SOLID STATE PHYSICS PROGRAM

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CARL ANDEEN, JOHN FONTANELLA, AND DONALD SCHUELE

CASE WESTERN RESERVE UNIVERSITY

UNIVERSITY CIRCLE   CLEVELAND, OHIO 44106

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A CAPACITIVE GAGE FOR THE ACCURATE MEASUREMENT

OF HIGH PRESSURES

Carl Andeen, John Fontanella, and Donald Schuele

Case Western Reserve University, Cleveland, Ohio 44106

A highly accurate and precise pressure gage which utilizes the $35^{\circ}C$, 2000 Hz dielectric constant of an ionic crystal, CaF_2, is presented. The accuracy, stability, and convenience of such a gage are discussed comparing it to dead weight and manganin wire resistance gages. From a calibration at the National Bureau of Standards, the defining equation was found to be:

$$C/C_o = 1 - AP + BP^2$$

where C_o is the zero pressure capacitance, C is the capacitance at absolute pressure P, and the characteristic constants are:

$$A = 2.6063 \times 10^{-7}/(psi)$$

and

$$B = 1.302 \times 10^{-13}/(psi)^2.$$

INTRODUCTION

In order to measure accurate values of pressure from atmospheric to 35,000 psi., either a dead weight gage or a manganin wire resistance gage is usually used.

The former yield satisfactory results up to and slightly beyond the 0.01% level, but are expensive, both to purchase and operate, bulky, and time consuming.

Manganin wire resistance gages, on the other hand, have none of these drawbacks, but when requirements approach accuracies of 0.01% a host of other problems arise. It is well known that temperature and pressure cycling affect the pressure coefficient of resistance.[1,2] The above types of cycling also affect the position of the resistance-temperature maximum, which, of course, affects the temperature coefficient of resistance.[3] A method has been adopted[4] in an attempt to compensate for the temperature effects, but, due to the change of the position of the resistance-temperature maximum with pressure[5,6] this technique is not really valid. In addition to ambiguities arising within a given system, intercomparison between different gages is difficult because the pressure coefficient of resistance is dependent upon gage construction, and size, chemical composition,

and metallurgical condition of the wire. This, then, necessitates calibration of individual gages.

In addition, there are problems associated with intrinsic time constants[3] and the Bridgman "water kick."[7] These will be discussed later.

Even a five point calibration[8] coupled with careful control of internal variables gives accuracies only on the order of 0.03% for the linear term and 1.2% for the quadratic contribution.

THE GAGE

In this gage the change in the capacitance of an
ionic crystal with pressure is used as the pressure
sensor.   In recent years, capacitance measuring tech-
niques have advanced to the point where it is common
practice to measure such capacitance changes very
accurately. Measurements can be done using a trans-
former ratio arm bridge capable of sensing three-ter-
minal capacitances. The only other equipment necessary
is the associated null detection system, pressure
vessel, and reference capacitor. The prototype system
is as follows.

The ionic crystal chosen was CaF_2 because of its
low loss, hardness, insolubility, non-hygroscopic
nature, and availability. The samples studied here
are among those for which the low frequency dielectric
constants were reported in a previous article.[9] Aluminum
films approximately 2000 Å thick were evaporated onto
the surfaces to serve as electrodes. This was done in
three steps.

First, the guard electrode was evaporated using
a carefully machined mask that covered the center portion
of the crystal. In addition, the rim of the crystal was
protected by means of a ring that fits around the outside

edge of the crystal. Next, the guarded center electrode
was evaporated using a washer type mask whose inside
diameter was slightly smaller than the inside diameter
of the resultant guard electrode. The centering of the
sample was done under a microscope using three indep-
endent screw type mechanisms. The sample was spring
loaded to allow for isotropic thermal expansion. In
this manner it was possible to achieve gaps between the
guard and guarded electrodes which were less than twelve
microns wide. Finally, a full electrode was plated on
the other face of the crystal. All evaporations were
done with the sample at $150^{\circ}C$ after having been out-
gassed at that temperature for an hour.

