AIP | Review of Scientific Instruments

Compressibility Measurement at High Pressures by the Inductance Coil Method

A. A. Giardini, E. H. Poindexter, G. A. Samara, and J. D. Barnett

Citation: Review of Scientific Instruments **36**, 1742 (1965); doi: 10.1063/1.1719454
View online: http://dx.doi.org/10.1063/1.1719454
View Table of Contents: http://scitation.aip.org/content/aip/journal/rsi/36/12?ver=pdfcov
Published by the AIP Publishing

at the plates after the sweep is applied we have

$$y_a = (ect_d V_{y0}/bmv_{x0})(1+[a[t_d-2(t_i-t_c)]/2]). \quad (13)$$

It can be shown that for $t_d<2/a$, Eq. (13) is to be used for calculating the time resolution. Thus, for a linear sweep the time resolution is from Eq. (13)

$$\Delta t = \Delta y_p/ay_{a0} = D\Delta y_p/aV_{y0}. \quad (14)$$

If the voltage applied to the image converter is 5 kV and $d=1$ cm, t_d, from Eq. (6), is 0.24 nsec. At 5 kV the deflection factor is around 125 V/cm. Thus, for a deflection voltage of 400 V and $\Delta y_p=0.3$ cm the time resolution for a sweep voltage in the form of a step is from Eq. (8) equal to 5.7×10^{-12} sec. For a linear sweep with sweep speed aV_{y0} equal to 600 V/nsec we get, from Eq. (14), $\Delta t=0.06$ nsec.

Equations (10) and (14) give the time resolution of the image converter for the case in which the electron beam formed by the photoelectrons has a very small width at the screen of the image converter. As a result of the finite width of the photocathode, however, the beam has a finite width when it strikes the screen face. In order to determine the effect on time resolution introduced by this finite width of the electron beam, we proceed as follows. Again, we assume that transit time spread is zero so that Eqs. (10) and (14) apply to a beam of very small width. In the image converter tube here employed, the width of the electron beam at the screen face is equal to the width of the phosphor. Now a time Δt, given either by Eqs. (10) or (14), is required for the inner edge of the beam to sweep over the phosphor. However, just as the inner edge of the beam goes past the phosphor the outer edge strikes the phosphor and again takes a time Δt to sweep over it. Thus, the whole beam takes a time $2\Delta t$ to go past the phosphor and Eqs. (10) and (14) must be multiplied by a factor of 2 to correct for the finite width of the beam.

ACKNOWLEDGMENT

The author would like to thank W. L. Shoemaker for his assistance with the experiments.

THE REVIEW OF SCIENTIFIC INSTRUMENTS VOLUME 36, NUMBER 12 DECEMBER 1965

Compressibility Measurement at High Pressures by the Inductance Coil Method

A. A. GIARDINI,* E. H. POINDEXTER, G. A. SAMARA,† AND J. D. BARNETT‡

U. S. Army Electronics Laboratories, Fort Monmouth, New Jersey

(Received 28 May 1965)

Volume compression of isotropic solids at high pressure may be determined by measurement of the inductance of a coil wound tightly around the sample. For isotropic compression of a coil, inductance is exactly proportional to the cube root of coil volume. Cores of tungsten, strontium titanate, cadmium sulfide, and sodium chloride were compressed to 55 kbar to test coil performance. The reproducibility of compression values varied from ±5 to $\pm15\%$. The observed compressions agree closely with other published results on compression and elastic constants. For nonconductive samples, no corrections need be applied to the volumetric data.

INTRODUCTION

POLYMORPHIC transitions in solids at high pressures may be readily detected by monitoring of the inductance of a small coil wound on the sample core.[1] The coil technique greatly reduces problems arising from mechanical friction by virtue of its immediate conversion of volumetric information to an electrical signal at the site of the sample itself. The coil is one of the few methods available for study of volume changes in the case of quasi-hydrostatic solid environments. For the coil *per se*, there is no inherent upper limit to the maximum usable pressure. The coil has the advantage that the mathematical relation between the volumetric input and electrical output is simple and exact: $L \propto V^{\frac{1}{3}}$. It has a further significant advantage in that essentially no corrections need be applied to the measurements in a properly arranged sample environment. This is in marked contrast to mechanical volumetric techniques, where corrections arising from distortion of the apparatus may actually exceed the quantity to be measured.

