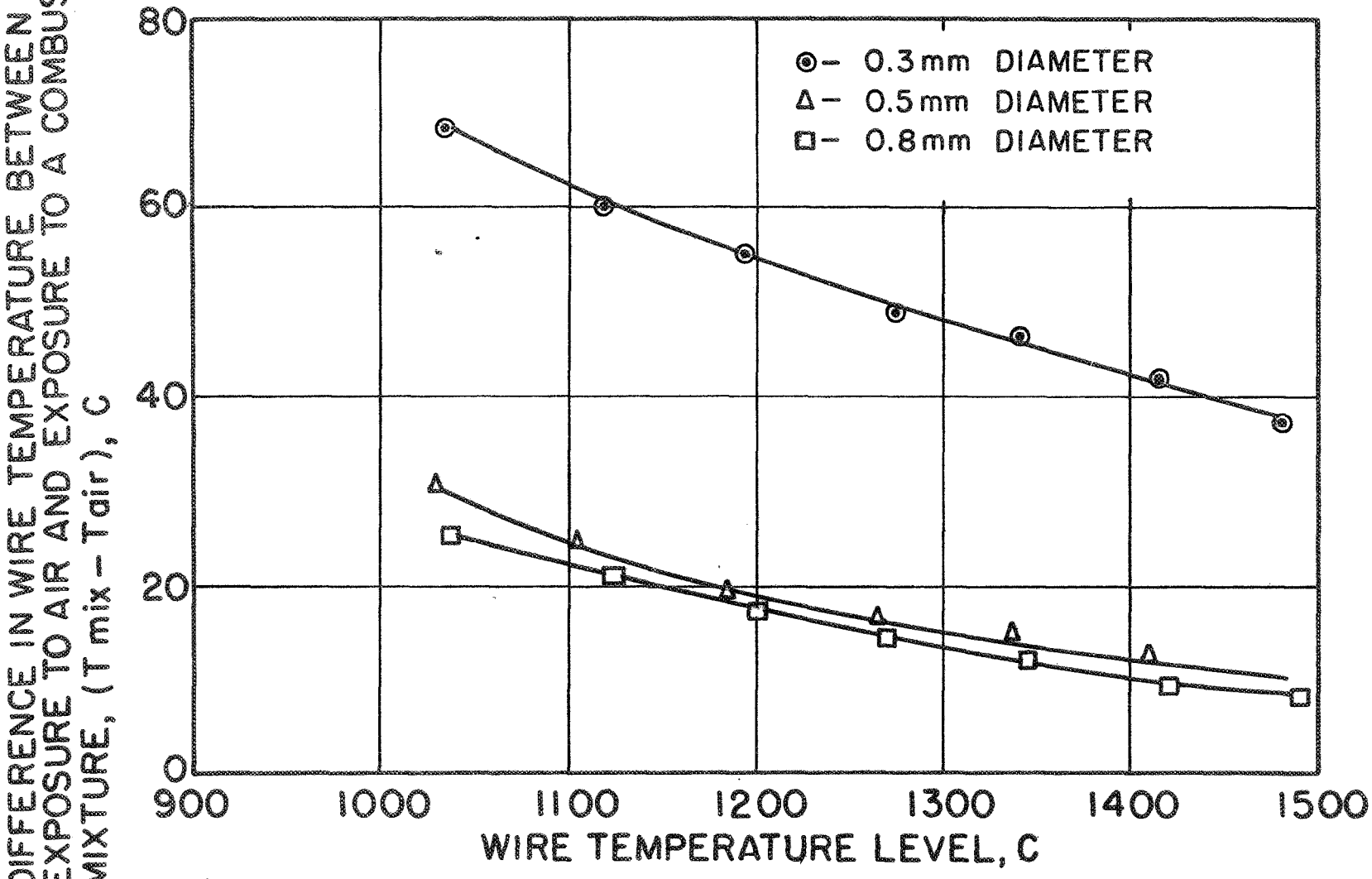

FIGURE 10.– CATALYTIC ERROR FOR VARIOUS SIZES OF PLATINUM WIRE AT A VELOCITY OF 6.6×10^{-3} METER PER SECOND IN AN (AIR + 2% CO + 0.01% H_2) MIXTURE.