The samples were 25 mm in diameter and 1.6 mm
thick. The guarded electrode was 14 mm in diameter,
and, thus, the condition that the fringing field
effects be eliminated:

$$\frac{\text{Guard Ring Width}}{\text{Sample Thickness}} > 3 \qquad (1)$$

is more than satisfied. It should be noted that the only
other consideration that needs to be made with respect to
geometry is to make the gap between the guard and guarded
electrodes as small as possible as is done above.

The fact that CaF_2 has a temperature coefficient of

capacitance on the order of 250 ppm per Kelvin degree at room temperature[10] necessitates temperature control. If the reference capacitor is thermally sunk to the pressure vessel, then control of $0.01 \, C^o$ is adequate. This can easily be done using a thermostatic system with a glass-encapsulated thermistor as the sensing element.

The pressure vessel and sample holder were of standard design. In this system, the reference capacitor was not thermally connected to the pressure bomb, and, thus, greater care was taken with temperature control.

The transformer ratio arm bridge used for the measurements was constructed in our lab. It was operated at 2005 Hz, and the ratios read from this instrument were accurate to approximately one part in ten million under the gage operating conditions.

CALIBRATION

Eight samples, which were obtained from the Harshaw Chemical Co., with capacitances on the order of six picofarads, were calibrated over the pressure range 0-35,000 psi at the National Bureau of Standards using a dead weight gage accurate to 60 ppm. All of the data was used to generate a fit to the following second order equation by the method of least squares:

$$C/C_o = 1 - AP + BP^2 \qquad (2)$$

and the coefficients were found to be:

$$A = 2.6063 \times 10^{-7}/(psi) \qquad (3a)$$

and

$$B = 1.302 \times 10^{-13}/(psi)^2, \qquad (3b)$$

and P is the absolute pressure. A rather striking observation is that <u>every data point for all eight samples agreed to within 2 psi of this best fit curve</u>.

In order to graphically show the fit between the actual data and the least squares curve, the difference between the capacitance change obtained from the least squares fit and the actual data is plotted versus pressure in Figures 1 and 2 for samples 1 and 2. This difference in capacitance change is plotted as a pressure differential. All of the data for sample 1 is

found to be within 0.75 psi of the pressure. Above 2000 psi, the data agrees to within 0.01% of reading.

In addition, Figure 2 shows the pressure differentials when first a quadratic fit to sample 2's data is used as a reference, and then a cubic fit to its data is used. The former shows a maximum spread of 0.5 psi while the latter shows even better agreement. The results of a quadratic fit to each sample's data are listed in Table I. An analysis of the calibration equation for each sample shows that all eight expressions predict pressures that are within 0.01% of the master equation obtained from a quadratic fit to all of the data. The consistency of the results is all the more striking when it is realized that some of samples 1-8 had been pressure-cycled and others heat-treated at 280°C for four hours at 10^{-5} Torr prior to calibration. The third order effect was studied further by fitting all of the data to a cubic equation. The first and second order terms changed very slightly from A and B of the quadratic fit with a resultant third order coefficient on the order of $6 \times 10^{-20}/(psi)^3$. It is seen, then, that to 0.01% accuracy of using CaF_2 as a pressure gage, third order effects need not be considered at the 35,000 psi level.

Finally, all of the data was fed into the computer in the reverse order, and the functional pressure gage equation was found to be:

$$P = \alpha(C_o - C)/C_o + \beta(C_o - C)^2/C_o^2 \qquad (4)$$

where:

$$\alpha = 3.8358 \times 10^6 \text{ psi} \qquad (5a)$$

and

$$\beta = 7.725 \times 10^6 \text{ psi.} \qquad (5b)$$

COMPARISON WITH MANGANIN WIRE

A CaF_2 sample and a length of manganin wire were
placed in the same pressure system, and the changes in
capacitance and resistance with pressure monitored. The
resultant curve of capacitance versus resistance was
fitted to an equation which had terms to second order in
the resistance. The difference between the experimental
data and the fitted equation is plotted in Figure 3.