The coil should be useful for a study of ordinary bulk compressibility as well as for sudden volume changes. Initial attempts supported this possibility,[2] but were none too promising with regard to the precision of the results. We

* Present address: Department of Geology, University of Georgia, Athens, Georgia.

† Present address: Sandia Corporation, Albuquerque, New Mexico.

‡ Permanent address: Department of Physics, Brigham Young University, Provo, Utah.

[1] A. A. Giardini, E. H. Poindexter, and G. A. Samara, Rev. Sci. Instr. 35, 713 (1964).

[2] A. A. Giardini, E. H. Poindexter, and G. A. Samara, Trans. Am. Soc. Mech. Engrs. Ser. D, 86, 736 (1964).

have since then significantly reduced a number of sources of error. Under carefully controlled conditions, the coil now appears to be a useful tool for determination of simple bulk volume compressions.

We discuss the more important experimental modifications in the use of the coil and conclude with several examples representing a wide range of compressibility. The purpose of this paper is to illustrate the operation of the coil in this application; the observed compression data are not intended to be new standard values.

The theory of the coil method has been discussed in detail previously,[1] and is not repeated here.

EXPERIMENTAL IMPROVEMENTS

The measurement technique is, of course, exceedingly simple in principle, as is the basic design of the sample container. A coil of about twenty turns of enameled copper wire is wound on the sample core; an appropriate jacket of pyrophyllite or silver chloride or other material is slipped over, and the whole is contained in a sample block of pyrophyllite. Representative photographs have been shown previously.[1]

It was observed that the performance of the coil in studies of both bulk compressions and polymorphic transitions was often quantitatively reproducible to better than 2% of the quantity $(\Delta V/V_0)$ measured, provided that sample, coil, and environment were exactly duplicated. Thus the errors are for the most part systematic for the experimental procedures we have used. Listed below are the five major operational complications and sources of error which we have attempted to rectify.

(1) Conformity or "fit" of coil to sample core may vary over the course of a pressure run.

(2) Distortion of the sample core may change the inductance of the coil quite apart from any net volume change.

(3) Distortion or displacement of the input wire leads connecting coil to bridge may change the lead inductance in an unknown fashion.

(4) The range of electrical parameters for practical coils on samples of 0.25 to 1.0 cm^3 is near the limit of precise inductance determination on even very good bridges.

(5) The electrical skin effect complicates interpretation of volumetric inductance data for conductive samples.

The first two—coil conformity and sample distortion—are the prime sources of error and also the most difficult to alleviate.

The theoretical relation $L \propto V^{\frac{1}{3}}$ is, of course, satisfied in application only if the coil is coupled tightly and consistently to the sample core throughout the course of a compression experiment. The coil must not be free to slip along the surface of the core, lest the turns be spread apart or pressed together without regard to the actual dimensional change of the sample. Moreover, the "closeness of fit" must remain constant; the change in effective coil diameter must be forced to follow the diameter change of the sample core.

The coupling between coil and core may be improved by threading the core to receive the coil wire directly. Earlier, we had either wound the coil on a smooth sample with adjacent turns in contact, or else on a thin threaded sleeve of pyrophyllite or AgCl enclosing the core. The threading of the sample itself prevents any slippage of coil wires along the surface of the core. It also essentially obviates any correction *a priori* for the mechanical filling factor arising from inclusion of foreign materials between coil and and core. In actual practice, the improvement afforded is significant, although not as striking as might be expected. This method, nonetheless, was used exclusively for all compressibility measurements.

