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FIFTH QUARTERLY REPORT

PRESSURE MEASURING SYSTEMS FOR CLOSED CYCLE LIQUID METAL FACILITIES

Anthony J. Cassano

PREPARED FOR

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

CONTRACT NAS 3-4170

June 28, 1965

Consolidated Controls Corporation
Bethel, Connecticut

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FOREWORD

The major contributors to this development program are Mr. R. Engdahl, Project Manager, Mr. Anthony Cassano, Mr. David Mends and Mr. Philip Tubman.

ABSTRACT

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Continuing development of a pressure transducer system for liquid metal applications is described. During the report period, emphasis was placed upon fabrication of an experimental transducer incorporating a thermionic diode sensor. Problems encountered in fabricating the transducer are outlined along with the anticipated solutions. A potassium compatibility test facility has been constructed and checked out. A complete set of single convolution pressure capsules and electrical terminals has been charged with liquid potassium and is undergoing the initial stages of the compatibility test procedure. Preliminary analyses are presented for different configurations of differential pressure transducers.

Author

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1.0 Introduction

The objective of this program is to develop pressure transducers which can be used in advanced closed cycle power systems using liquid metals such as mercury, sodium, potassium and other alkali metals as working and heat transfer media at elevated temperatures. Accurate pressure measurements in the high temperature liquid, vapor, and two phase streams are required for research, design and control purposes. In addition, space flight requires light weight systems capable of enduring long periods of unattended operation.

Liquid metal pressure measurements at elevated temperatures pose many design problems demanding the best from available materials. To establish a firm design base for the transducer equipment, four diaphragm materials and two transducer systems have been chosen for evaluation. The selected transducer system using a thermionic diode sensor will be developed for use as either ground or flight hardware for measuring absolute and differential pressures. The absolute pressure instrument will be developed for a full scale range of 80 psia and the differential instrument for ±5 psid.

2.0 Summary

During the report period, emphasis was placed on the fabrication of an experimental transducer incorporating a thermionic diode sensor. The main problem areas involve the encapsulated heater assembly and the technology required to obtain reliable metal-ceramic seals. Initial difficulties encountered with the encapsulated heater assembly have been alleviated by the introduction of a flat alumina coil form on which the heater is wound, and the use of alumina insulators in the casting cavity to avoid shorting of the heater to the emitter housing. Present plans also call for the introduction of a rhenium heater coupled with rhenium heater power leads to improve high-temperature reliability.

The pressure-temperature life test on a sample metal-ceramic seal planned for use in the transducer design was concluded after 2400 hours at 1800°F, the last 1000 hours of which were at 1000 psia pressurization. The sample employed Lucalox/Nb-1Zr seals fabricated by use of a tungsten/yttria metallizing

and a nickel/niobium/titanium braze material. This technique is being used in the fabrication of the experimental transducer discussed above. Efforts are being made to obtain a meaningful metallographic analysis of the test sample.

Construction and initial check out of the potassium compatibility test chamber was completed. A complete set of single convolution pressure capsules and electrical terminals has been charged with liquid potassium, installed in the compatibility test chamber and is undergoing the initial stages of the compatibility test procedure. It is planned to expose the test capsules to liquid potassium at 1800°F (corresponding to a potassium vapor pressure of about 80 psia) for a series of five 100 hour test cycles. This will result in a total of 500 test hours, at the conclusion of which the test capsules will be examined for possible potassium attack.

Preliminary analyses were performed on possible configurations for the differential pressure transducers. Results indicate the feasibility of obtaining 0.001 inch motion of the active collector of a thermionic diode sensor. Work has started on the design and assembly of a mock-up configuration to verify the results of the analysis.

3.0 Thermionic Diode Pressure Transducer

A preliminary design for a complete pressure transducer was presented in the Fourth Quarterly Report (Reference 1). The design, incorporating a double convolution pressure capsule and a thermionic diode sensor, is shown in Figure 1. Work on the first experimental device has concentrated on the fabrication of the encapsulated heater assembly, the preparation of its ceramic base and the development of the metal-ceramic technology necessary to obtain the required seal integrity. For the first device, the heater assembly will be mounted on its ceramic base, the ceramic base will be brazed into a Nb-1Zr support plate and the entire assembly will be installed in a micrometer head fixture (Figure 9, Reference 2; Figure 15, Reference 3) which allows controlled movement of a simulated active collector

Initial efforts to fabricate the encapsulated heater assembly utilized a bare tungsten wire of 0.003 inch diameter spirally wound on a 0.004 inch diameter mandrel. The resultant heater had an outer diameter of 0.010 inch and a length of 0.875 inch. The heater was threaded through alumina insulating tubes and cast into the molybdenum emitter housing with an alumina/beryllia mixture. The heater power leads were 0.010 inch diameter molybdenum wire connected to the heater by plasma-spraying the joint with tungsten and embedding the joint in the casting. Three difficulties were noticed with this technique.

1. The threading of the heater through the alumina tubes to form a module suitable for insertion into the emitter housing cavity proved to be a delicate and time-consuming operation.

2. Four or five separate casting operations were required to completely fill the voids left after the heater had been inserted into the cavity. In addition to the space remaining around the heater, the casting material must also fill the void space inside the alumina

insulating tubes. After each cast (3550°F), a certain amount of motion was noticed in the heater wire, apparently due to softening of the alumina tubes at the casting temperature. The wire motion resulted in shorting of the heater to the emitter housing. In two instances, large portions of the heater were shorted in this manner, resulting in the loss of the entire assembly.

3. Mounting the encapsulated heater assembly on its ceramic base requires that sharp bends be made in the molybdenum heater power leads so that the wires may be inserted in the proper holes in the ceramic base. However, after the casting operations, the molybdenum wires become brittle and thus subject to breakage during the bending operation. In this event, a 0.010 inch diameter tantalum extension lead was welded to the molybdenum stub and brought through the holes in the ceramic base.

The technique now under development for building the heater is designed to alleviate the difficulties presented above. The alumina insulating tubes have been replaced by a flat grooved alumina coil form on which the heater wire is wound. The void spaces inside the tubes are thereby eliminated. To further eliminate voids in the cavity and to prevent the heater shorting to the emitter housing during the casting, pieces of alumina are placed between the heater and the walls on all four sides and at the bottom of the cavity. In addition, by minimizing the voids, a reduction in the total number of casting operations needed to fill the cavity was possible.

The above described heater assembly is shown in Figures 2 and 3. The basic heater assembly, consisting of the alumina coil form, the bare tungsten heater and the molybdenum power leads, is shown in Figure 2. This basic heater assembly is then inserted in the emitter housing cavity along with the alumina insulating plates mentioned above. Figure 3 shows this assembly prior to the casting operation. The casting temperature (3550°F) prohibits the installation of the actual emitters until the casting is completed. The cavities for the two emitters are shown in Figure 3.