The first effect to be noted is the anomalous
behavior at low pressures. This was attributed to the
Bridgman "water kick."[7] This is due to the leakage
resistance of the manganin wire to ground through the
pressure medium. The loss mechanism is water contamination
in the pressure fluid. The effect is seen to disappear
at around 10,000 psi. An independent investigation was
made in which the resistance of the fluid as a function
of pressure was observed. The resistance showed an in-
crease from a finite value at small pressures to essen-
tially infinity at nominally the same pressure at which
the capacitive aberration disappeared. Thus, it was
effects associated with the wire and not the CaF_2 that
caused the low pressure anomaly. It was found that this
effect can be greatly reduced by using high quality oils,

such as the Spinesstic 38 series, but even the finest oils contain some water.

Also evident from Figure 3 is a hysteresis loop. This was shown to be caused by an intrinsic time constant associated with the manganin wire by the following experiment. A sample and manganin wire were pumped up to three kilobars, and the capacitance and resistance monitored for several hours. Figure 4a defines the quantity δ, which was essentially a measure of the deviation of the system from ideal behavior. This is plotted against time in Figure 4b. The slow asymptotic approach to equilibrium is shown. The sample was left at that pressure while the manganin wire was reduced to atmospheric pressure. After several days, the manganin wire was pumped up to three kilobars, and the sample hooked into the system. Again, the same sort of relaxation occurred, thus showing that the manganin wire was responsible for the hysteresis.

Thus, the dielectric constant of ionic crystals appears to be a much more well-behaved physical property than the resistivity of manganin wire. This may be in part due to the fact that single crystals tend to propagate almost pure hydrostatic stresses as opposed to the ill-defined nature of the stresses felt in wires of alloyed material. This, then, has application as an

accurate, stable, and easy-to-use pressure gage. It
may be that the range of pressures definable by such
a gage is quite high. Investigations are underway to
determine the validity of the necessary extrapolations.
This gage is currently being used in the determination
of the pressure derivatives of the static dielectric
constants of the other alkaline earth fluorides and
some alkali halides from $4.2^{O}K$ to room temperature.

