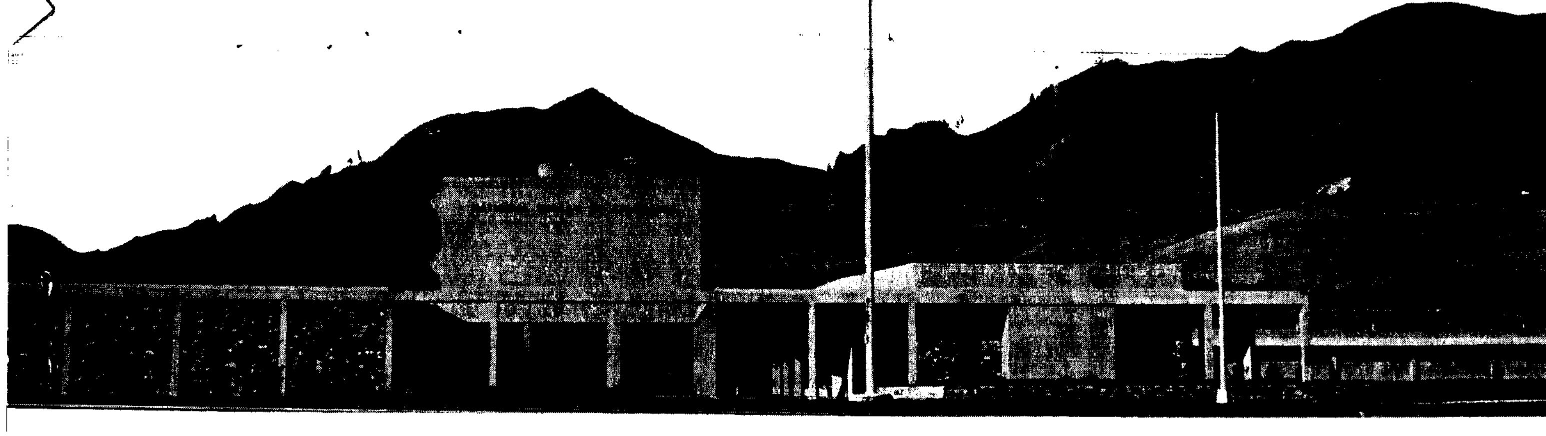

NBS REPORT

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FINAL REPORT TO THE SPONSOR ON HYDROSTATIC PRESSURE TRANSDUCERS OF CARBON AND YTTERBIUM

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FINAL REPORT TO THE SPONSOR

ON

HYDROSTATIC PRESSURE TRANSDUCERS

OF CARBON AND YTTERBIUM*

by

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Cryogenics Division
Institute for Materials Research
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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

FINAL REPORT TO THE SPONSOR ON
HYDROSTATIC PRESSURE TRANSDUCERS OF
CARBON AND YTTERBIUM

J. W. Dean and R. J. Richards

This report describes measurements of the hydrostatic pressure coefficients of electrical resistivity for carbon and ytterbium wire samples near 20°K, 76°K, and 300°K. Pressure coefficients ranging from $-2.16 \times 10^{-4} cm^2/kg$ to $-1.79 \times 10^{-4} cm^2/kg$ were found for carbon and $-1.75 \times 10^{-4} cm^2/kg$ to $-0.69 \times 10^{-4} cm^2/kg$ were found for ytterbium wire for pressures up to $70 \ kg/cm^2$. An analysis is given of the available bridge output from these sensors, and a prototype static pressure transducer is described. The frequency response of the carbon sensor is also included. Pressure transducers with adequate electrical output for moderate pressures can be made from these elements, but temperature stabilization will be required.

Key Words: Carbon, hydrostatic, pressure transducer, ytterbium.

1. Introduction

It has been shown [1] that problems with pressure transducers often result from the changes in the properties of the component materials. The temperature dependent change in the modulus of elasticity of the force-summing element--either a Bourdon tube, a diaphragm, or a bellows--is a cause of sensitivity shifts. The temperature dependence of the expansivity of the metals comprising the various transducer linkage elements is the prime contributor to the temperature dependence of zero shifts. In order to avoid these temperature effects and to increase precision, it was thought useful to design a pressure transducer without the usual diaphragm and linkage elements, a transducer which responds directly to a hydrostatic pressure.

It is known that if materials are subjected to an increase in hydrostatic pressure, their electrical resistivities will change [2]. This effect is not to be confused with a similar effect exhibited by mechanically-stressed semiconductor resistive elements found in some strain-gage pressure transducers and called piezoresistivity. The resistance will usually decrease with pressure, although in some metals it increases, and in cesium it decreases first and increases at high pressures. The effect has been studied by Bridgman [2] and is caused by an isotropic compression or by a distortion of the crystal lattice in the metal brought about by the external pressure [3].

For many metals, over limited ranges of pressure variations and at constant temperature, the resistance changes linearly with a variation of pressure and can be expressed as

$$R = R_o [1 + \beta (P - P_o)] \tag{1}$$

where R_o is the resistance at pressure P_o, usually one atmosphere, and $\beta = \frac{1}{R}\left(\frac{dR}{dP}\right)_T$ is the pressure coefficient of resistance for various metals as given in Table I.