The first test device was constructed using the heater assembly of Figure 3 in conjunction with the ceramic base shown in Figure 4. The complete assembly, with the Nb-1Zr support plate, is shown in Figure 5. Three metal-ceramic joints were used in the complete assembly. These include:

1. Mounting of the encapsulated heater assembly (molybdenum) to the Lucalox ceramic base,
2. Mounting of the Nb-1Zr support plate to the Lucalox ceramic base and
3. Mounting of the reference collector surface to the Lucalox ceramic base.

All the above joints must, of course, maintain mechanical stability. In addition, joining operations (1) and (2) must take place at a low enough temperature (below 2300°F) to prevent damage to the thermionic emitters. The reference collector may be mounted at a higher temperature since this operation takes place prior to mounting of the encapsulated heater assembly (see Figure 4). For the first test assembly, tungsten/yttria was used (1) as the metallizing material for the posts on which the encapsulated heater assembly was brazed, (2) as the metallizing material for the braze between the ceramic base and its Nb-1Zr support plate and (3) to establish the reference collector surface. A nickel/niobium/titanium braze material was used to make both the heater assembly/ceramic and the ceramic/support plate joints.

The assembly of Figure 5 was mounted on the vacuum flange equipped with the micrometer head, installed in a chamber of the Vacuum Test Facility and baked-out to 1800°F with a chamber pressure of 3×10^{-8} torr. At this point, the encapsulated heater was energized and emitter activation was begun. After about 16 hours of operation, during which time the emission currents from both emitters were close to the values predicted by space-charge theory, the heater developed an open circuit. Preliminary examination indicated that the open circuit occurred in the first heater turn around the alumina coil form, which includes one of the plasma-sprayed joints. Efforts are being made to disassemble the heater to determine exactly where the failure occurred.

Present plans call for the use of rhenium wire instead of tungsten wire to improve the high-temperature characteristics of the heater and its external leads. The use of rhenium should bring about the following improvements.

1. A rhenium/rhenium weld joint will replace the tungsten/molybdenum plasma-sprayed joint now in use. A stronger, more stable connection should result.
2. The improved ductility of rhenium will minimize the breakage problem caused by bending the external heater leads after casting.

At present, 0.010 inch diameter rhenium wire has been obtained and will be used for the external leads.

4.0 Metal-Ceramic Seal Tests

The thermionic diode pressure transducer requires the use of metal-ceramic seals capable of maintaining vacuum integrity under high-temperature conditions. In the event of pressure capsule failure, the metal-ceramic seals must also be able to withstand liquid metal attack. At present, two approaches are being evaluated, both of which show promise of being satisfactory seal techniques.

1. A group of electrical terminal configurations (Figure 13, Reference 2) was obtained for evaluation. The terminals employed Nb-1Zr/Lucalox seals. One of the terminals was subjected to three consecutive test cycles with the following parameters.

   400 hours, 1800°F, 300 psia external argon pressure
   400 hours, 1800°F, 600 psia external argon pressure
   400 hours, 1800°F, 1000 psia external argon pressure

   No electrical or mechanical failure was observed during the course of the test (Reference 3).

Four of the group of terminals were prepared for, and at present are undergoing, potassium compatibility testing (see Section 5.0).

2. A life test was performed to evaluate the metal-ceramic joining technique presently being used to fabricate the initial thermionic diode pressure transducer models. The test sample (Figure 87, Reference 1), as with the electrical terminals mentioned above, contained Nb-1Zr/Lucalox seals. The Lucalox was metallized with a tungsten-yttria compound and a nickel/niobium/titanium braze material was used. The test schedule was as follows.

1000 hours, 1800°F, 500 psia internal argon pressure
100 hours, 1800°F, 600 psia internal argon pressure
100 hours, 1800°F, 700 psia internal argon pressure
100 hours, 1800°F, 800 psia internal argon pressure
100 hours, 1800°F, 900 psia internal argon pressure
1000 hours, 1800°F, 1000 psia internal argon pressure

> This test represents a more stringent evaluation than that performed on the terminals described above. Internal pressurization was used, resulting in tensile stress on the seals.
>
> The external pressurization applied to the terminal configuration produced compressive stress components which tended to maintain the seal integrity.

The test was terminated by a failure in the weld between the argon pressurization tubing (Nb-1Zr) and a Nb-1Zr disk, not involving the metal-ceramic joints under test. No electrical or mechanical failure was observed during the test. Efforts are being made to obtain a meaningful metallographic analysis of the test unit. Present plans call for preparation of test units for inclusion in the potassium compatibility test program.

5.0 Compatibility Test Program

The compatibility test chamber, with its associated heaters, shields and cooling water coils, was completed and installed on its vacuum system (100 liters per second ion type pump). Initial testing of the heaters and bake-out of the chamber internal surfaces were completed. The chamber temperature was monitored by two thermocouples. At 1800°F, there was a difference of less than 5°F between the readings of the thermocouples. At 1800°F, a chamber pressure of 5 X 10^{-7} torr was obtained; at room temperature, the chamber pressure dropped to 10^{-8} torr. Figure 6 shows the internal components of the test chamber. Figure 7 shows the test chamber in operation with its vacuum system.

Four single convolution test capsules of C-129Y, FS-85, T-222 and W-25 Re alloy and one test capsule containing four representative electrical terminals have been charged with liquid potassium and installed in the compatibility test chamber. The test capsules were suspended in the chamber from the capsule support plate shown in Figure 6. The test capsule design configurations are described in the Third Quarterly Report (Figures 54, 55 and 56, Reference 3). Figure 8 is a photograph of the set of completed test capsules.

Chemical analyses have been obtained of the potassium used to fill the five (5) test capsules. Samples of the potassium were cast in a stainless steel tube under a vacuum of 5 X 10^{-5} torr in an electron beam welding chamber, subsequent to filling the last capsule. The potassium samples were analyzed for oxygen by the mercury amalgamation method (helium cover gas) and for metallic impurities by spectrographic techniques. The results are as follows:

Oxygen as K_2O: 3.4/7.6 ppm

Metallic Impurities in KCl:

Ag, Al, Cb, Co, Cr, Cu, Fe, Mg,
Mn, Mo, Ni, Pb, Sn, Ti, Zr: less than 1 ppm

Ca: 1 ppm

V: less than 5 ppm

Na, Si: 5 ppm

A test program has been developed to evaluate the potassium compatibility of the four candidate alloys and the metal-ceramic seals. The program includes the preparation of the test capsules, the actual test procedures, and post-mortem testing. The program is defined as follows.

1. Preparation of Test Capsules
   a. Obtain a chemical analysis of the capsule materials.
   b. Obtain a chemical analysis of potassium batch.
   c. Fill the test capsules with potassium and identify the capsules.
   d. Weigh the completed test capsules.