Another aspect of the conformity problem, however, is not solved by threading. The other problem arises from the tightness of the winding itself, and is by no means a trivial consideration. It is not easy to wind soft copper wire with sufficient tension to straighten out all minor bends and force it to grip the threaded core in tight and perfect contact. A variation of a few thousandths of a millimeter in the effective intimacy of fit can introduce considerable error in the case of relatively incompressible cores. It was observed that if the surrounding jacket were markedly "softer" than the sample core, the resulting apparent volume changes were too high. This may well occur because the copper wire is essentially without strength, and is simply pushed in more tightly against the sample. Conversely, a jacket too "hard" might be expected to shield the coil and core from feeling full pressure and result in compressions which are too small.

In the present tests, we selected jackets of pyrophyllite itself for relatively incompressible samples, and silver chloride for more compressible ones. This is, of course, not an ideal solution. Direct deposition of the coil on the core, by plating or sputtering, might be a workable improvement. Alternatively, the encasing jacket might be made of sample material itself, and threaded internally to screw snugly over the coil and core. For certain samples an excellent solution would seem to be casting the coil directly in molten sample material. We have not yet tried any of these ideas.

Sample distortion was the second major obstacle. Any change in the coil length/diameter ratio introduces changes in inductance. Certain types of barrel, pincushion, and helical distortion of the core also alter the inductance. These inductance changes are independent of any net volume compressions.

We have reduced distortion by a number of modifications.[3] Preformed gaskets are machined on the pyrophyllite sample container to reduce large scale extrusion of pyrophyllite during gasket formation. Metal flow seals were used to further reduce this extrusion. The sample core volume was but a small portion of interanvil volume, about 1 or 2%, and occupied the homogeneous central part of the working space. The use of appropriate jacket or sleeve materials directly around the sample core also reduces distortion. In this case, though, the simple expedient of a "soft" jacket is not always of value, because of the increased shear strength of usual soft solids (like AgCl) under high pressures. Rather, we attempted to strike a proper rheological balance among components around the sample core—the criterion, of course, is minimum distortion.

Resultant distortions were unmeasureable (less than 0.001 cm, or roughly 0.1% of sample dimensions) on strong materials, but could amount to as much as 2% on weaker samples.

The major proportion of processes affecting both coil conformity and sample distortion appear to take place during the initial stages of an experiment—at pressures below 5 kbar. The inductance of the coil may increase sharply in the first stages of pressure application, or it may decrease. The sign of the change was not well correlated with known sample and experimental conditions, although softer samples suffered larger initial changes. We can offer no detailed explanation of the mechanism of these irregularities. It was observed, however, that preseasoning the sample block assembly up to 5 kbar would often ameliorate the coil behavior. This preseasoning presumably effects the major part of the distortion to be encountered on a given compression experiment and also eliminates voids between coil wire and core. The coil and core, even though perhaps no longer the original perfect cylindrical shape, then maintain the same shape and mutual coupling throughout the experiment. The actual geometrical form of the sample is of no concern, provided that it does not change during the course of the compression.

Distortion problems would be entirely eliminated in a truly fluid medium. It is, of course, necessary to confine the the coil wire by a rigid sealed jacket to force conformity to the core. We have tried to use the coil method inside a high pressure fluid chamber,[4] but in these initial attempts the seal ruptured and the coil failed to follow the core.

A major source of error in early experiments was the use of parallel input leads to connect the coil inside the pyrophyllite sample container to the coaxial cable running to the bridge. Distortion and displacement of these leads during pressure application introduced serious and erratic scatter in inductance readings. The magnitude of the scatter was sometimes comparable to the over-all compression of NaCl at 50 kbar. This difficulty has been largely eliminated by use of a coaxial connecting lead. Shielded thermocouple lead with magnesia insulation and stainless steel jacket proved satisfactory. The inductance of the coaxial lead increases only about 0.003 μH as it is flattened under pressure to 65 kbar. In relative terms, this represents about one-third of the expected inductance change for a 1 μH coil wound on a diamond core subjected to the same pressure. The coaxial lead introduces a systematic, smoothly monotonic and fairly reproducible source of error; the total scatter referred to compressions of diamond is only about $\pm 10\%$. For substances such as the alkali halides, the coaxial scatter is entirely inconsequential.