The relative change of resistance for most metals is of the order of 10 percent for a change of pressure of 10, 000 kg/cm^2; for manganin it is only 2. 3 percent. The linear relationship of equation (1) is, of course, only an approximation. The maximum deviation from linearity in the pressure range from 0 to 12, 000 kg/cm^2 can be found in Table I. Experimentally the resistance change follows the pressure variation without noticeable time lag or hysteresis.

Table I

Average Pressure Coefficients of Resistance β for Different Metals in the Range 0 to 12,000 kg/cm² at Room Temperature (25° C)*

Metal	Pressure coefficient cm^2/kg	Max. deviation from linearity
Aluminum	-3.8 to -4.2×10^{-6}	-0.001
Antimony	$+11.1 \times 10^{-6}$	Large
Bismuth	$+21.4 \times 10^{-6}$	Large
Cadmium	-9.1×10^{-6}	-0.006
Copper	-1.8×10^{-6}	-0.0004
Iron	-2.3×10^{-6}	-0.0005
Lithium	$+7.72 \times 10^{-6}$	
Manganin	$+2.3 \times 10^{-6}$	
Mercury:		
Liquid below 6,500 kg/cm^2	-22.4×10^{-6}	
Solid above 7,600 kg/cm^2	-23.6×10^{-6}	
Platinum	-1.9×10^{-6}	-0.003
Silver	-3.3×10^{-6}	-0.001
Sodium	-37×10^{-6}	Large
0 - 1000 kg/cm^2	-60×10^{-6}	Large
Strontium, at 50°C	58.3 to 61.5×10^{-6}	

*P. W. Bridgman, Proc. Natl. Acad. Sci. U.S., 3, 10 (1917). Conversion to engineering units of 1/psi may be done by multiplying by 0.0703.

The pressure sensitivity (or the coefficient "β" above) is largest for the alkali metals and for bismuth and antimony, but transducers made from such materials may not be practical, and others may be preferred. Mercury has the advantage that it can be produced with uniform purity, but it undergoes a phase transition near room temperature in the pressure range between 6,500 and 7,600 kg/cm^2; in this range the pressure coefficient of resistance changes slightly (see Table I).

Bridgman [2] has described a pressure-sensing element made from a coil of manganin wire. Manganin was chosen because of its very low temperature coefficient, $\frac{1}{R}\left(\frac{\partial R}{\partial T}\right)_P$, allowing normal temperature variations to be ignored. This device is suitable for measuring very high pressures, but does not have sufficient sensitivity to allow the use of field type instruments to read its output over the engineering pressure range of 0 to 70 kg/cm^2. Thus, if a hydrostatic pressure transducer useful to engineering applications is to be built, a material with a much higher pressure coefficient than those given in Table I must be found. Furthermore, the material should have a low temperature coefficient and both the pressure and temperature coefficient should be independent of temperature over a wide temperature range.

A literature search showed that both ytterbium and carbon have anomalously high pressure coefficients at room temperature. Work by Stromberg and Stephens [4] gave experimental values of the relative resistance of ytterbium at very high pressures and extrapolated the resulting curve to zero pressure. Estimates based on this work showed the pressure coefficient of ytterbium to be some thirty times larger than that of manganin. Measurements by Miller [5] on carbon showed that its pressure coefficient is near one hundred times greater than that of manganin. Thus, materials were available with sufficiently large pressure coefficients to make hydrostatic pressure transducers for application to pressures encountered in engineering. However, little was known about the temperature dependency of the pressure coefficients.

This work measures the pressure coefficient of ytterbium and carbon at liquid hydrogen, liquid nitrogen and room temperature. An analysis of the application of these materials to pressure transducers is made and a prototype static pressure transducer is described.

2. Experimental Procedure for Resistance Measurements

The electrical resistivity and the pressure coefficient of ytterbium was measured as a function of pressure near 20°K, 76°K, and 300°K in a temperature-controlled liquid bath cryostat. An encapsulated germanium thermometer was used to determine the sample-holder temperature stability prior to making the ytterbium and carbon measurements.

2.1 Description of Cryostat and Probe Sample Holder

The apparatus consisted of a small dewar constructed with a copper inner wall attached to a stainless steel neck and a stainless steel outer wall. The inside dimensions of the dewar were 2-1/4 inches in diameter by 24 inches deep. The top plate contained a fill tube, vent, liquid level indicators (two carbon resistors) and a "quick" coupling for the probe sample holder. The probe (see figure 1) was made from a 1/2-inch stainless steel tube with a copper block on one end for thermally attaching the lead wires and mounting the samples. This copper block was covered with a copper tube sealed by pipe threads for pressure tests. The probe was built for a working pressure of 1000 psi. At the other end of the probe are a pressure tight seal for the lead wires and a fitting for pressurizing the sample with helium gas. The probe was inserted through the quick coupling ("O" ring seal) which allowed it to be moved in and out of the liquid for thermal cycle tests. A schematic sketch (figure 2) shows the associated piping, instrumentation, valves, and manostat that were used to control the pressure on the liquid bath in the dewar. The change in resistance was measured with a commercial Mueller bridge using a four lead system.

