The Inductive Coil Technique for High-Pressure Measurements: An Analysis of Nonhomogeneous Material Environment as a Source of Irreproducibility and Error

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INTRODUCTION

The theory and methodology of the inductive-coil technique for bulk physical measurements under high pressures have been described in detail previously (1,2,3).[1] The device enjoys simplicity in principle and practice. Pressurewise, its range of application is essentially unlimited. Its sensitivity is high. Short-term volume changes to at least 0.01 percent can be detected without difficulty. Polymorphic transformation and bulk compressions have been quantitatively reproduced to better than 2 percent.

Neither high sensitivity nor reproducibility, however, assure accuracy. Reproducibilities of 2 percent require exact duplication of all experimental components and parameters. Variations in either or both have yielded, in some cases, differences in data as great as 50 percent. In the past, "best experimental arrangements" for given types of materials were determined empirically. Criteria consisted of a ± 2 percent residual distortion limit, and close correlation with data on equivalent or analogous materials obtained by other techniques. Neither requirement resolves the question of accuracy.

An attempt is made here to analyze significant sources of error based on experimental experience and elastic theory. Some sources are as obvious as their remedies. Others are more subtle but of surprising potential import. All are mechanical in nature and all are subject to corrective measures. Corrections are suggested which are considered sufficient to permit accuracy to be included with the already established qualities of versatility, simplicity, and sensitivity.

DISCUSSION

It has not been possible to eliminate stress gradients completely in high-pressure equipment which use solids for pressure transmission. This is especially true during pressure buildup or release. A hydrostatic environment is preferred, of course, for operation of the inductive-coil device. Stress differences in some types of solid apparatus, however, can be minimized locally to the point where they are of negligible consequence for most applications (4).

It has been found that minor simple deformations of the coil-specimen assembly to approximately ± 2 percent can be tolerated without seriously impeding performance (3). The effects of such distortion are subject to some degree of mathematical treatment, but direct experimental evaluation is simpler, more versatile and specific, and has been used for the most part. Reproducibilities of 2 percent are possible within the ± 2 percent deformational limit.

In practice, the bulk of minor displacements occur during the early stages of pressurization and during the last stages of pressure release; that is, during the period of maximum gasket instability in the case of apparatus which operate on the principle of the compressible gasket. Their effects on coil performance generally can be circumvented by back extrapolation of data from higher pressures. Injudicious exposure of coil assemblies to excessive shear will not be considered. Two principal sources of error in the inductive-coil technique are the coil-specimen coupling and the interrelationship of elastic properties of the solids involved. Let us first consider the coil.

One simple but significant experimental difficulty with the coil has been mismatching between the wire and its receiving groove on the specimen core. This difficulty is more pronounced when cores are electrical conductors and an insulating film must be maintained. Tolerance generally results in initial unstable coupling between the coil and specimen. This case is illustrated by Fig.1A.

If the core and pressurizing solid both possess a greater resistance to plastic flow than the wire, the latter will yield under pressure to com-

[1] Numbers in parentheses designate References at the end of the paper.

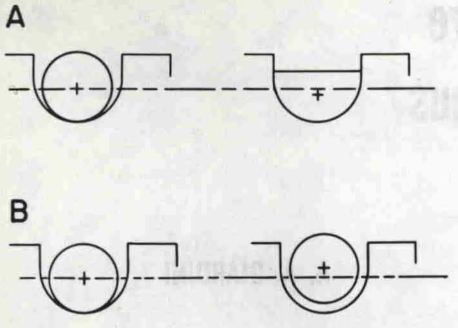

Fig. 1 Part A illustrates the centerpoint depression of a circular coil wire in an oversize circular groove under pressure when wire possesses a lower yield strength than pressurizing solid and core. Result is an apparent volume decrease of core. Part B illustrates case of a similar coil pressurized by a material less resistant to plastic deformation than wire. Result here is a virtual suspension of coil in pressurizing material. Magnitude of error from this source is dependent upon relative compressibility of core and presence of slack in coil winding

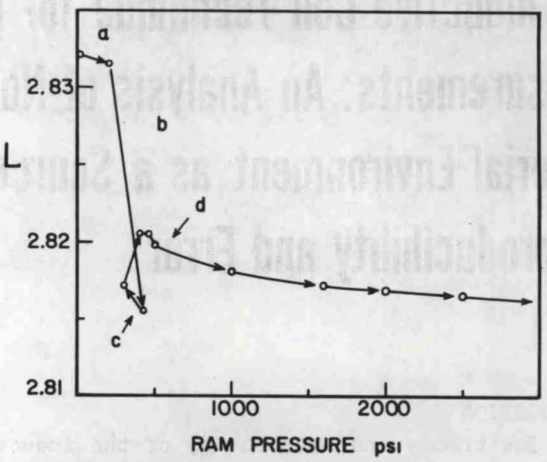

Fig. 2 Inductance, L, response to approximately 15 kilobars of pressure from a copper coil hand-wound on a threaded tungsten core. The coil-core assembly was jacketed by a thin pyrophyllite sleeve, encapsulated in silver chloride, and compressed in a standard pyrophyllite block. The interpretation of coil behavior is as follows: (a) Initial compression of the coil; (b) yield, collapse and compliance to groove geometry as illustrated in Fig. 1A; (c) a transitory pressure drop and minor deformation of coil caused by seating of preformed gaskets on pyrophyllite block; (d) stabilization and full coupling of coil to core

ply with the profile of the groove. The cross-sectional centerpoint of the wire will be depressed radially with respect to the core, thus effecting an apparent reduction in specimen volume. A simple calculation for the case of a "standard" 0.005-in-dia wire wound on a 0.250 in. x 0.312-in-long core threaded with a +0.0005-in. groove shows a reduction in coil volume of approximately 1 percent. This is an appreciable quantity when dealing with compression measurements on relatively incompressible materials. Should slack occur during winding of the coil, an excess of copper will result. This excess will cause a raising of the centerpoint and thus either compensate for or exceed the apparent volume reduction which a wire-groove mismatch otherwise would produce.

If the core material possesses a lower shear strength than either the pressurizing solid or wire, then a preferential displacement of the core will occur. This generally is reflected primarily by a shortening in length of the specimen, although concavity of the ends also has been observed.

Errors which result from dimensional mismatch between wire and groove, or slack in coil winding, are fixed and persist through the course of an experiment. They are difficult to evaluate quantitatively. Corrective measures are simpler. Zero tolerance between wire and groove can be achieved by pre-experimental prestressing to enforce wire-groove compliance, or by employing wire and grooves of rectangular cross section. Coil slack can be eliminated by mechanical winding under controlled tension.

An additional type of coil difficulty is illustrated by Fig.1B. Here consider an environmental pressurizing material less resistant to plastic deformation than the wire. Take, for example, silver chloride enclosing a copper coil wound on a threaded tungsten core. Pressurization will cause the chloride to flow about the wire. The result is a virtual suspension of the coil which will include any slack in the winding. Coil response with respect to volume change would be essentially that of its own compression. In this particular case, however, the relative strain-hardening characteristics of the silver chloride and copper are such that coupling between coil and core does occur with increasing pressure owing to the preferential hardening of the AgCl. Corrective measures for this source of error are as follows:

(a) Use of a thin protective jacket around the coil to prevent intrusion of the pressurizing solid about the coil.

(b) Use of a more rigid pressurizing material.

(c) Use of a less rigid coil wire such as lead.

(d) Zero tolerance between wire and groove.

(e) Tensional winding of the coil.

The effectiveness of the protective jacket has been verified experimentally. It will be