The range of practical coil and core sizes results in coil inductance and resistance near the useful limit of most bridges. To obtain the needed precision, it is necessary to read the inductance to at least four figures. With a coil of 1 μH this placed the measurement near the extreme sensitivity of our bridge.[5] The bridge also limits the maximum resistance of the coil, which prevents use of extra fine wire for improved conformity. In the most sensitive range, many bridges require consideration of residual inductances within the bridge for highest accuracy. In the present work no attempt was made to refine the electrical measurements. The errors were thought to be negligible in comparison with others. Further, the coil technique requires only relative inductance over the course of a compression, not absolute values.

Interpretation of the inductance to yield core volume is straightforward for substances with high resistivities. The particular bridge which we have used operated effectively at 1000 cps. For resistivities above 100 $\mu\Omega$ cm, correction for skin effect is negligible. For more highly conductive materials, a correction must be applied. At 1000 cps, with samples of about 0.2 cm^3, the coil should be considered readily useful for materials of resistivities to 5 $\mu\Omega$ cm. At about 2 $\mu\Omega$ cm the skin-depth correction would be about equal to the inductance change for a substance whose compression ($\Delta V/V_0$) is about 0.015 at 65 kbar (i.e., about the compression of tungsten). The skin-effect correction is, of course, independently measurable. It is not closely analogous to the corrections arising from apparatus deformation in mechanical volumetric indicators.

The skin-depth correction may be roughly deduced from published curves for the complex impedance of coils with conductive cores.[6] Unfortunately, many such curves are computed for infinitely long cylindrical cores, spheres, or hollow tubular cores; we know of none for short solid cylinders. The evaluation of the correction for a given coil

[3] A. A. Giardini and G. A. Samara, Am. Soc. Mech. Engrs. Paper No. 64-WA/PT-10.

[4] We thank Dr. T. E. Davidson at Watervliet Arsenal for his cooperation and use of his equipment.

[5] General Radio Company No. 1632-A.

[6] *Nondestructive Testing Handbook*, edited by R. C. McMasters (The Ronald Press Company, New York, 1959), pp. 36.1-39.19.

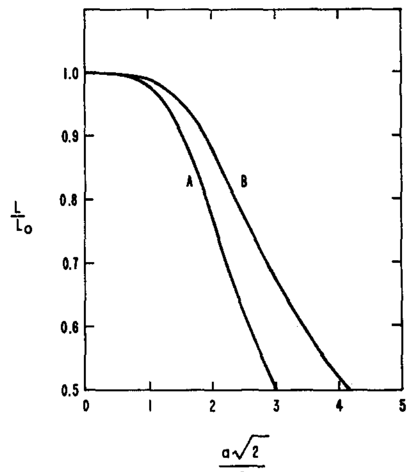

FIG. 1. The relative inductance of a coil tightly wound on an electrically conductive core. Curve A was calculated from formulas by Scott (Ref. 7); curve B was empirically derived with appropriate sample cores. Coil radius is a, and electrical skin depth is s.

and core combination is readily accomplished empirically. In our case, cores of copper, brass, and titanium of identical size were inserted in a representative coil. The complex impedance of the coil was then obtained as a function of frequency for each core.

The inductance of the coil on an infinite core has been computed by Scott.[7] Figure 1 shows Scott's computed results and our observed results for cylindrical cores with length/diameter ratio unity. As might be expected, the infinite core effects a greater reduction in coil inductance than the finite one. The empirical curve was used in our analysis of conductive samples.

EXAMPLES AND DISCUSSION

Several substances of different compressibilities were chosen to demonstrate the coil operation: tungsten, strontium titanate, cadmium sulfide, and sodium chloride. The compressibilities of these samples cover a range of ten to one. We are concerned here with the measurements to illustrate the application of the coil; no physical interpretation is offered.