2.2 System Temperature Stability

A fully encapsulated germanium thermometer was installed in the probe to verify the temperature stability of the test section. The temperature remained steady within 0.01°K as the pressure was varied from 0 to 1000 psi and the liquid level dropped some 6 inches. However, these results were not immediately obtained. Inadequate lead-wire tempering--

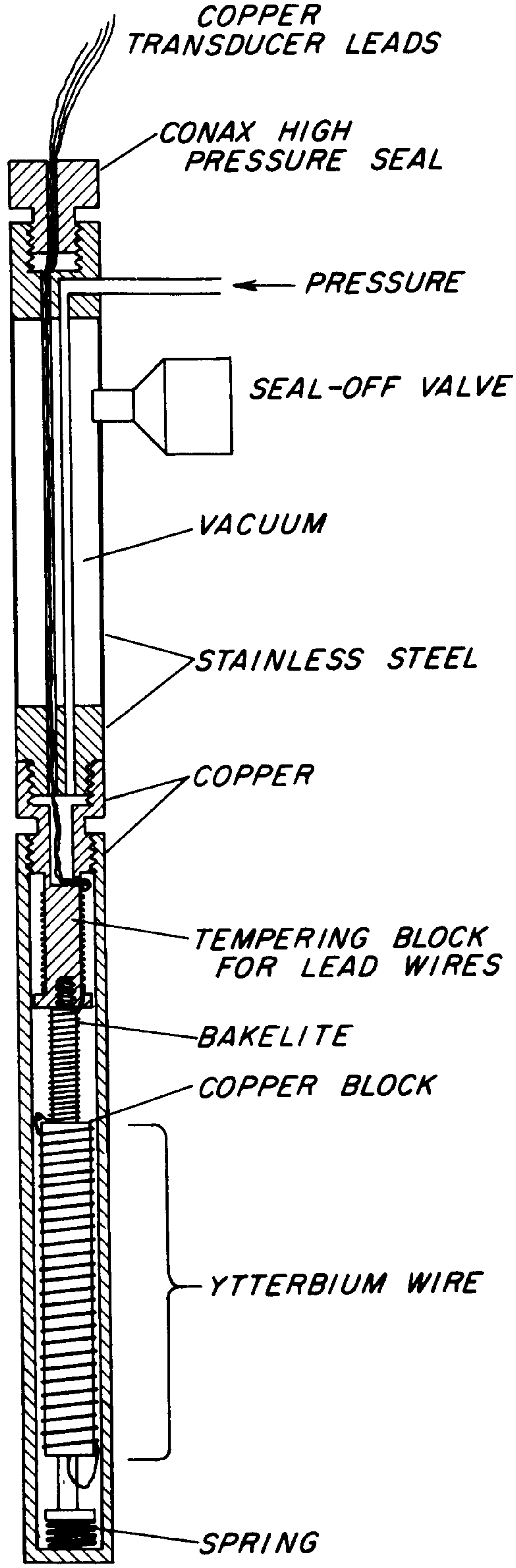

Figure 1. Probe Sample Holder.