2. Test Procedure
   a. Install the test capsules into the compatibility test chamber.
   b. Raise test chamber temperature to 1800°F. At no time shall the chamber pressure exceed 10^{-5} torr. For chamber temperatures above 700°F, the chamber pressure shall not exceed 5×10^{-7} torr.

c. Maintain test chamber temperature at 1800°F for a period of 100 hours. This will establish a potassium vapor pressure inside the test capsules of about 80 psia. After 100 hours, bring the test chamber to room temperature.

d. Perform 2.c above five times to accumulate a total of 500 hours operation at 1800°F.

e. Remove the test capsules from the test chamber.

3. Post-Mortem Tests

a. Weigh the test capsules.

b. Visually inspect the test capsules for possible failures.

c. Extract the potassium from the test capsules and perform a chemical analysis on the potassium.

d. Test the capsules for leakage on a mass spectrometer.

e. Cut up, section and perform metallographic examination of diaphragms, weld joints, braze joints, ceramics, etc.

f. Perform chemical or microprobe analysis of any corroded layers indicated in 3.e above.

At present, the five (5) test capsules are starting the second 100 hour test cycle outlined in 2.c and 2.d above. At the conclusion of the first 100 hour test cycle, the chamber pressure was 5 X 10^{-8} torr. Upon returning the chamber to room temperature, the pressure dropped to 6 X 10^{-10} torr. It is expected that the time needed to raise the chamber temperature to 1800°F, subject to the pressure requirements oi 2.b above, will decrease as operating time at 1800°F is accumulated. The chamber pressure at 1800°F should also decrease for each successive 100 hour test cycle as the chamber becomes more thoroughly out-gassed.

6.0 Differential Pressure Transducer

Preliminary analyses have been performed on three mechanical configurations for the differential pressure transducer (±5 psid). Figure 9 shows the three configurations schematically. Each configuration utilizes the deflection of a composite beam assembly consisting of (1) an outer tubular beam member and (2) an internal beam member whose termination acts as the active collector surface for the thermionic diode sensor. The outer beam member will be exposed to the liquid potassium environment. A wall thickness of 0.025 inch has been assumed to be sufficient for liquid metal containment. The internal beam member will be sealed from the liquid potassium and connected to the vacuum environment of the thermionic diode sensor.

The envisioned differential pressure transducer is of the same basic design as shown in Figure 1. For the differential pressure application, the thermionic diode sensor will be removed from the interior of the pressure capsule, isolated from the pressurized volumes, and probably embedded in the capsule housing. The pressures P_1 and P_2, whose differential $(P_1 - P_2)$ is to be monitored, are applied to the external and internal surfaces, respectively, of the pressure capsule. The main capsule housing inner diameter is assumed to be about 1.5 inches and the active collector surface extends 0.375 inch beyond the fixed support of the outer beam member.

For Case I (cantilever, end load), the displacement of the pressure capsule is transmitted directly to the cantilevered end of the outer beam member, establishing an end slope θ_1 relative to the horizontal position. The deflection of the active collector surface from equilibrium therefore becomes $(0.75 + 0.375)\,\theta_1$ inch less the deflection of the outer beam end. For Case II (end couple, one end fixed, one end supported), the pressure capsule displacement is transmitted by a yoke arrangement as a couple acting on a supported pivot point at one end of the outer beam member. If the end slope at the supported pivot point is θ_2, the active collector deflection from equilibrium becomes (1.5 + 0.375) θ_2 inch. Case III (cantilever, combination end load and end couple) involves the removal of the supported pivot point of Case II, allowing free movement of the outer beam member subject to the superposition of two actions; i.e., the pressure capsule deflection transmitted by the yoke arrangement as (1) an end load and (2) an end couple.

The applicable relations are as follows (Reference 4).

Case I (cantilever, end load)

$$\theta_1 = - \frac{W t^2}{2EI} \tag{1}$$

$$y_1 = \frac{W t^3}{3EI} \tag{2}$$

Case II (end couple, one end fixed, one end supported)

$$\theta_2 = \frac{MT}{4EI} = \frac{Wt(2t)}{4EI} = \frac{Wt^2}{2EI} \tag{3}$$

Case III (cantilever, combination end load and end couple)

Due to end load:

$$\theta_{3L} = -\frac{WT^2}{2EI} = -\frac{2Wt^2}{EI} \qquad (4)$$

$$y_{3L} = \frac{WT^3}{3EI} = \frac{8Wt^3}{3EI} \qquad (5)$$

Due to end couple:

$$\theta_{3C} = \frac{MT}{EI} = \frac{Wt(2t)}{EI} = \frac{2Wt^2}{EI} \qquad (6)$$

$$y_{3C} = -\frac{MT^2}{2EI} = -\frac{Wt(4t^2)}{2EI} = -\frac{2Wt^3}{EI} \qquad (7)$$

Combining end load and end couple effects

$$\theta_3 = \theta_{3L} + \theta_{3C} = 0 \qquad (8)$$

$$y_3 = y_{3L} + y_{3C} = \frac{2Wt^3}{3EI} \qquad (9)$$

In these equations, the sign convention employed is that end slopes and deflections are positive when in the upward direction (direction of force W in Figure 9) and

θ_1 = tubular outer beam end slope (Case I) (radians)

θ_2 = tubular outer beam end slope (Case II) (radians)

θ_3 = tubular outer beam end slope(Case III)(radians)

y_1 = tubular outer beam end deflection (Case I)(inch)

y_3 = tubular outer beam end deflection(Case III)(inch)

W = force exerted by pressure capsule on beam (assumed to be 1 pound)

t = tubular outer beam length (Case I) (0.75 inch)

T = tubular outer beam length(Cases II & III)(1.5 inch)

M = moment produced by pressure capsule (Cases II & III) (Wt pound-inches)

E = modulus of tubular outer beam material (assumed to be 30×10^6 psi)

I = moment of inertia of tubular outer beam section = $0.049\ (D^4 - d^4)$ inch^4

D = outer diameter of tubular outer beam (inch)

d = inner diameter of tubular outer beam (inch)

The results of the preliminary analysis may be presented as follows.

Tubular outer beam	O.D. (inch)	0.25	0.20	0.15
	I.D. (inch)	0.20	0.15	0.10
Active collector deflection (10^{-3} inch)				
	Case I	-0.052	-0.109	-0.294
	Case II	0.155	0.328	0.884
	Case III	0.083	0.175	0.472

The data show that Case II holds promise of providing an active collector deflection of 0.001 inch for reasonably sized tubular beams. The value of 0.001 inch full scale deflection has been found to result in a feasible output for the thermionic diode sensor (Figure 83, Reference 4). The difficulties inherent in Case II, which requires that the supported pivot point be inside the main capsule housing, make it mandatory to concentrate further analysis on Cases I and III, for which the fabrication requirements would be far less stringent. Case III has the further advantage of providing a purely vertical active collector travel, since $\theta_3 = 0$. Work is in progress to fabricate a mock-up of the Case III configuration to verify the results of the analysis.