Data were appraised by noting the final condition of the sample core and the nature of the observed curve of inductance vs pressure. The sample was required to be in one piece, or if fractured, with parts not displaced with respect to each other. Distortion was required to be less than $\pm 2\%$ of any sample dimension; only sodium chloride showed this much distortion. In all other cases distortion was unmeasureable.

The inductance curve was required to be smooth and monotonic, even below 7 kbar. In itself, however, the data below 7 kbar are not considered useful. Data below 7 kbar are readily obtained in truly hydrostatic systems, or even from a simple extrapolation of elastic constants. For our own curves, the data below 7 kbar are extrapolated from the results above 7 kbar.

[7] K. L. Scott, Proc. IRE 18, 175 (1930).

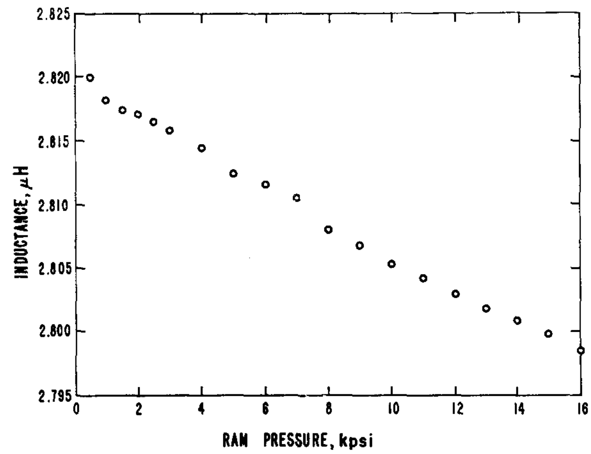

FIG. 2. The directly observed inductance of a coil on a tungsten core as a function of hydraulic pressure applied to rams.

Tungsten

The tungsten core selected for the final test was 0.724 cm in diameter by 0.929 cm long. It was found necessary to paint the core with machinist's blue layout dye to prevent electrical contact between coil and core. The enamel insulation on the wire occasionally failed, sometimes erratically, sometimes very smoothly. Sharp and erratic failures are readily recognized from the inductance of the coil. Smooth, gradual failures may be detected by an anomalous drift in the resistance of the coil, which is also displayed on the bridge. The coil and core were enclosed in a pyrophyllite jacket in hopes that this would more closely approximate the desired rheological balance than silver chloride.

The directly observed inductance data are plotted in Fig. 2. The compression $\Delta V/V_0$ at 55 kbar is 0.0145,

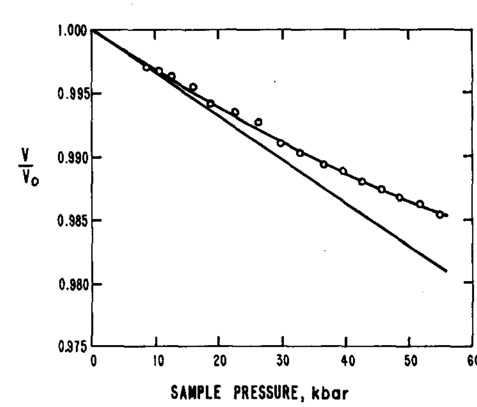

FIG. 3. The computed relative volume of the tungsten core of Fig. 2. The straight line is an extrapolation based on the elastic bulk modulus at atmospheric pressure.

TABLE I. Compression of $SrTiO_3$ at 55 kbar.

Sample size diam cm	length cm	Observed $\Delta V/V_0$
0.821	1.053	−0.024
0.699	0.813	−0.028
0.864	1.078	−0.027

after correction for skin effect. Bridgman obtained 0.0135.[8] A linear extrapolation of the zero-pressure elastic bulk modulus yields 0.0183[9]; this figure, of course, is always slightly higher than the directly observed compression. The elastic data are tangent to the compression data at zero pressure, as shown in Fig. 3.