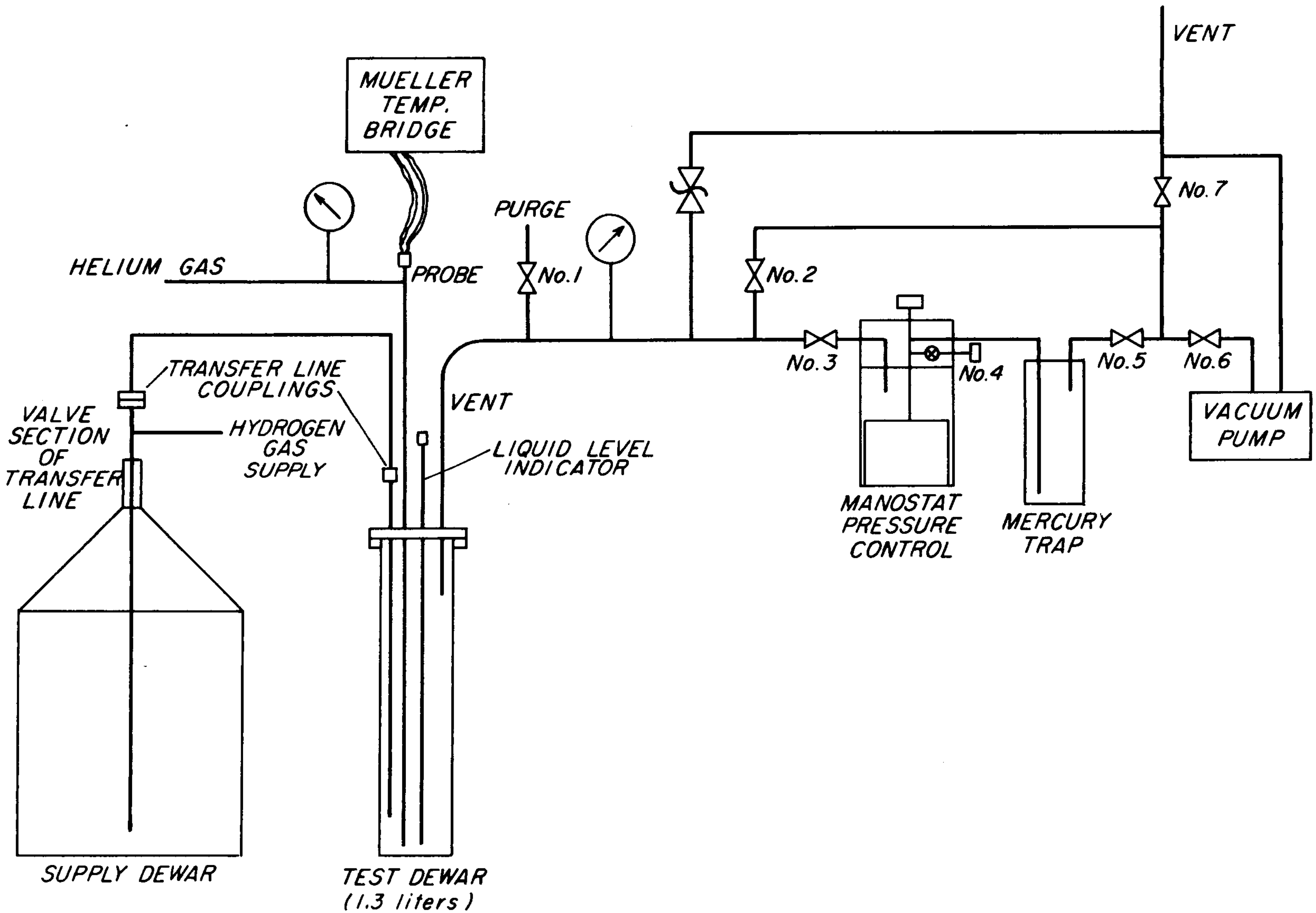

Figure 2. Schematic of Apparatus.

thermally anchoring the lead wires to the copper section--caused the germanium thermometer to at first detect liquid level changes. This was remedied by installing the tempering block shown in figure 1.

Pressure coefficient measurements were made at 20°K and 300°K on a commercial 1000 ohm, 0.1 watt carbon composition, resistor primarily to verify the results obtained with the measurement system using cryostat baths of liquid nitrogen and water. The results obtained agreed well with results reported by Miller, et al. [5] and Herr, et al. [6].

2.3 Ytterbium Samples

Samples of ytterbium metal purchased for these tests were wires 0.127 cm in diameter, 30 cm long with a resistance of 0.0816 ohms at room temperature and with a stated purity of 99.9+ percent. The wire was obtained from a commercial supplier at a cost of $3.00 per cm. Because of the small resistance, one of the wires was swaged to 0.068 cm in diameter and 86.4 cm long, increasing the resistance to 0.830 ohms. Further swaging without annealing caused breakage due to work hardening. The equipment needed to anneal the sample without rapid oxidation was not available. The samples as received were very ductile, oxidized rapidly, and were very difficult to solder or weld. Copper leads were attached to the ytterbium by wrapping them on, and then "scrubbing" a gallium wetting agent onto the sample without flux. All of the fluxes tried caused rapid oxidation.

3. Resistance Measurement Results

3.1 Resistivity of Ytterbium Wire as a Function of Temperature

Results of resistivity as a function of temperature were obtained on the two samples of ytterbium wire: (1) 0.127 cm dia. x 30 cm long, and (2) 0.068 cm dia. x 86.4 cm long. The rest of the tests were made on the long sample only. The measured resistivity of the short sample was as given in Table II and the long sample is shown in Table III.

TABLE II

Resistivity of Ytterbium Short Sample

300°K	3.31×10^{-5} ohm cm
76°K	2.08×10^{-5} ohm cm
20°K	1.24×10^{-5} ohm cm

TABLE III

Resistivity of Ytterbium Long Sample

300°K	3.46×10^{-5} ohm cm
76°K	2.01×10^{-5} ohm cm
20°K	1.22×10^{-5} ohm cm

3.2 Ytterbium Resistance Repeatability

Resistance repeatability was determined by thermal cycling the 86.4-cm sample between room temperature and liquid hydrogen temperature (approximately 19.6°K). Nine cycles produced a maximum deviation of 0.0004 ohms from an average resistance value at liquid hydrogen temperature of 0.2717 ohms.

Resistance repeatability for pressure cycling was determined on the same sample. Six pressure cycles were run at liquid hydrogen temperatures from zero gauge pressure (approximately 0.86 kg/cm^2 at Boulder, Colo.) to 35.15 kg/cm^2 and 70.30 kg/cm^2 gauge. Results of these tests are shown in Table IV.

TABLE IV

Resistance Repeatability for Pressure Cycling near 20°K

	Test pressure, gauge $35.15\ kg/cm^2$	$70.30\ kg/cm^2$
Number of cycles	6	6
Average resistance at zero gauge pressure-ohms	0.2740	0.2890
Maximum deviation at zero gauge pressure-ohms	± 0.0001	± 0.0003
Average resistance at full test pressure-ohms	0.2723	0.2855
Maximum deviation at full test pressure-phms	± 0.0001	± 0.0001

The difference of the average resistance values of the $35.15\ kg/cm^2$ and the $70.30\ kg/cm^2$ tests is due to obtaining different bath temperatures. The local barometer and manostat setting varied for the two series which were done on different days. The reported deviations are one-half of the range of the data; however, they represent the limitations of the measurement system rather than the repeatability of the ytterbium. The limit of error of the resistance bridge is ± 0.0002 ohms, which is very nearly the deviation observed. Thus better sample preparation techniques need to be developed in order to obtain higher resistance samples or better instrumentation is required.

The repeatability of carbon during pressure and temperature cycling has been reported elsewhere [5], [7] as slightly less than one percent of the absolute resistance.

3.3 Pressure Coefficient of Resistance

Resistance measurements as a function of pressure were taken near 300°K, 76°K, and 20°K for the 86.4-cm ytterbium sample and a 1000 ohm 0.1 watt carbon composition resistor. The results of these measurements are shown in figure 3. The slope of the curves are the pressure coefficients given in Table V.

