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STABLE PRESSURE TRANSDUCER

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ABSTRACT

In this report we describe the progress over the past year in the development of a capacitive pressure transducer which utilizes solid dielectric capacitors. The pronounced temperature dependence of the individual capacitors is circumvented by measuring the ratio of two capacitors having the same temperature dependence but markedly different pressure dependence. Of the materials measured to date, calcite crystals cut parallel and perpendicular to the crystalline axis most closely meet our criteria. An Invar sample holder has been fabricated which fits inside a commercial pressure vessel. The combination effectively cancels out adiabatic heating effects within the pressure vessel which will greatly simplify the thermostating requirements of the transducer.

INTRODUCTION

We are endeavoring to develop a pressure transducer that is capable of high precision and will possess long term stability. The device is intended for use as a transfer standard in the dissemination of the absolute pressure values determined by piston gauges in standards laboratories, but could be used in any application where pressure measurements of high accuracy are required. Current developments have centered on a capacitance gauge in which the capacitor is a disc of solid dielectric material with metallic electrodes deposited directly on its surfaces. With a capacitor made from a hard, preferably crystalline, material and subjected only to hydrostatic forces within a pressure vessel, one would not expect any physical deterioration of the capacitor with use.

For most materials which are of interest to us, the capacitance changes about 1% upon being subjected to a pressure of 140 MPa (20,000 psi), the maximum pressure we are currently considering. Using modern ac bridge techniques, three terminal capacitors, and accurate ratio transformers, capacitances can be readily measured to $1:10^7$, so that pressure resolution of about 700 Pa (0.1 psi) can be achieved. The major drawback with the device is that the capacitors are, in general, very temperature dependent. For example, with the widely studied material CaF_2, a change of 1 mK is equivalent to a pressure change of 7000 Pa (1 psi). The search for materials having a smaller temperature dependence while still maintaining a sufficiently large pressure dependence has produced some improvement in this regard, but it has not substantially altered the problem. BN and As_2S_3 are the most notable examples in that their ratios of temperature to pressure dependences are an

order of magnitude smaller than CaF_2 but they would still require millikelvin thermostating in order for the achievable pressure resolution to be realized.

Two separate schemes have evolved as ways of getting around the temperature problem; both use two transducer-type capacitors in opposite arms of the measurement bridge so that it is the ratio of the two capacitors that is being determined. The first approach, which is being pursued by James Miller of the Redstone Arsenal, uses two capacitors of the same material. One capacitor is placed in the pressure vessel and the other is outside at atmospheric pressure. In this arrangement, the temperature dependence of the pressure measurement is canceled to first order when the two capacitors are at the same temperature but this condition creates a difficulty since the two capacitors must be separated by a pressure-bearing wall. The second approach, the one being pursued in this laboratory, is to place both capacitors in the pressure vessel and to use two materials having the same temperature dependence but widely differing pressure dependences. This approach puts stringent requirements on the choice of materials since the temperature and pressure derivatives of the ratio measurement

$$R = C_1/C_2$$

can be written as

$$\frac{1}{R}\frac{dR}{dT} = \frac{1}{C_1}\frac{dC_1}{dT} - \frac{1}{C_2}\frac{dC_2}{dT}$$

and

$$\frac{1}{R}\frac{dR}{dp} = \frac{1}{C_1}\frac{dC_1}{dp} - \frac{1}{C_2}\frac{dC_2}{dp} .$$

The behavior of the pair, therefore, depends on the fractional dependences which are properties of the material and do not depend on the magnitude of the individual capacitances. When we began pursuing this course a year ago there were no known pairs of materials that met our requirements. We began our search by looking at several anisotropic materials since crystals of these materials cut parallel and perpendicular to the crystalline axis frequently

have widely different properties. Capacitors with intermediate values could then be obtained by cutting the crystal at the proper angle between the major axes. This approach did not produce the anticipated results but we did find that the parallel and perpendicular cuts of calcite ($CaCO_3$) as a pair came close to meeting our requirements. We have since made detailed measurements with the calcite pair against a piston gauge and found the results very encouraging. Accordingly, we have begun the development of a prototype device with the idea of using a calcite pair as the capacitor materials. In this report we describe the pressure vessel and sample holder that has been fabricated for the device. We discuss the considerations that went into the design of the device and the thermostating that will be required for its effective operation.

Thermal Equilibration and Thermostating Considerations

To establish the thermal requirements for a pressure transducer utilizing a pair of calcite capacitors we must first determine the pressure resolution that is to be sought. We are predicting that the uncertainty in the capacitance bridge which arises from the limited accuracy of the ratio transformer will be about $1{:}10^7$. With the calcite pair this corresponds to an uncertainty in the pressure determination of about 1700 Pa (0.25 psi). Since the bridge resolution will exceed this value, let us consider the thermal requirements we will need to achieve a pressure resolution of 700 Pa = 0.1 psi.