Strontium Titanate

Three experiments on $SrTiO_3$ satisfied the criteria mentioned earlier. Three sample cores were used, with lava sleeves. In no case was the sample permanently deformed by the application of pressure, within the limits of measurement (0.001 cm). Small cracks were sometimes visible within the samples after test runs. The observed data are presented in Table I. The average compression at 55 kbar is 0.026. The extrapolated elastic data yield 0.032.[10] The total spread of corrected data is $\pm 10\%$ of the volume compression itself.

Cadmium Sulfide

Cadmium sulfide was examined as part of a more thorough study of this material.[11] It undergoes a polymorphic transition at 23 kbar, and we present here compressibility data to that point. The low pressure phase is hexagonal (wurtzite structure); so the collapse of the coil does not strictly yield bulk compression. Its elastic behavior, however, is nearly isotropic[12] and no significant error is introduced. Samples were recovered undamaged and without measurable distortion. The observed results are listed in Table II. The average compression is 0.042. The zero-pressure bulk compressibility (16.6×10^{-13} cm^2/dyn) would yield 0.038.[12] The results are a good indication of the reproducibility of the coil behavior.

TABLE II. Compression of CdS at 23 kbar.

Sample size Diam cm	Length cm	Observed $\Delta V/V_0$
0.635	0.634	−0.041
0.635	0.635	−0.045
0.622	0.755	−0.041
0.901	1.263	−0.042

TABLE III. Compression of NaCl at 55 kbar.

Sample size Diam cm	Length cm	Core volume cm^3	Observed $\Delta V/V_0$
0.90	1.13	0.72	0.115
0.63	0.67	0.21	0.148
0.89	1.07	0.67	0.129
1.15	1.29	1.35	0.113
0.70	0.81	0.31	0.147

Sodium Chloride

Finally, we tested sodium chloride as an example of a material of relatively high compressibility. Salt caused by far the greatest operational difficulties, with many runs which failed to fulfill the criteria of acceptability. Most of this difficulty is probably due to the low shear strength of NaCl. An AgCl jacket was used, but even so, the coil was recovered with the wire pressed inconsistently into the threads on the crystal. The crystals were usually recovered in one piece, but considerable distortion and change in dimension were common.

The observed data are summarized in Table III. The average compression is 0.13. For comparison, Bridgman obtains 0.132 compression at 55 kbar.[13]

There was no apparent correlation between the character of the permanent distortion and the observed result. It is noted, however, that there is a one-for-one correlation between core volume and the observed compression. This suggests a need for adjustment of the rheological environment.

REMARKS ON POLYMORPHIC TRANSITIONS

In the course of measuring compressions, many samples containing bismuth, thallium, and barium have been studied. The coils have in all instances revealed the transitions sharply. The Bi I → II and II → III changes, for example, have been displayed separately in every case. The friction present in many mechanical compression testers, such as piston-cylinder apparatus, is sufficient to obscure these individual volume changes for Bi. The sensitivity of the coil to small changes ($\Delta V/V_0 = 0.001$) has been noted previously.[1] The sensitivity and resolution are especially useful features of the coil method in studies of sharp, sudden volume changes at high pressures.

[8] P. W. Bridgman, *The Physics of High Pressure* (G. Bell and Sons, Ltd., London, 1952), pp. 160–161.
[9] C. Kittel, *Introduction to Solid State Physics* (John Wiley & Sons, Inc., New York, 1956), p. 93.
[10] J. B. Wachtman, Jr., M. L. Wheat, and S. Marzullo, J. Res. Natl. Bur. Std. **67A**, 193 (1963).
[11] G. A. Samara (to be published).
[12] S. S. Kabalkina and Z. V. Troitskaya, Doklady Akad. Nauk SSSR **151**, 1068 (1963)[English transl.: Soviet. Phys.—Doklady **8**, 800 (1964)].
[13] P. W. Bridgman, Ref. 8, p. 163.