TABLE V

Ytterbium and Carbon Pressure Coefficients of Resistance

Temperature °K	Pressure coefficient cm^2/kg Carbon	Ytterbium
300	-2.16×10^{-4}	-0.69×10^{-4}
76	-1.82×10^{-4}	-1.27×10^{-4}
20	-1.79×10^{-4}	-1.75×10^{-4}

the magnitude of these values--of the order of fifty times larger than manganin--the linearity of the resistance-pressure function is what makes ytterbium and carbon attractive as pressure transducers.

4. Analysis of Hydrostatic Coefficient Applications to Pressure Instrumentation

The electrical resistivity of conducting materials is a function of pressure, temperature, and mechanical strain as well as other variables such as magnetic fields and nuclear radiation. This work is concerned with separating the primary effects of temperature and pressure in the absence of other variables. The resistive elements are assumed to be mounted in a strain-free manner. Writing the total differential for the primary variables yields the expression

$$dR = \left(\frac{\partial R}{\partial T}\right)_P dT + \left(\frac{\partial R}{\partial P}\right)_T dP. \tag{2}$$

Dividing both sides of equation (2) by R, the results are as follows:

$$\frac{dR}{R} = \alpha\, dT + \beta\, dP, \tag{3}$$

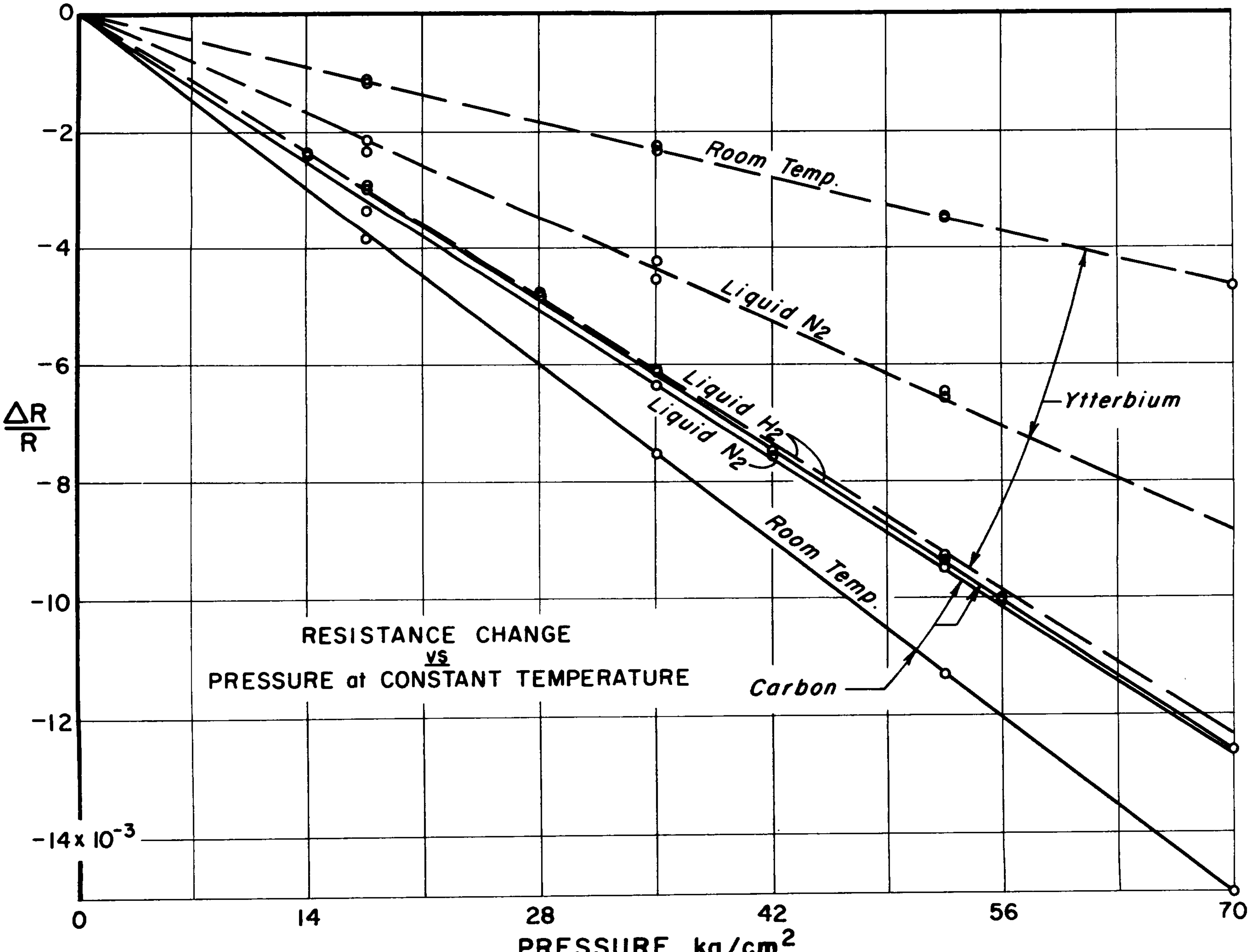

Figure 3. Resistance Change vs. Pressure at Constant Temperature.

where

$$\frac{1}{R}\left(\frac{\partial R}{\partial T}\right)_P = \alpha$$

$$\frac{1}{R}\left(\frac{\partial R}{\partial P}\right)_T = \beta.$$

Equation (3) is an expression for the unit change of resistance of a material in terms of the pressure coefficient and the temperature coefficient and is more general than equation (1).

