## HIGH PRESSURE TRANSITIONS

FIG. 1. The normalized impedance of a coil surrounding a conductive core with unity filling factor. The ordinate is the inductive reactance; the abscissa is the net increment to the ac resistance of the coil. The letters A, B, C, D denote regions discussed in the text. Skin depth decreases clockwise on the curve.



discussed above are indicated in Fig. 1 by A for  $a/s \rightarrow 0$ , B for  $a/s \rightarrow \infty$ , and C for  $a/s \approx 1$ . There is a fourth special region D, where volume and resistivity changes will be completely mixed and reflected solely by an inductance change. No separation of information is possible in region D.

In the discussion so far we have confined ourselves to the case where the core material has a permeability of essentially unity, i.e., a diamagnetic or a paramagnetic material. Such a material is well suited for the present considerations since, in general,  $\mu$  will not be appreciably affected by pressure and can thus be treated as a constant in the analysis of the data. The situation is more complicated when dealing with ferromagnetic core materials. For these materials,  $\mu$  is usually far greater than unity (ranging up to several thousands), and it is known that it shows wide variations with pressure. Some Ni-Fe alloys can be completely demagnetized by applying pressures of less than 50 kbar.<sup>7</sup> The change in  $\mu$  with pressure is, of course, reflected in the impedance of the coil.

There will be a quantitatively different impedance curve (Fig. 1) for each value of  $\mu$ . Impedance changes due to variations in permeability will be mixed with volume and resistivity effects; it is not possible to separate three variables with only two observed parameters. If it is desired to study permeable samples, an additional coil may be wound as a measure of mutual inductance which is determined by core permeability.6

## EXPERIMENTAL TECHNIQUE

## **Electrical Considerations**

In general, it is desirable to have as high a filling factor as possible. This not only increases sensitivity (especially to volume changes) but also minimizes uncertainties regarding the details of the collapse of the coil when the flow pattern of the pressure medium is considered. It is sometimes feasible to immerse the coil completely in the test material, although generally an adequate filling factor is obtained by winding the coil no more than a couple of wire diameters from a cylindrical sample.

<sup>7</sup> G. A. Samara (unpublished data).

The test coil should have an inductance suitable to the operating frequency. In region A, the inductance of the coil should exceed inductance of the leads by a factor of at least 3 or 4. This is readily arranged at low frequencies by use of coaxial or twisted leads, which allow leads 6 or 8 ft long before the inductance reaches 1  $\mu$ H. In region B, frequencies of several megacycles may be needed, and then it is often practicable to use a similar length of RF coaxial cable and correct the data with a Smith chart. In region B, the ac resistance of the coil will usually far exceed the resistance of the leads. It turns out that a coil of about 1-10 µH of inductance, readily fabricated with a singlelayer winding of 10-50 turns on samples about  $\frac{1}{10} - \frac{1}{2}$  cm<sup>3</sup>, is suitable for most measurements from 1 kc to 10 Mc.

The impedance of the coil is also affected by the proximity of the press anvils. This interference is usually not serious with carbide anvils if the coil occupies no more than a few percent of the interanvil volume.

Low frequency measurements were taken on a General Radio inductance bridge No. 1632, and high frequency measurements on a Boonton RX meter No. 250A.

## **Mechanical Details**

All experiments performed in this study were carried out in a Barogenics 2000-ton cubic multianvil pressure apparatus. Details on the unit are already in the literature.8 Massive pyrophyllite (American Lava Company, Grade A Lava) was used for specimen containers and as the solid pressure-transmitting medium. The inductive sensing method under discussion is adaptable, however, to any type of high pressure equipment not plagued with unreasonable or unpredictable specimen distortion.

Copper wire insulated with Formvar was used for the coil in the room temperature work presented here. Copper has suitable electrical and mechanical properties which vary smoothly with pressure. The upper part of Fig. 2 presents a perspective drawing of the basic experimental arrangement used in this work. The lower part of Fig. 2 shows an exploded view of a typical assembled coil with a specimen core.

A number of specific details were considered in design of the apparatus and measurement procedure:

(1) Highly refined (99.999%) bismuth was used as a specimen core material. Bismuth has been more thoroughly studied under pressure than any other material. It also has the advantage of possessing several distinct polymorphic transitions.

(2) Integral preformed gaskets were machined on the pyrophyllite specimen container to minimize sample deformation during initial stages of pressure application.9

<sup>&</sup>lt;sup>8</sup> A. Zeitlin, Mech. Eng. 83, 37 (1961). <sup>9</sup> G. A. Samara, A. J. Henius, and A. A. Giardini, Am. Soc. Mech. Engrs. Paper 63–WA–341 (1963).



FIG. 2. Top: A cutaway schematic view of the pyrophyllite sample container. Bottom: An exploded view of the coil and sample core assembly.

(3) Metal seals were employed to reduce specimen distortion at higher pressures due to pyrophyllite flow at the unconstrained corners of the container.<sup>9</sup>

(4) A silver chloride jacket was used around the specimen core in order to produce a nearly hydrostatic environment.

(5) All electrical connections were soldered.

(6) Coil leads were inserted through interanvil gaskets to insulate the circuit from the pressure apparatus.

(7) The connecting coaxial cable was rigidly enclosed and thermally insulated.

A view of an actual assembled coil with specimen core prior to compression is shown in the left half of Fig. 3, and one recovered after compression to 65 kbar is shown on the right. The recovered specimen shows no appreciable distortion, and is essentially free of shear strain.



FIG. 3. Left: A typical coil assembly before compression. Right: After compression. This coil is 1.25 cm long.

Bobrowsky<sup>10</sup> has described severe inhomogeneities in pressure which are introduced by inclusion of sample jackets whose rheological properties are markedly different from other components of the system. In order to assess any such effects, test coils were wound with wire ranging from 0.007–0.03 cm in diameter, with 20–100 turns, on jackets of lava, bismuth, nylon, and AgCl, over cores of bismuth, Bi-Lucite mixtures, lava, nylon, and ruby laser rod. No serious distortion of the coil or breakage of the coil wire was observed. Brittle samples were usually recovered uncracked. Polymorphic transitions were never seriously "smeared out." From these results, it may be concluded that there were no serious stress inhomogeneities, either static or dynamic, in the useful region of the lava specimen container.

Most measurements were made with a standard coil and core assembly: 25 turns of No. 35 copper wire (diame-



Fig. 4. Cross section of a standard coil and core after compression.

ter 0.014 cm) wound on a silver chloride jacket, diameter, 0.8 cm, length, 1.25 cm. The standard sample core was 0.63 cm in diameter by 1.25 cm long. Figure 4 shows a polished cross section of a recovered standard coil and bismuth core. The specimen had been compressed to 65 kbar.

For operation in region C of Fig. 1, a special filamentary core was made. This was assembled from 400 needles of bismuth, diameter, 0.04 cm, length, 0.2–0.3 cm, enclosed in four stacked lava disks, each drilled with 100 holes. This proved much more satisfactory than cemented powders, which suffer erratic insulation breakdown under pressure. A cross section of the recovered filamentary core is shown in Fig. 5.

<sup>10</sup> A. Bobrowsky in Ref. 1, p. 95.