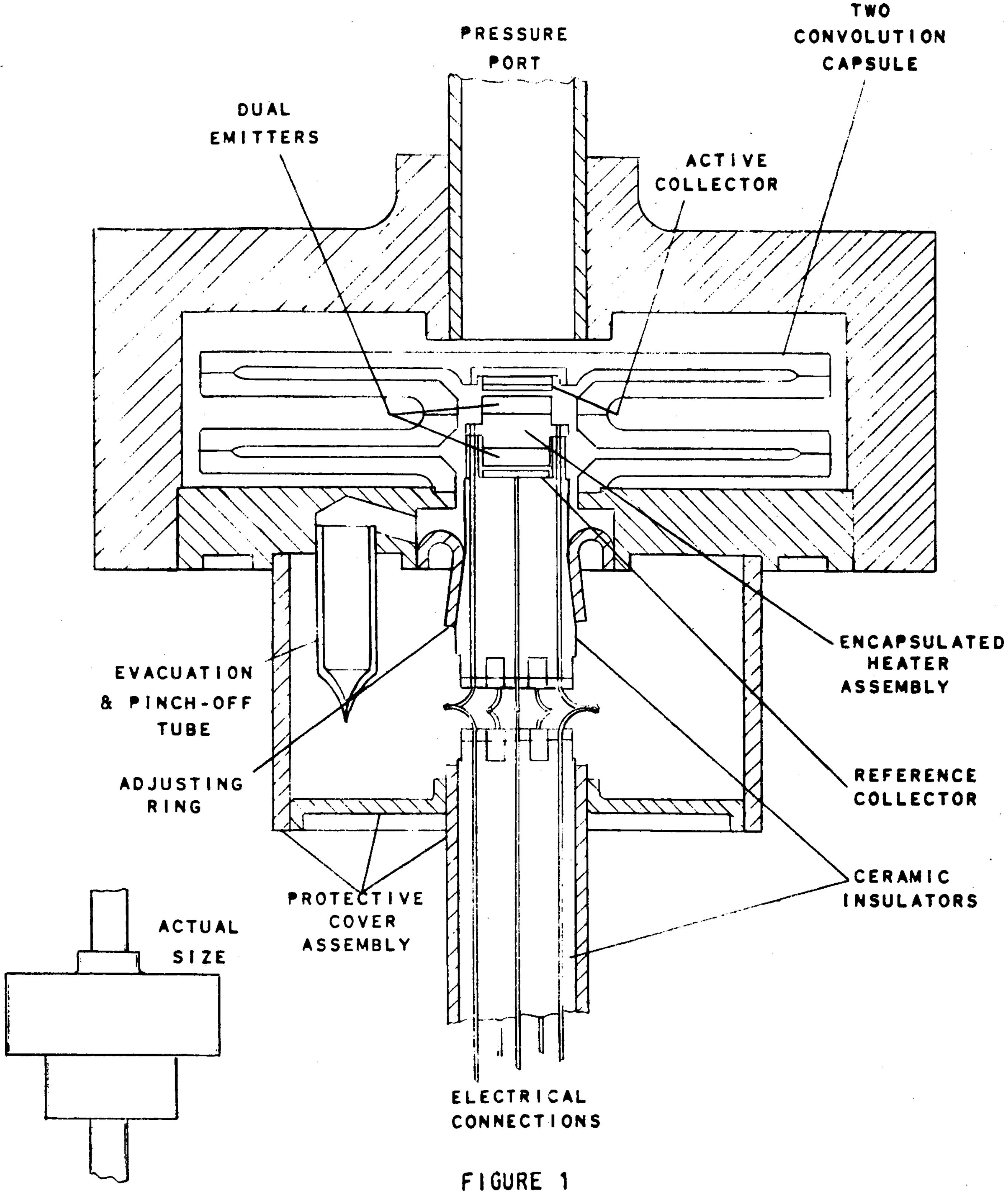


FIGURE 1

THERMIONIC DIODE PRESSURE TRANSDUCER

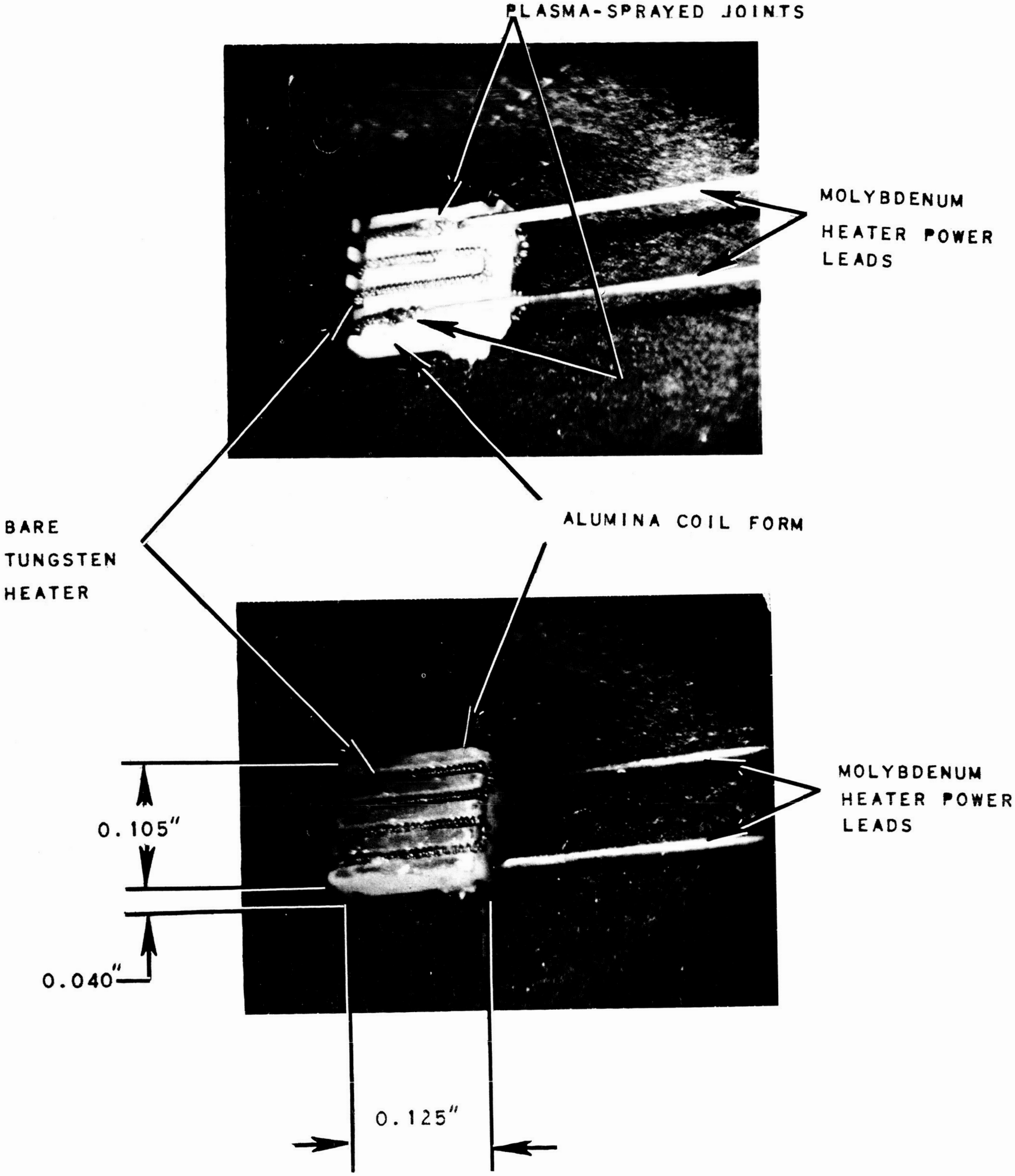


FIGURE 2

BASIC HEATER ASSEMBLY

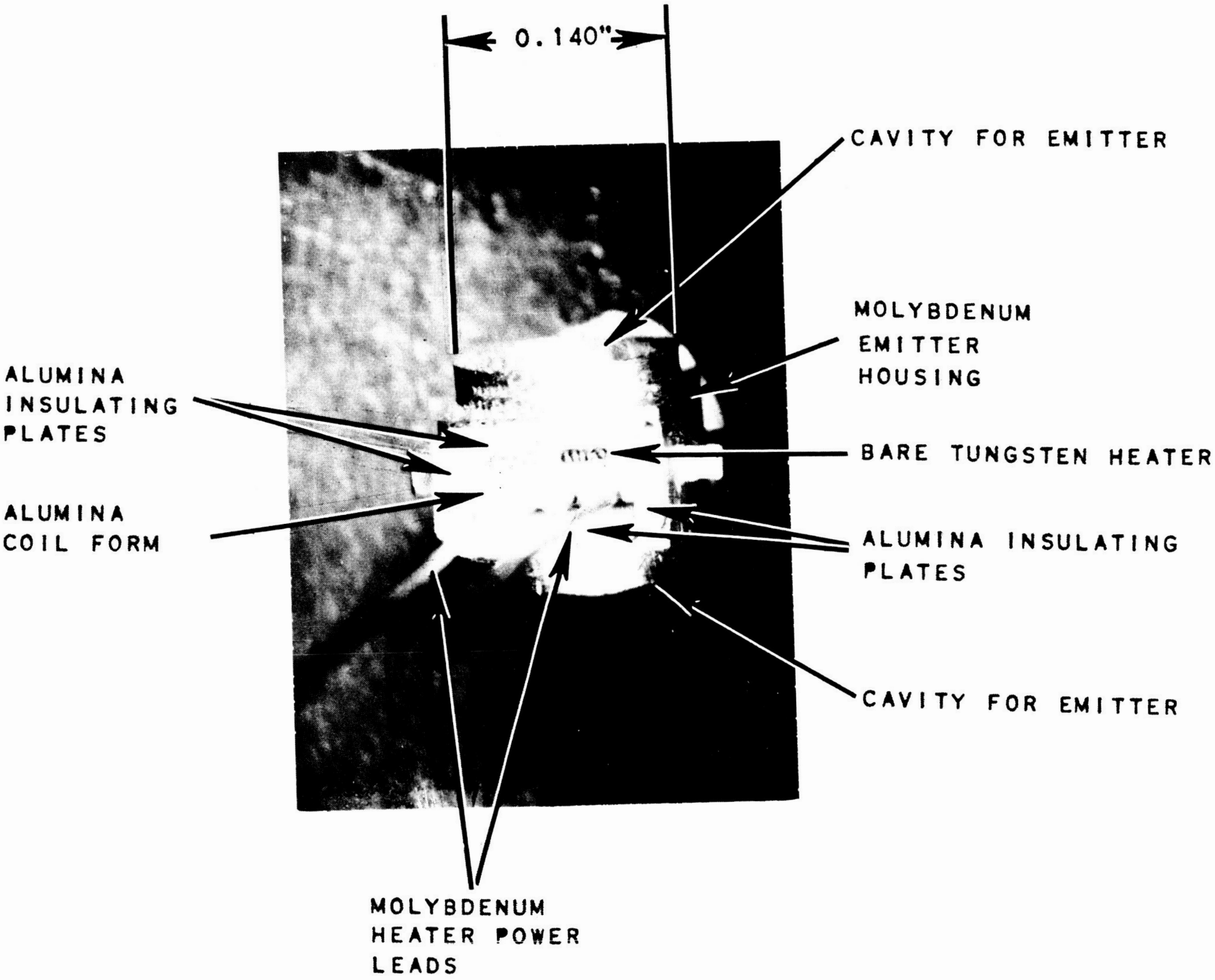


FIGURE 3
HEATER ASSEMBLY BEFORE CASTING