We can identify three ways that thermal effects can cause errors in the pressure determination. The first and most obvious is for the two capacitors to be at a different temperature because of a temperature gradient within the system. This error depends on the temperature dependence of the individual capacitances and for resolution of 700 Pa requires that the two capacitors be within 0.1 mK of each other.

The second effect comes from the fact that a pressure determination depends on a ratio measurement made at high pressure relative to one made at atmospheric pressur The temperature of the transducer at the two pressures must be within limits which, for the calcite pair, are determined mainly by the temperature dependence of the ratio measurement. The pressure resolution of 700 Pa requires that these two temperatures be within about 10 mK.

The third temperature effect arises because the pressure dependence of the ratio measurement is itself temperature dependent. As a result the pressure gauge must be used at the same temperature as it was during its calibration. The calcite pair is very good in this respect and the temperature of the device need only be maintained with a precision of about 75 mK in order to have precision in the pressure measurement to 700 Pa at pressures as large as 140 MPa.

Adiabatic Heating

The thermostating of the pressure transducer is complicated by the adiabatic heating effects which occur whenever the pressure is changed. When the pressure transducer is pressurized, all the material inside the pressure vessel (the samples, sample holder, and pressurizing oil) are under compressive stress and increase in temperature. The pressure vessel itself is placed under a net tensile stress when the pressure increases and as a result, it cools. The reverse occurs when the transducer is depressurized. Consequently, in thermostating the transducer one would need, in general, the capability of both heating and cooling the system. Also, with part of the system warming and others cooling, a rapidly-responding thermostat would probably prolong the period of equilibration.

We are attempting to get around this problem by trying to equate the heating and cooling that occur within the transducer during any pressure change, so that there will be little or no temperature change when thermal equilibrium is reestablished.

The amount of heating that occurs in a piece of material under compressive or tensile stress is given by

$$Q = T\bar{V}\bar{\beta}\Delta P,$$

where $\bar{\beta}$ is the average thermal expansion coefficient for the range of the pressure change, ΔP, and $\bar{V}$ is the average volume.

The cooling that occurs in the pressure vessel itself when (see Appendix, April 1975 report) it is internally pressurized is equal in magnitude to the amount of heating that would occur upon externally pressurizing a piece of steel

of the same composition as that of the vessel and having the same volume as the internal volume of the vessel. In other words, if we could completely fill the inside of the vessel with a steel plug and then pressurize the system using a negligibly small amount of oil, there would be no temperature change when equilibrium is reestablished.

For steels such as that of the pressure vessel, the thermal expansion coefficient is about 55 in units of 10^{-6}/K. The paraffin oil we have been using as a pressurizing fluid has a coefficient of about 750. Since we must have at least a small quantity of oil in the transducer, we need to offset its large coefficient by including in the pressure vessel a large amount of material with a thermal expansion coefficient smaller than steel. Fortunately, there is a suitable material, Invar, a high nickel steel, that has a thermal expansion coefficient of only 3.6 units. By limiting the volume of oil in the pressure vessel to about 7% of the total volume and filling the rest of available volume with Invar, we calculate (see April, 1975 report for detailed calculations) that temperature changes can be kept at a negligible level. The heating effects cannot be eliminated completely because the oil is highly compressible so that its thermal expansion is a marked function of the pressure. Even so, we predict that the temperature changes of the transducer for pressure changes in the range 0 to 140 MPa will not exceed 3 mK. This value is well below the 10 mK limit required for 700 Pa resolution with a calcite pair transducer.

We have checked out these ideas in a laboratory experiment in which the constant temperature bath of our experimental system was used as a crude calorimeter. The pressure vessel was alternately fitted with a steel or Invar plug which filled about 90% of the internal volume of the vessel. The relative temperature changes of the system with the different plugs were then observed for various pressure changes. The results indicate that our calculations were basically correct. There was an asymmetry in the results in

that there was a small net heating in the pressurizing-depressurizing cycles. This could be an experimental error but it may also be a ramification of the internal friction of the materials involved. We do not think that this net heating effect is large enough to be of concern. The results also indicate that the expansion coefficient of the oil may not be as pronounced a function of the pressure as originally thought. If this is true, it may be possible to keep the temperature changes in the system smaller than originally calculated.

In any case, the predicted maximum temperature change of 3 mK upon pressurizing or depressurizing the transducer is sufficiently below the 10 mK stability requirement stated in the previous section, that the temperature of the transducer will not require any adjustment for the capacitance measurements. All that is required is that the transducer be well insulated except for a weak thermal link to a thermostated heat sink so that its long term temperature stability is assured.

Pressure Vessel and Invar Sample Holder

For the prototype pressure transducer we are using a commercial (High Pressure Equipment Company) pressure vessel with a pressure rating of 20,000 psi (138 MPa). The vessel has a 1½ inch I.D. and a depth of 6 inches and comes with a pressure connection in the bottom of the vessel and one in the center of the one-piece threaded closure. We have modified the closure with three additional pressure connections to allow for the separate entry of the three electrical feed-throughs required for the capacitance measurements.