Equation (3) indicates that the application of a resistive element to a hydrostatic pressure transducer requires that a material be found that has an α value much less than the β value, or that the temperature must be held constant, or that some means must be found to reject the temperature dependent contribution to the resistance change. It also says that even if the effect of α is negated by common mode rejection techniques, sensitivity temperature compensation is necessary since β is a function of temperature as shown in Table V.

A possible criterion for choosing a material for applying to a hydrostatic pressure transducer is the ratio $|\beta/\alpha|$. A material with the largest $|\beta/\alpha|$ will have the greatest pressure sensitivity combined with the least temperature sensitivity. This ratio is given in Table VI for a few selected materials at 25° C.

TABLE VI

Figure of Merit $|\beta/\alpha|$ at 25°C

Material	α °C^{-1}	β cm^2/kg	$\lvert\beta/\alpha\rvert$ °C-cm^2/kg
Carbon	-2.5×10^{-4}	-2.16×10^{-4}	0.85
Copper	-39.0×10^{-4}	-1.8×10^{-6}	0.0005
Manganin	$+0.2 \times 10^{-4}$	$+2.3 \times 10^{-6}$	0.115
Platinum	$+30.0 \times 10^{-4}$	-1.9×10^{-6}	0.0006
Ytterbium	$+14.0 \times 10^{-4}$	-0.69×10^{-4}	0.05

From the above table carbon is seen to be by far the best candidate for a hydrostatic pressure sensor. However, at liquid hydrogen temperature the temperature sensitivity increases by a factor of two hundred causing the figure of merit to decrease. Manganin was not investigated at low temperature since its pressure sensitivity is too low except for application to extreme pressures. Ytterbium's figure of merit remains more nearly independent of temperature.

Bridgman chose to use manganin wire because of his interest in extreme pressures and the insensitivity of manganin to temperature variations. The authors know of no similar suitable material that will allow this approach to pressure measurement in the normal engineering pressure range (say up to 350 kg/cm^2). We have used a temperature stabilized Wheatstone bridge with carbon sensors in the construction of a hydrostatic pressure transducer.

The linearized equation for the output of a Wheatstone bridge (near null) is

$$de_o = \frac{ne_i dR}{4R} \tag{4}$$

where

e_o = output voltage

e_i = input voltage

n = number of active arms.

Equation (4) is derived on the basis that equal resistance variation in the bridge arms does not contribute to the bridge output. This is an idealization that may be expressed in the terminology of the electronic engineer as an infinite common mode rejection ratio. The common mode that we are interested in rejecting is temperature. To achieve this in a bridge requires that each bridge arm have identical absolute resistance and temperature coefficient values as well as identical temperatures. This is difficult to achieve, to say the least, but the use of the bridge transducer configuration is helpful over small temperature ranges.

An estimate of a hydrostatic pressure transducer output may be made by writing equation (4) in terms of the pressure coefficient; thus,

$$de_o = \frac{ne_i \beta dp}{4}. \qquad (5)$$

The electrical output of a bridge with two active arms using carbon composition resistors at room temperature for a pressure of 70 kg/cm^2 and a 19 volt excitation is 0.075 volts. The output for ytterbium at room temperature would be 0.024 volts. Although these are low voltages they are well within the capability of even field instruments to read accurately.

5. Prototype Pressure Transducers

5.1 Static Pressure Application

The ytterbium sample resistance was not sufficient to construct a prototype pressure transducer. The low sample resistance obtained in this work would have required 10 amp of current for the preceding example. However, the use of the 1000-ohm carbon resistor results in a current of 0.010 amp and a power dissipation of 0.025 watts per resistor.

A prototype pressure transducer was constructed by sealing four 0.1-watt carbon resistors into a copper block with epoxy resin as shown in figure 4. What is seen in figure 4 is the resistor lead wires extending out of the epoxy. Prior to installing the resistors, two ports had been drilled at right angles to the resistor holes serving to pressurize the resistors. Stainless steel tubes, carrying the pressurizing gas, supports the copper block. The copper block is suspended inside a stainless steel can (not shown) which is bolted to the top flange. The can is evacuated, thermally isolating the copper block. The heater and temperature sensors then control the temperature of the block near 50°C by means of a