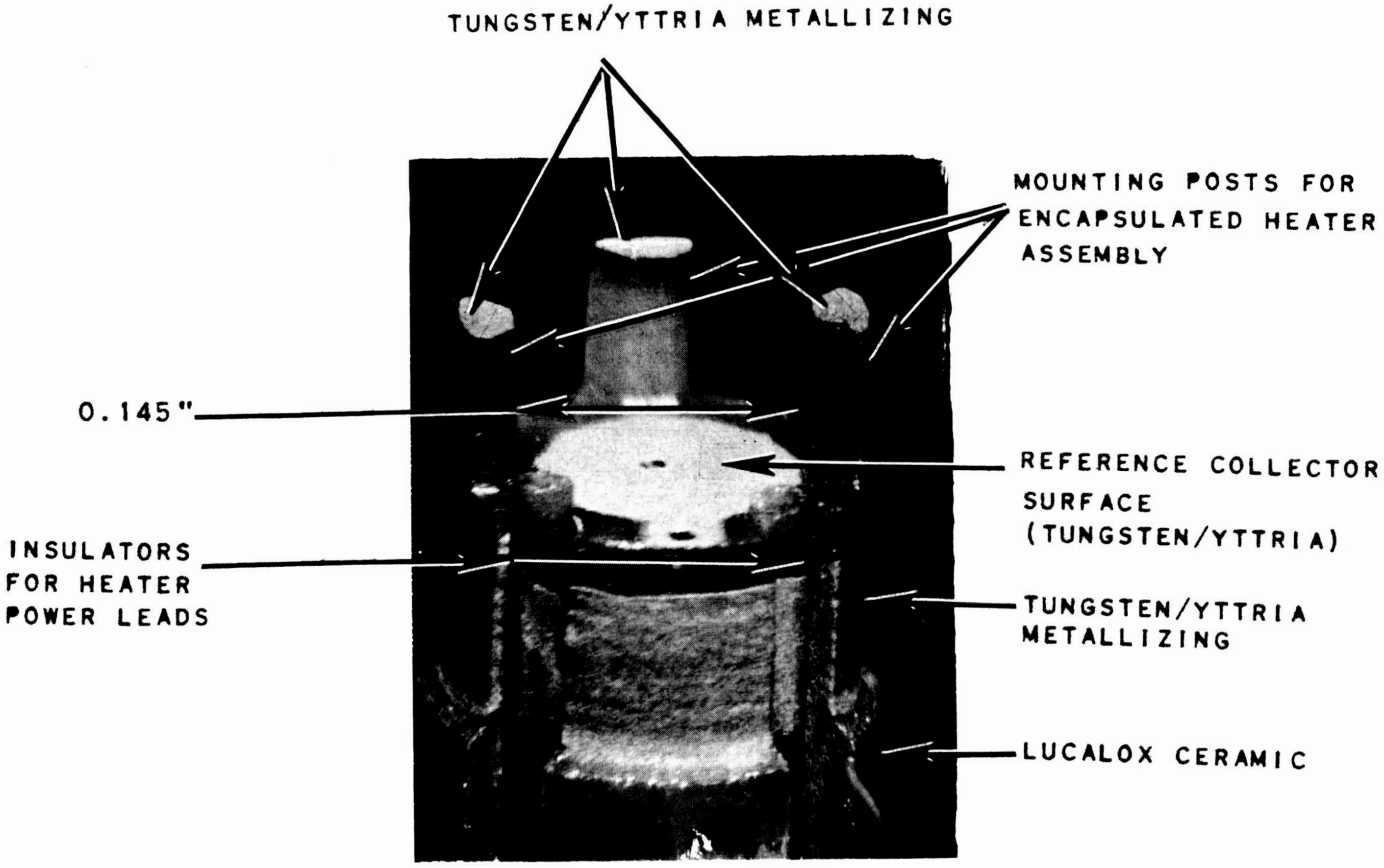


FIGURE 4

CERAMIC BASE

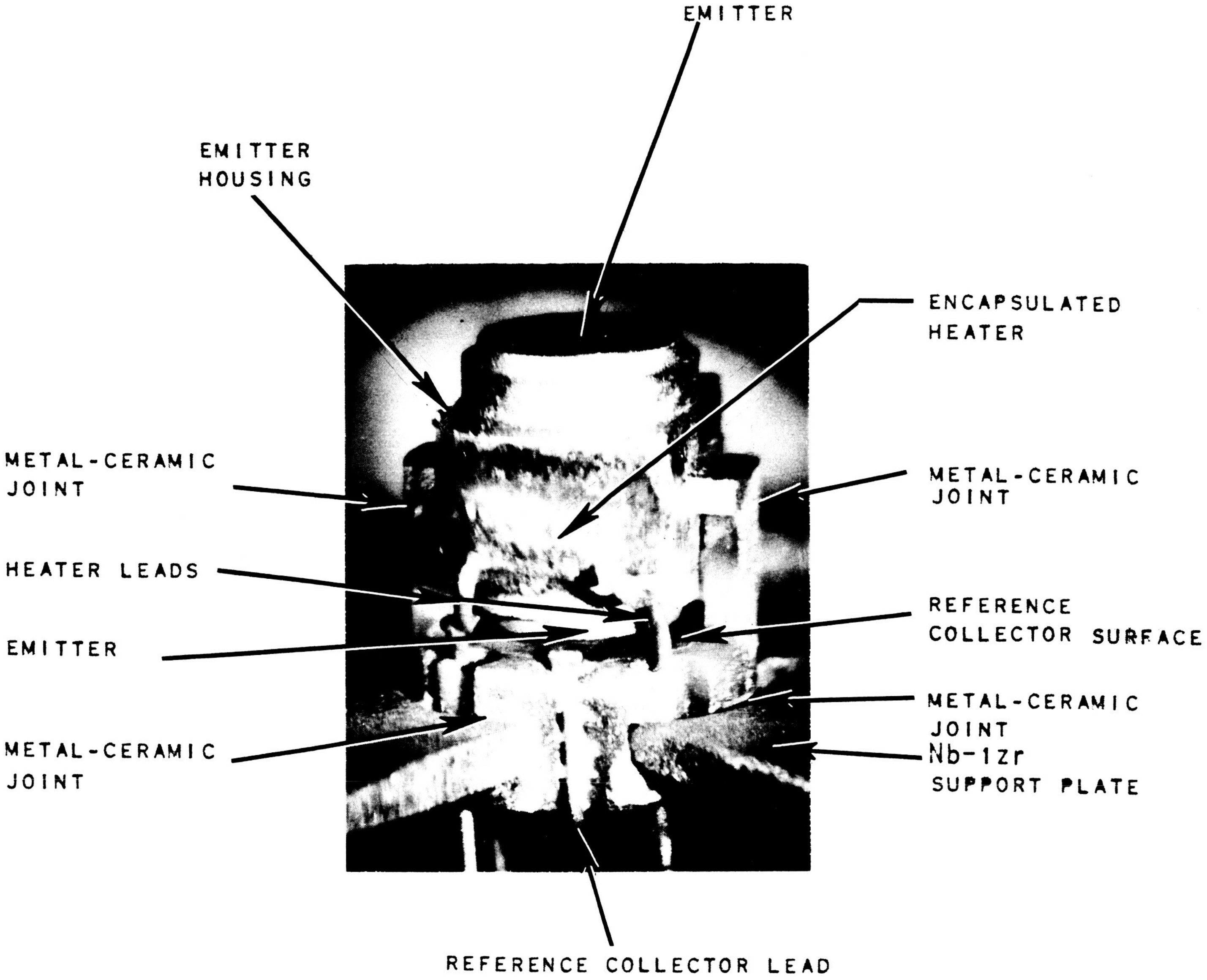


FIGURE 5

ENCAPSULATED HEATER-CERAMIC BASE-SUPPORT PLATE ASSEMBLY

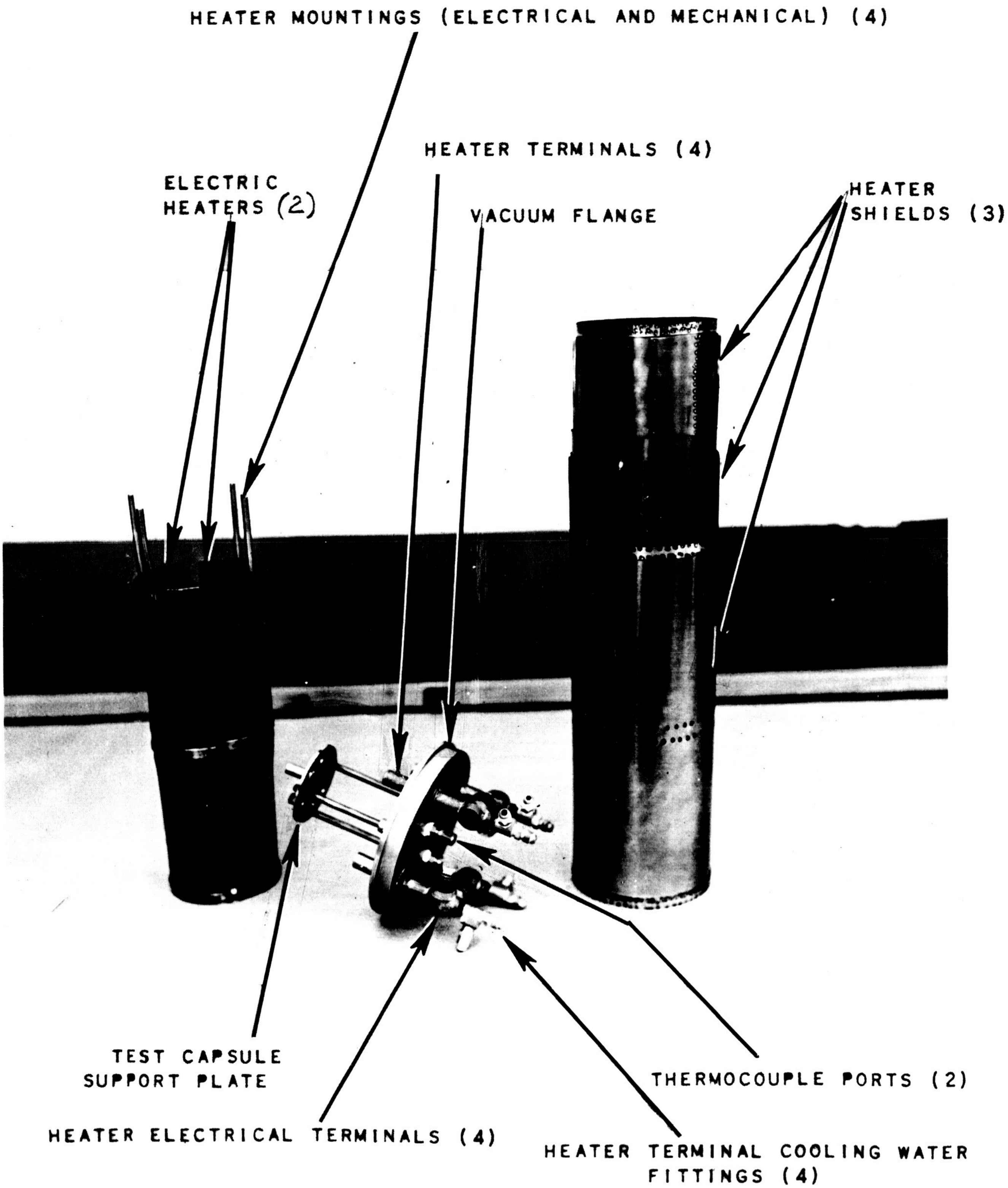


FIGURE 6