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Figure 1. The difference between the capacitance change obtained from a quadratic least squares fit to all the data and the actual data (given as a pressure differential) plotted versus pressure for sample 1.
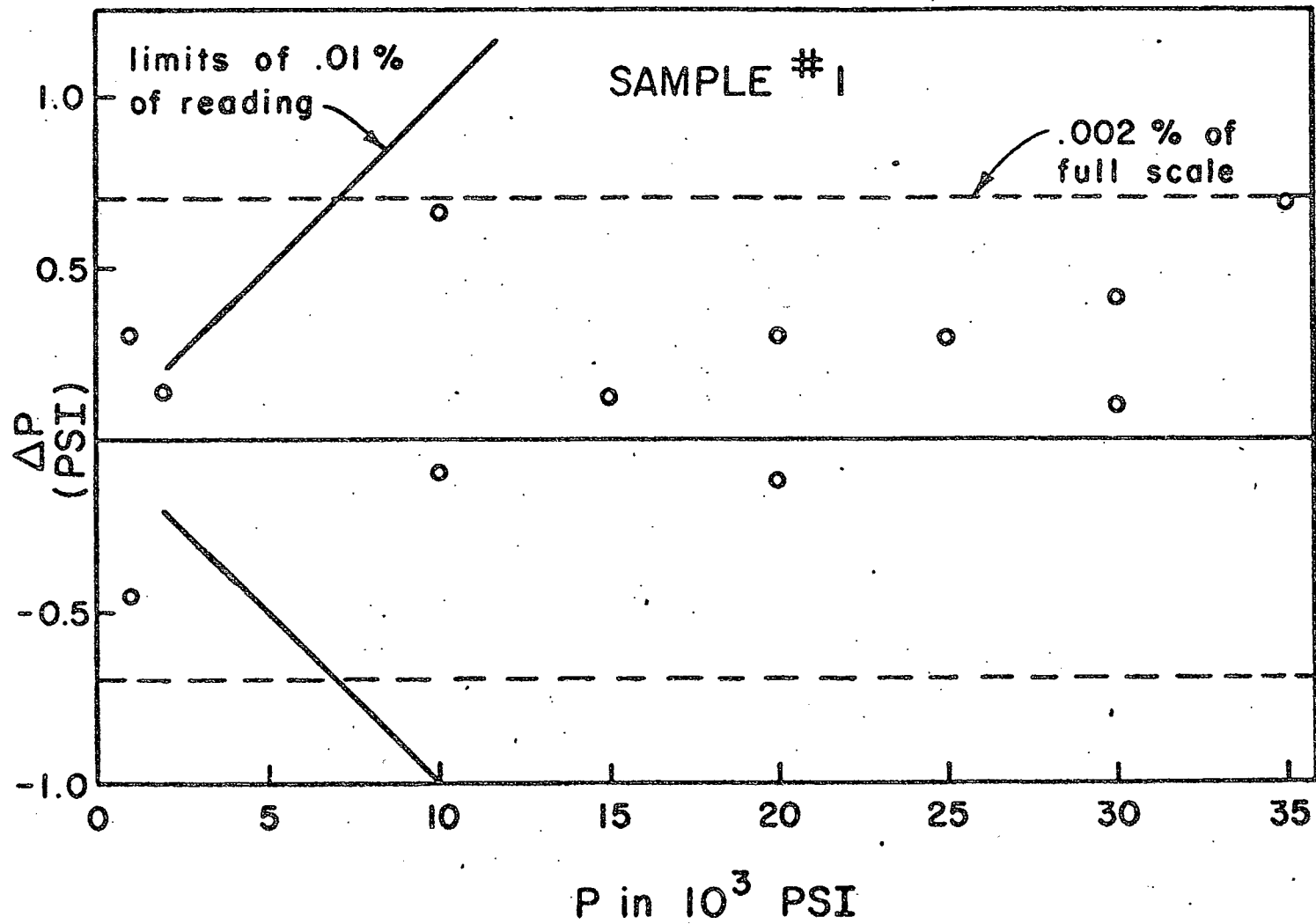