An Invar sample holder has been fabricated which attaches to the bottom of the pressure closure and effectively fills the entire volume of the pressure vessel. The sample holder, diagramed in Fig. 1, was made from six ¼" plates, which, when bolted together, form the cylindrical plug shown on the left. The central two pieces hold the low voltage electrode, which is common to both capacitors, and the capacitors themselves fit into recesses on the outside of these pieces. The next two pieces hold the high voltage electrodes, which make contact with the back of the capacitors. The three electrodes are made from small gold-deposited bellows, the same as used in our experimental system. The position of two outer electrodes can be adjusted to ensure sufficient force on the contacts for electrical continuity. The outer two plates simply fill out the volume.

The volume of oil which the pressure vessel will hold with this Invar sample holder in place is less than that estimated for minimizing adiabatic heating effects. This can easily be adjusted later by machining away part of the Invar holder or preferably substituting plates fabricated from steel or some other solid material with a thermal expansion coefficient larger than Invar.

The sample holder was designed so that the two capacitors are situated in axially symmetric positions within the pressure vessel. As a result the capacitors should always see the same temperature gradients and should be at the same temperature. Whether they will remain within the 0.1 mK temperature difference desired for the transducer with calcite capacitors will have to be determined.

The new pressure vessel and sample holder will be tested with capacitors in place as soon as new electrical feedthroughs can be made. The feedthroughs will be similar to those in our experimental device which use the sheathed thermocouple cables.

Capacitor Materials and Deposited Electrodes

Although we are proceeding with the development of a transducer using calcite capacitors, we have not ruled out the use of other capacitor combinations. In Fig. 2 are collected together values of the temperature and pressure dependence of the capacitance of numerous materials. What we require for a useful transducer are two materials that, on this plot, are displaced from each other by a considerable distance but along a vertical line. At this time, the only suitable combinations, in addition to the calcite pair, are the combinations of a calcite crystal with either KCl or KBr. The potential pressure resolution with a parallel-cut calcite capacitor in combination with KBr is more than three times that of the calcite pair. For the same resolution, therefore, the allowable temperature gradient between the two capacitors within the device could be three times greater with the calcite-KBr combination, but the other two thermal requirements will be similar for the two combinations.

We have attempted to test these combinations in the laboratory but got very anomalous behavior with all our potassium halide capacitors. The aluminum electrodes that had been evaporated onto these crystals looked good, but the adherence was very poor. The coatings could be removed completely with scotch tape. We think the erratic behavior we observed may be due to gaps developing between the crystal and the coating. Facilities to gold sputter electrodes onto samples may be available to us shortly and we will try this procedure with the alkali halides to determine if it will improve the adherence of the electrodes. It will also be worthwhile to test the gold sputtering with calcite

crystals, for there is some indication that the aluminum electrodes we have been using may be reacting with these crystals. We have detected a slight discoloration of the aluminum coatings on capacitors that have been made up for about six months. This could be due to oxidation of the outer aluminum surface, but it must be checked out.

It would be advantageous to find a material with a negative pressure coefficient and a temperature coefficient matching either BN or As_2S_3. A combination of this type with the smaller temperature dependence of the individual capacitors would alleviate the requirement of close thermal equilibration between the two capacitors that is so stringent with the calcite pair or calcite-potassium halide combination. Since the negative pressure coefficient is the most typical behavior, the chances are good that such a material exists. Accordingly, we are continuing to look at other materials that are appropriate as transducer capacitors.

Automatic Capacitance Bridge

The assembly of the limited-range automatic capacitance bridge intended for use with the pressure transducer is nearly complete. All the logic circuits have been built as originally designed and are operational. The bridge has been tested to some extent on the bench top using standard capacitors and will be check out shortly with the actual transducer.

One additional stage of amplification with additional filtering is going to be added, which will also include the automatic phase-balancing circuit. A small preamplifier has been built which will go directly on the pressure transducer, thereby reducing the effects of the capacitance in the leads to the bridge. The bridge circuits are completely documented in a way which should facilitate their reproduction. Complete blue prints have been maintained and, in addition, wiring locations have been put on punch cards. Print-outs of different sorting sequences of the cards have been valuable aids in both the original wiring and in trouble-shooting.

Figure Captions

Fig. 1 Invar sample holder shown assembled on the left and disassembled to the right. The sample holder is 1½ inches in diameter and and 6 inches long and fits snugly in the cavity of a commercial pressure vessel. The dielectric capacitors fit into recesses of the inner two plates and electrical connections are made using small bellows as spring contacts.

Fig. 2 The pressure dependence of the capacitance of various materials as a function of their temperature dependence. The temperature dependence is the atmospheric pressure value and the pressure dependence was determined at 35°C. The lines shown join the values measured parallel and perpendicular to the crystalline axis of anisotropic materials and represent temperature dependences that could be realized for crystals cut at intermediate angles.