COMPATIBILITY TEST CHAMBER-INTERNAL COMPONENTS

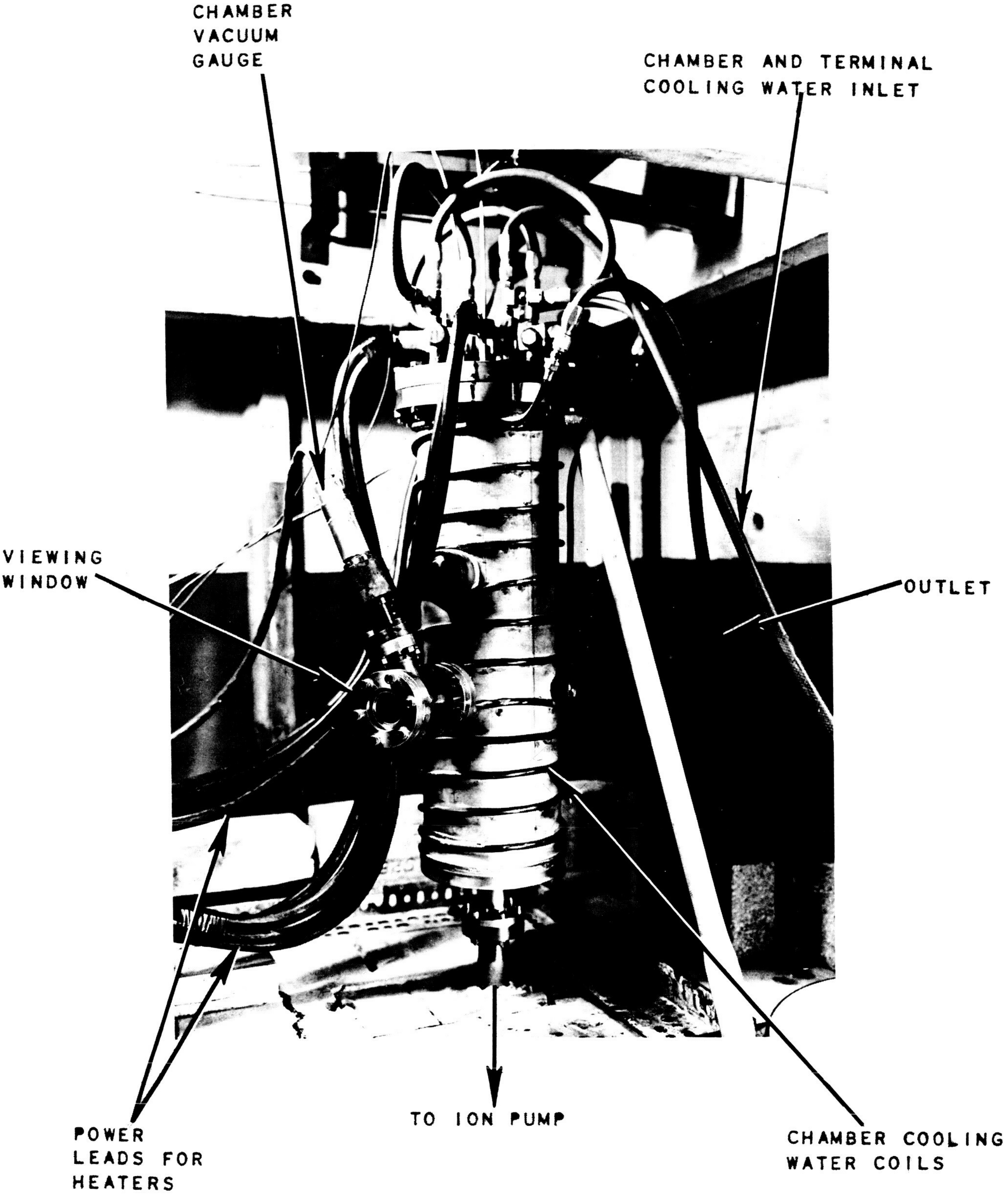


FIGURE 7

COMPATIBILITY TEST CHAMBER

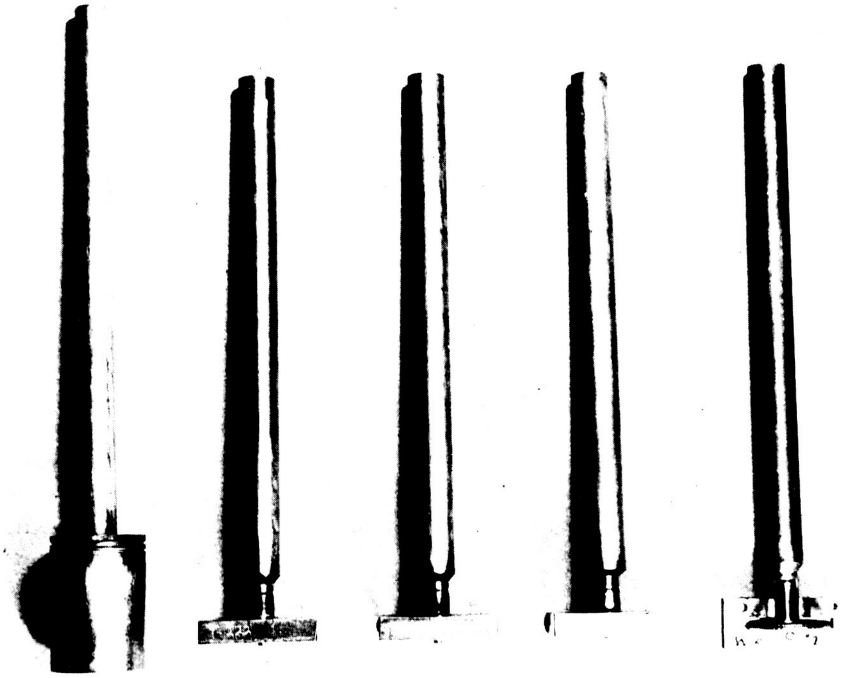
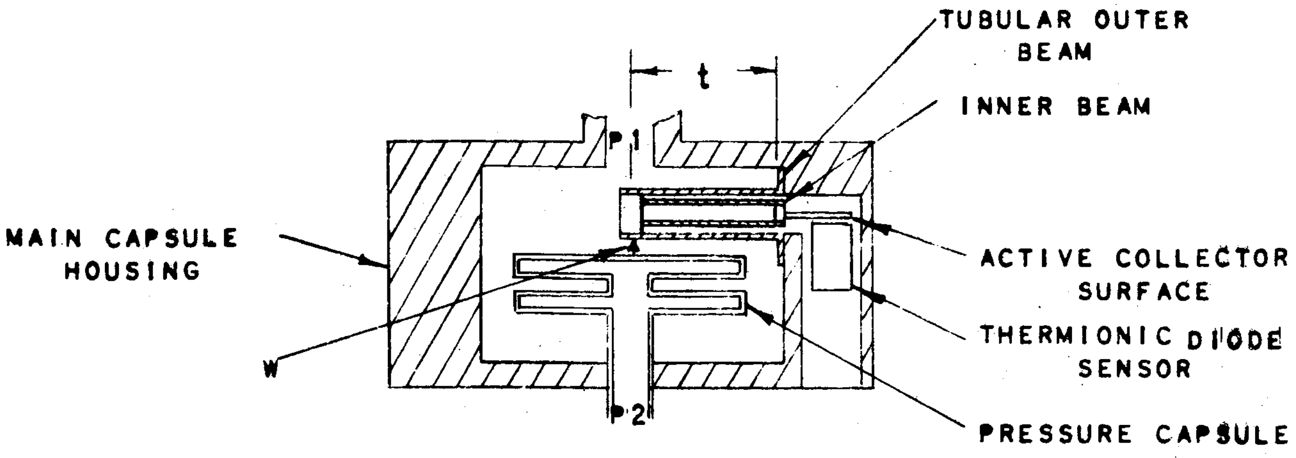
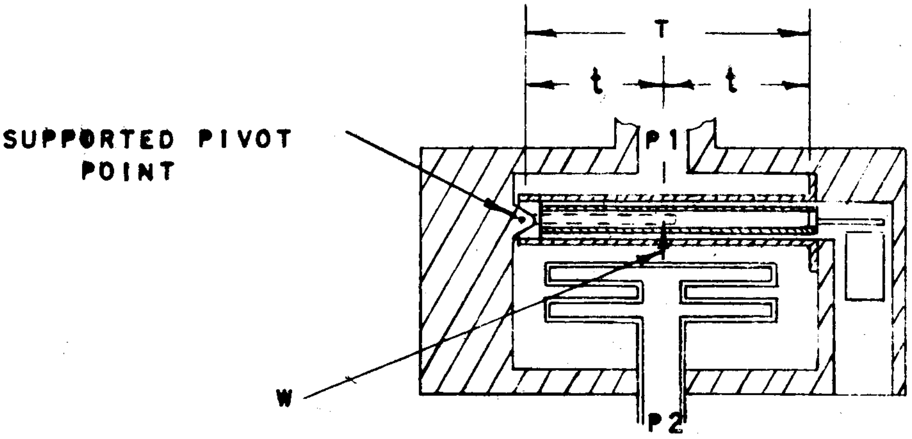


FIGURE 8