Figure 2. The difference between the capacitance change obtained from (a) a quadratic least squares fit to all the data, (b) a quadratic fit to sample 2's data, and (c) a cubic fit to sample 2's data, and the data plotted versus pressure for sample 2. Again, all capacitance changes are plotted as pressure differentials.
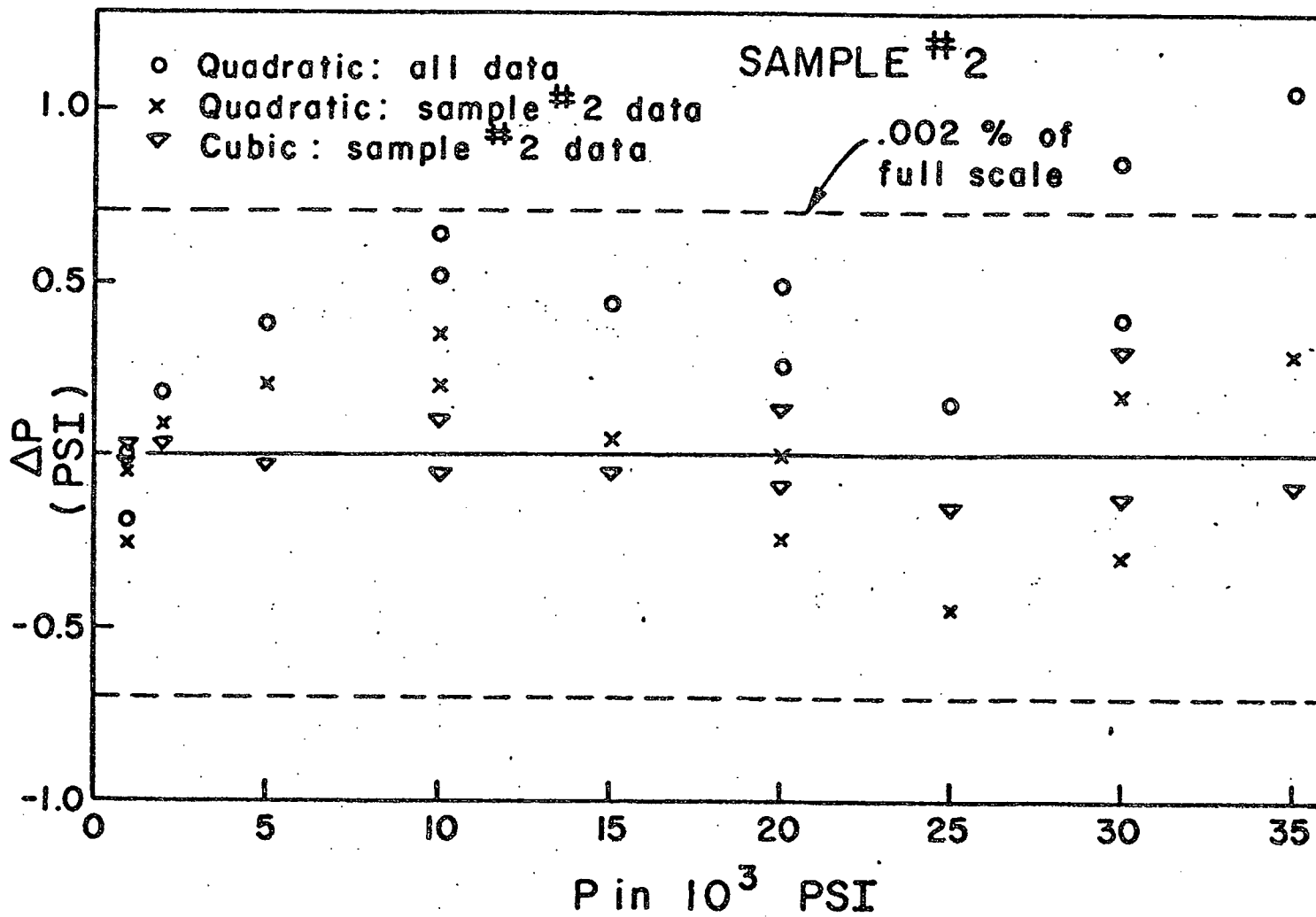
SAMPLE #2