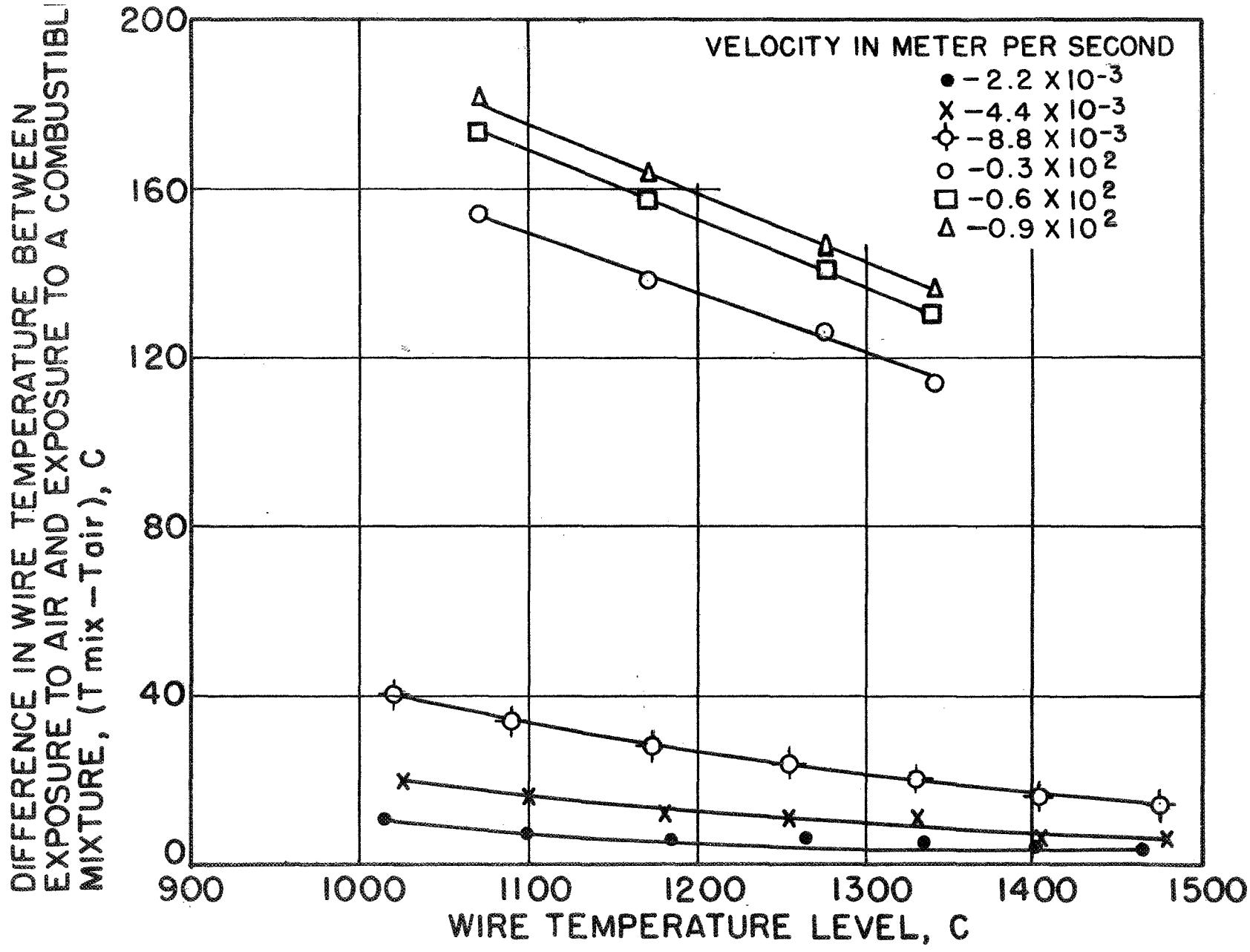

FIGURE 11.– VARIATION IN CATALYTIC ERROR FOR 0.5 mm DIAMETER PLATINUM WIRE EXPOSED TO (AIR+2% CO+0.01% H_2) MIXTURE FOR VARIOUS FLOW VELOCITIES