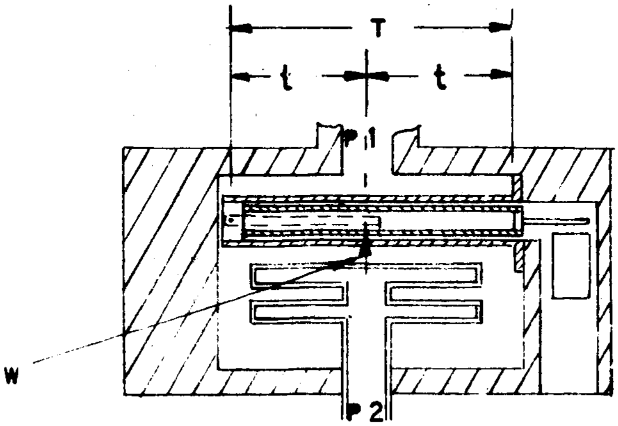
COMPATIBILITY TEST CAPSULES



CASE I - CANTILEVER, END LOAD



CASE II - END COUPLE, SUPPORTED PIVOT



CASE III - END COUPLE AND END LOAD

FIGURE 9

DIFFERENTIAL PRESSURE TRANSDUCER CONFIGURATIONS

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See also

Engdahl, R. E., ed.: First Quarterly Report Pressure Measuring Systems for Closed Cycle Liquid Metal Facilities. NASA CR-5414C July 2, 1964.

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