Legend:
- ○ Quadratic: all data
- × Quadratic: sample #2 data
- ▽ Cubic: sample #2 data

.002 % of full scale

ΔP (PSI)

P in 10^3 PSI

Figure 3. Comparison between manganin wire and CaF_2.

The C^0 scale is the residual capacitance

after the linear and quadratic contributions

have been removed. Two divisions are

approximately equivalent to 10^{-5} picofarads

on that scale. ΔR is the change in resis-

tance of a length of manganin wire in ohms.
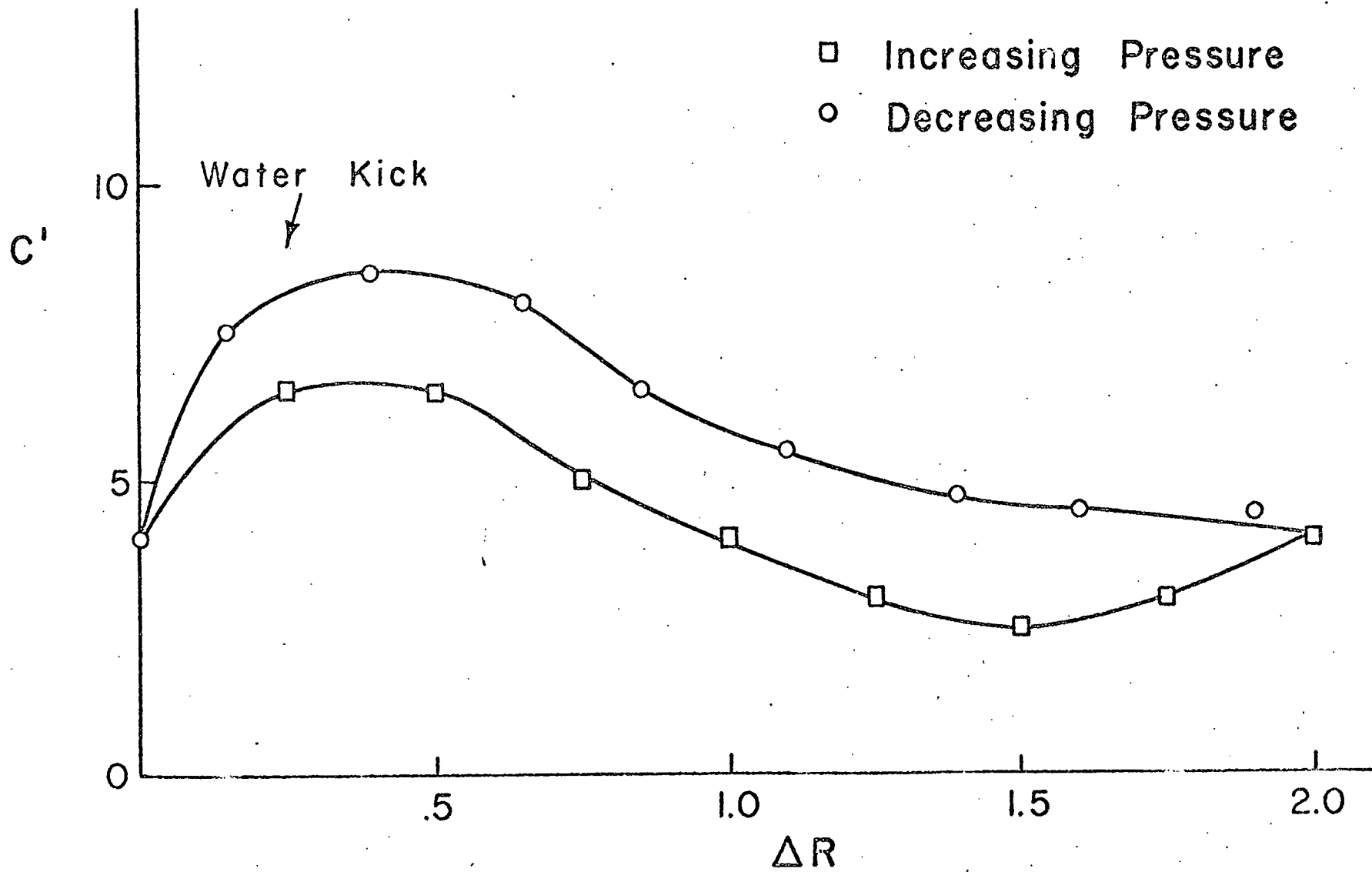
One ohm is equivalent to 1.4 kilobars.

