Measurement of Volume and Resistivity Changes in High-Pressure Experiments by an AC Inductive Technique

Published in:
J. Basic Engr.
86, 736 (1964).

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A coil wound on a sample core subjected to high pressures may be used to measure compressibility and electrical resistivity of the core. In a first approximation, the coil reactance reflects core volume, and the resistance reflects core resistivity. The exact impedance of the coil in the general case depends on the ratio of skin depth to sample size. With large skin depths, the sensitivity to volume changes is high, and with small skin depths, sensitivity to resistivity is high. Operation in these two cases was demonstrated for four materials of widely different hardnesses: barium, bismuth, titanium, and tungsten. In addition, operation was demonstrated at high temperatures by detection of the alteration of pyrophyllite to coesite plus kyanite. Permeability as a function of pressure was shown for an iron-nickel alloy. The chief advantage of the coil method is the remote, nonmechanical measurement of volume changes. In addition, it offers great versatility to favor many different types of sample material.

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We have been studying high-pressure phenomena in solid materials by indirect audio and radiofrequency techniques. The ac impedance of a small coil enclosing the sample material may be analyzed to indicate both volume (or density) and resistivity of the sample. As an approximation, it may be said that the reactive component reflects sample volume and the resistive component reflects resistivity.

The inductive method has a number of distinct advantages in comparison with the more usual methods. Resistivity is normally measured in a dc bridge, with direct electrical connection to the sample, usually by wire leads to the press anvils. Volume is usually measured by the position of a small piston which contacts the sample and extends to the outside of the high-pressure container. The dc resistivity technique is not afflicted with insurmountable problems, but the uncertainty of contact resistance can be troublesome, and also the very low resistance of metallic samples. The common volume-sensing technique, however, is increasingly awkward at higher pressures. The necessity for a moving mechanical connection severely restricts the design of the pressure vessel and press configuration and introduces difficulties with friction. The most important advantage of the inductive technique is the ability to study volume changes without any direct mechanical indicators. It is inherently free of any marked frictional effects.

In addition, there are numerous other advantages to the inductive method. No electrical connections to the sample are needed; this eliminates any possible problem with contact resistance. The impedance of the test coil may readily be chosen so that it is many times the impedance of the connecting leads. The operating frequency may be chosen over a range of about 100 cps to 100 Mc without difficulty in order to maximize the sensitivity for a particular property of a particular sample. The resistivity of samples ranging from conductors through semiconductors and even insulators may be measured easily. Solids, fragments, and compacted powders may be investigated. As a further bonus, once familiarity is gained in use of the primary inductive method, more elaborate dual coils may be used to study magnetic permeability under pressure. Further, all properties may be studied as a function of temperature at high pressure.

ELECTRICAL THEORY

Earlier it was stated that, roughly speaking, the inductance of the coil depends on sample volume, and the ac resistance on sample resistivity. While this is indeed nearly true over a certain range, the inductive method is much more useful if the general case is considered. The deviation from the rough rule stated above is due to the variation in the electrical skin depth of the sample core.

The impedance of a coil on a cylindrical core of conductive material has been calculated by Scott (1).[1]

$$\frac{\omega L}{\omega L_o} = (\sqrt{2}\ s/a)\ \frac{ber x\ bei' x - bei x\ ber' x}{ber^2 x + bei^2 x} \tag{1}$$

$$\frac{R-R_o}{\omega L_o} = (\sqrt{2}\ s/a)\ \frac{ber x\ ber' x + bei x\ bei' x}{ber^2 x + bei^2 x} \tag{2}$$

The symbols indicate these parameters:

L, R = inductance and resistance of coil with core

L_o, R_o = initial inductance and resistance of coil with core under pressure if eddy currents were absent

s = electrical skin depth of core, cm = $5033\ \rho^{\frac{1}{2}} (\mu f)^{-\frac{1}{2}}$

ρ = resistivity of core, ohm-cm

μ = permeability of core, cgs units

f = frequency, cps

a = radius of core, cm

$x = \sqrt{2}\ a/s$

By direct substitution for a few points in equations (1) and (2), we may calculate a rough