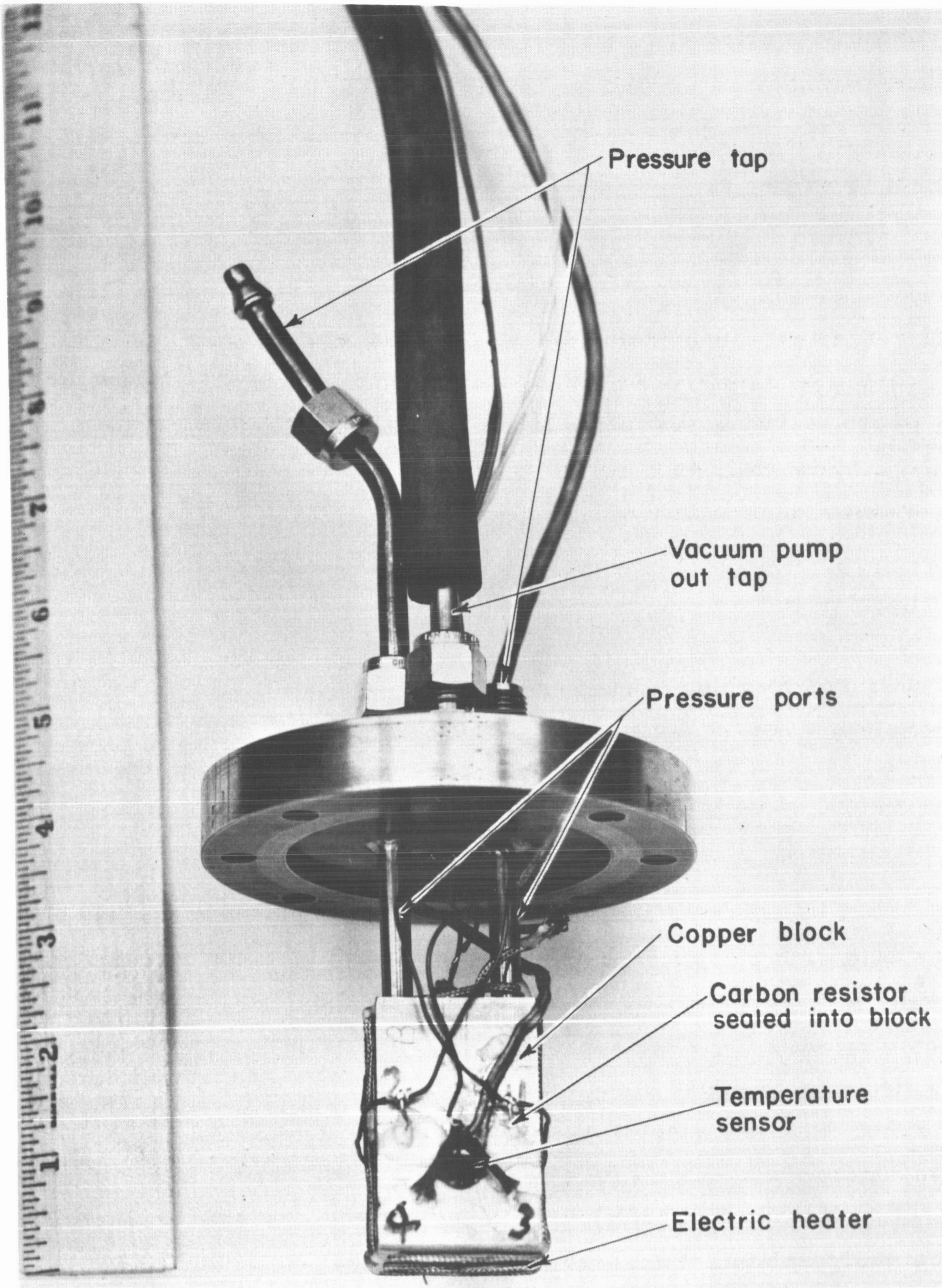

Figure 4. Prototype Carbon Resistor Static Pressure Transducer.

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temperature regulator. This temperature control scheme has since been simplified by the use of a bi-metallic strip regulator that controls the temperature to ± 0.5°C.

The output of the instrument was read on a digital voltmeter. A slight adjustment of the excitation voltage gave 0.070 volts output for 70 kg/cm^2 pressure allowing a direct reading of the pressure from the digital voltmeter. The cavity in the copper block containing the carbon resistor essentially forms a Helmholtz resonator, making this instrument only useful for static pressure measurements because of the pressure signal amplification caused by resonance in the cavity.

5.2 Dynamic Pressure Applications

It was reasoned that since the 0.1-watt carbon resistors were small and stiff that they ought to be able to sense high frequency pressure pulses. Therefore single resistors were tested in the sinusoidal pressure generator facilities of the Ground Test Instrument Laboratory of Marshall Space Flight Center at Huntsville, Alabama.

The sinusoidal pressure generator consisted of a piston vibrating in a cylinder driven by an electronic vibrator. Hydraulic oil flowed through the cylinder. The magnitude of the pressure pulse was controlled by the position of the piston in the cylinder and the frequency by the excitation to the vibration. Pressure pulses of 14 to 0.5 kg/cm^2 were obtained at frequencies ranging from 100 Hz to 10 kHz.

Three samples were prepared by epoxying the resistor into a small stainless steel pressure fitting. Two samples allowed the resistor to extend into the flow stream while the third sample was covered with the epoxy and recessed into the fitting.

Since only a single arm bridge could be used and low pressures were obtained, an amplifier had to be used in the transducer output before the signal could be read on a scope. The reference signal was obtained from a quartz piezoelectric pressure transducer and was displayed on the second beam of the scope. The output of both pressure transducers were first determined by comparison with a static pressure. Amplitude ratios were then obtained by comparing the signal height of both scope beams as a function of frequency.

The results of these tests are shown in figure 5. A flat frequency response was obtained to 2kHz for the bare carbon, and 3kHz for the epoxied carbon. Apparently the epoxy acted to stiffen the system, changing the response from typically a first order to a second order system.