Figure 4.   (a)   Definition of the quantity δ where C

is the capacitance of a CaF_2 sample, and

ΔR is the change in resistance of a length

of manganin wire.   (b) δ is plotted against

time for a system of manganin wire.   Eight

divisions of δ are approximately equal to

one milliohm, and the time is in hours.

(c) δ plotted against time, in this case,

after the manganin wire had been allowed to

relax to room pressure for several days.

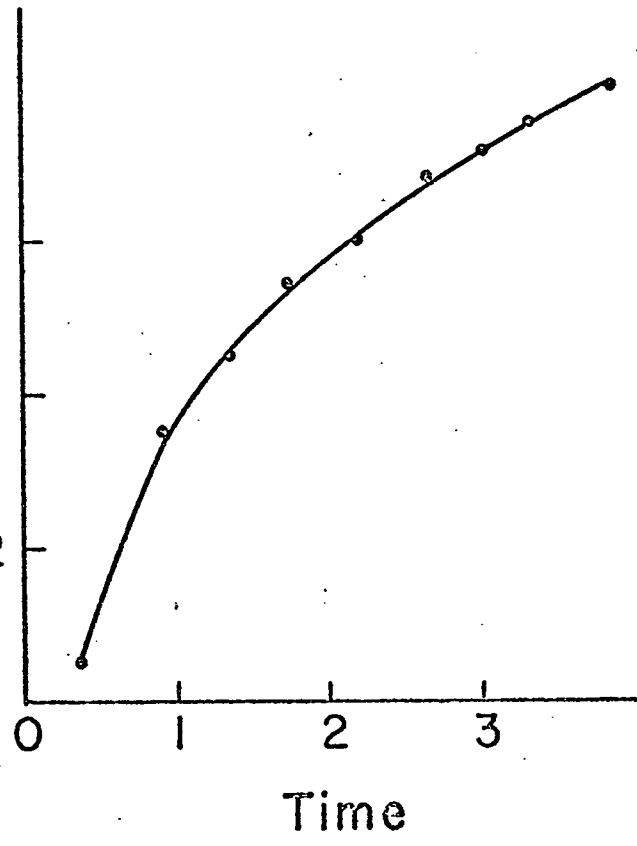The units are the same as for Figure 4b.
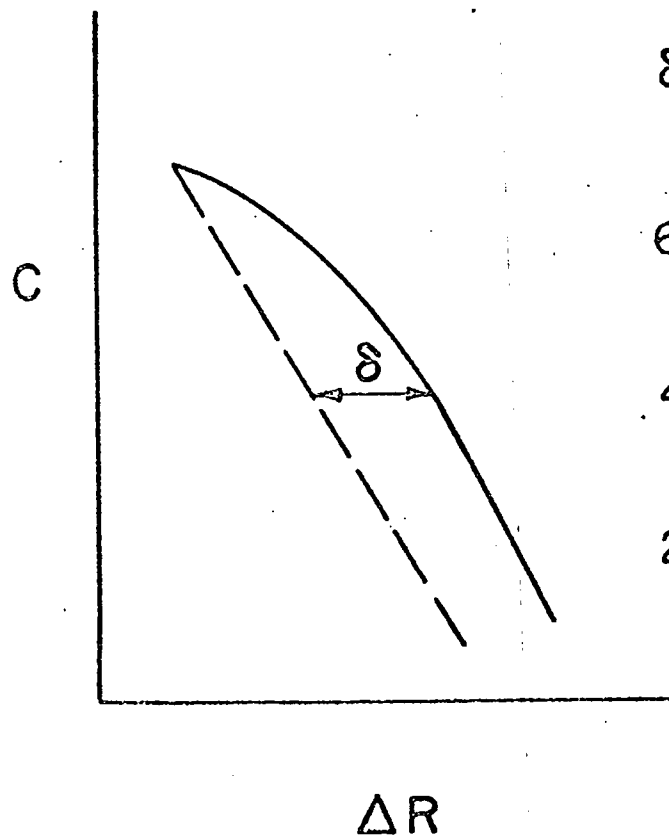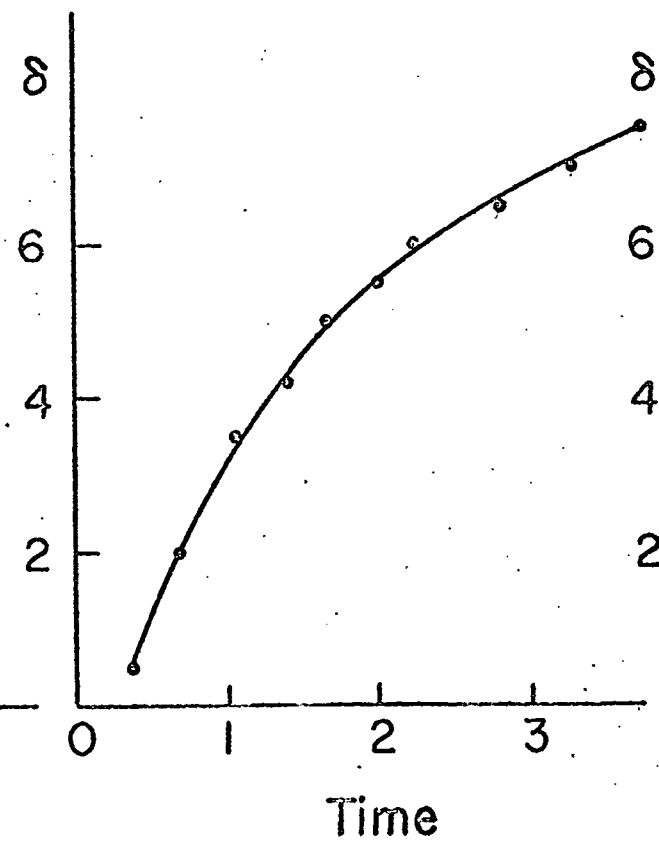
(a) (b) (c)

Table I. The first and second order coefficients in the equation $C/C_o = 1 - AP + BP^2$ where A is in units of $10^{-7}/(\text{psi})$ and B is in units of $10^{-13}/(\text{psi})^2$.

Sample	A	B
1	2.60617	1.298
2	2.60624	1.300
3	2.60614	1.298
4	2.60642	1.322
5.	2.60613	1.303
6	2.60631	1.303
7	2.60644	1.306
8	2.60672	1.318

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