6. Discussion and Conclusions

Hydrostatic pressure transducers may be made from ytterbium and carbon. Ytterbium appears to be more repeatable to both temperature and pressure cycling. This needs to be verified by performing more tests on different samples of higher resistance before conclusive proof is established. On the basis of the meager sample size of the test performed in this work and the obtainment of a repeatability of 0.1 percent in the absolute resistance, the precision of the measurement system, it seems possible that an unusually precise pressure transducer might be made from ytterbium. Such a pressure transducer would be limited to static pressure applications and would require temperature stabilization. A rather expensive instrument could be made now, incorporating a temperature stabilized ytterbium sensing element in a precision bridge powered by a low voltage source. The bridge output would have to be read by a microvoltmeter. A more practical instrument awaits the development of a higher resistance ytterbium sensing element.

The authors are aware of one commercial pressure transducer being manufactured in England that apparently uses carbon as a sensing element

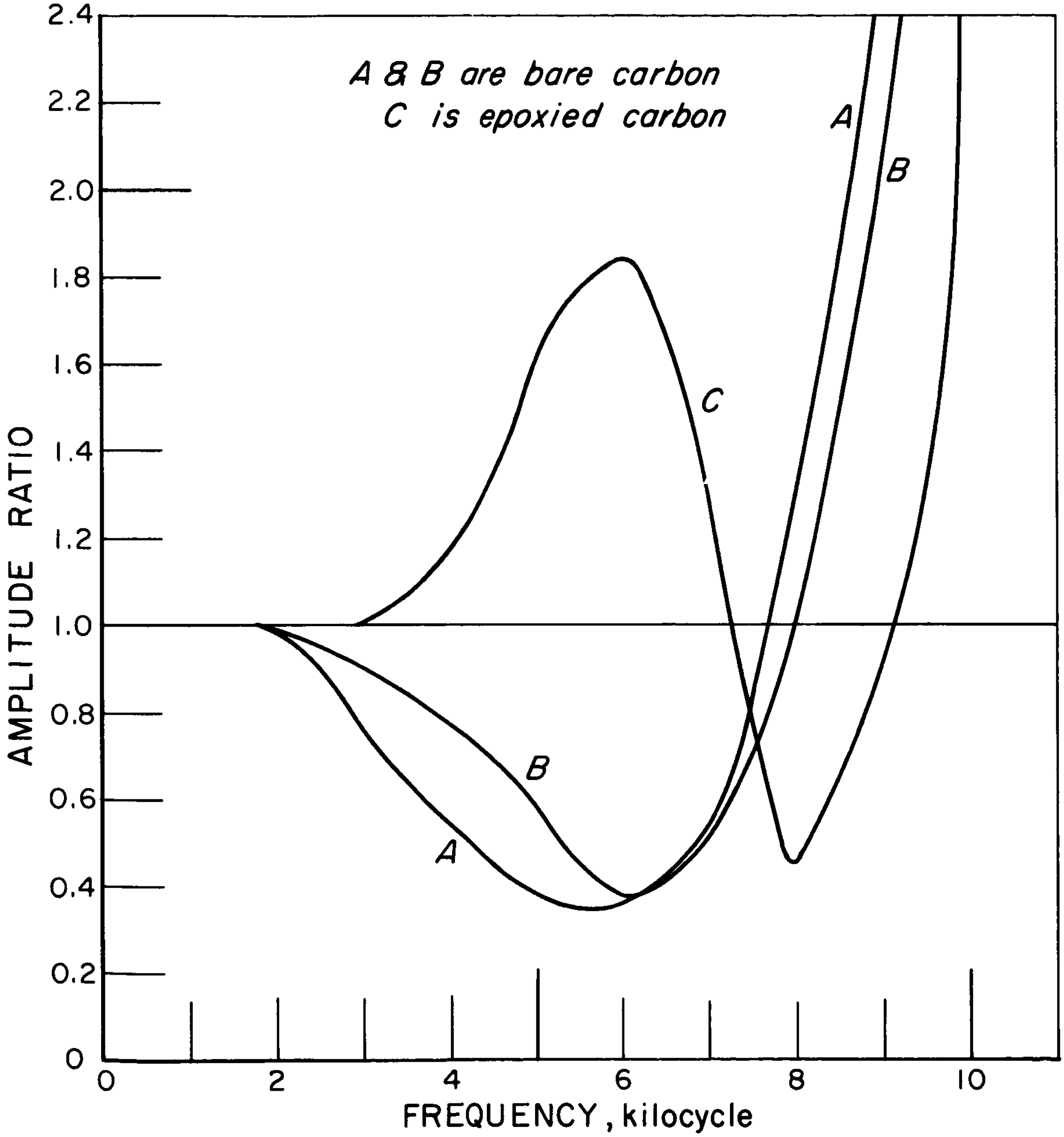

Figure 5. Frequency Response of Dynamic Carbon Resistor Pressure Transducer.

although this is not stated. Equation (4) accurately predicts the output of this transducer when the 300°K value of the pressure coefficient is used. The manufacturer terms his instrument a resistostrictive pressure transducer.

Inexpensive dynamic pressure transducers can be readily made from carbon radio resistors that are useful to 2 or 3 kHz and 350 kg/cm^2.

This work was initiated with the hope of finding a sensing element that would be insensitive to temperature while more sensitive to pressure than manganin. The high pressure sensitivity was found but the corresponding low temperature sensitivity was not. Thus this work cannot be called a success from the viewpoint of reducing pressure transducer temperature effects, but it has indicated the possible usefulness of ytterbium as a pressure transducer.

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