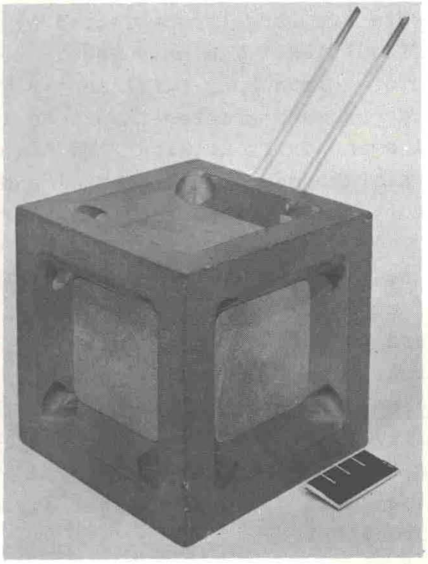

Fig.2 View of assembled pyrophyllite sample container. Wire leads are 14-gage bare copper; block is $2^7/8$ in. on a side

frequency measurements were made with an RX meter (7). An RF-bridge would simplify high-frequency studies by obviating the need for conversion of parallel impedances to series impedances. No bridge was available to us at the time of this study.

Mechanical Assembly

Sample Containers. The sample containers were made of massive pyrophyllite, which also served as the principal medium for pressure transmission. The pyrophyllite was used in the condition as received from the supplier, without heat-

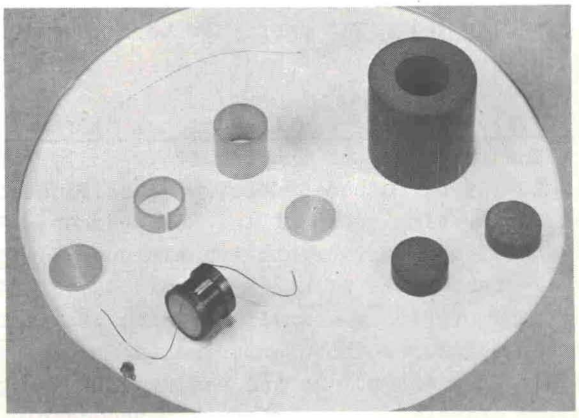

Fig.4 Polished cross section of a coil and bismuth core which have been compressed to 65 kb. The coil is small standard size, but core is $^1/2$ in. long in this case

treatment or desiccation. Preformed gaskets were machined on the pyrophyl-block. These minimized sample distortion during the early stages of pressure application. Metal seals were usually inserted on the depressed faces of the block to restrict pyrophyllite flow at the edges of the block between anvils. Fig.2 shows an assembled pyrophyllite container.

Silver-chloride jackets were usually placed around the coil or between the coil and core to produce a more nearly hydrostatic environment, and thereby reduce coil and core distortion. Fig.3 shows a typical core, coil, silver-chloride sleeve, pyrophyllite casing, and sample container block before assembly. Fig.4 shows a polished cross section of a coil and bismuth core after compression to 65 kb. The absence of severe deformation is noteworthy.

Pressure Apparatus. All experiments report-

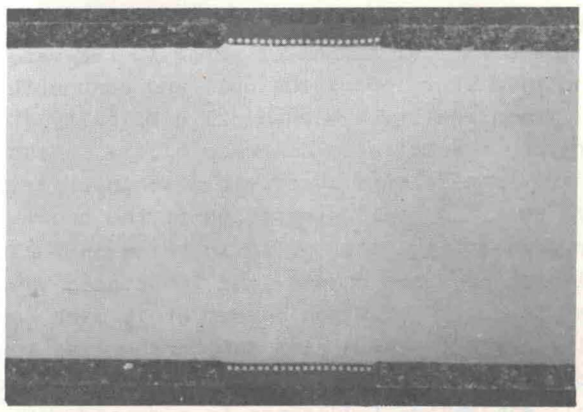

Fig.3 Components of a typical small standard coil-and-core assembly. Coil is wound directly on sample core, which is $^1/4$ in-dia by $^1/4$ in. long. Collars which retain ends of coil are silver chloride. Actual assembly, small silver-chloride band, which is cut to permit expansion, would be fitted around coil. This subassembly is then placed within the silver-chloride sleeve, and silver-chloride disks are used to plug end of sleeve. Resultant unit is put inside thick-walled pyrophyllite cylinder, whose ends are plugged with pyrophyllite disks. Whole unit is inserted into a hole bored through pyrophyllite sample-container block (not shown). Finally, end of this hole are filled by large pyrophyllite plugs. Coil would be connected to thin copper disks at each end of pyrophyllite sample cylinder, just beneath these plugs. Connecting leads are soldered to these disks

ed here were made with a hexahedral press of 2000-ton capacity (8). The press has been described previously (9).

Sensitivity, Errors, and Operational Difficulties

The precision of the inductive-coil method is logically discussed under two classifications, based on the time scale of the measurement. First is the study of polymorphic transitions (5). These normally take place within a few minutes of time and extend over only a small fraction of the total range of pressures in an experimental run. Observed values for volume and resistivity changes in these transitions are readily reproducible with good precision. Second is the study of slowly varying properties, which require measurement of small changes over the whole range of available pressures. An example of the second class is bulk compressibility. Measurements of this class of properties require that account be taken of all the slowly accumulated distortions of sample, coil, and connecting leads. The precision for slowly-varying quantities is considerably less than for rapid transitions.

Electrical System. The inductance of a typical coil used in this work may be read to five significant figures on the bridge at 1000 cps. The absolute accuracy of the measurement is, of course, less than this. It does suggest the ultimate sensitivity to changes in volume. We have been able to detect easily sudden volume changes of 0.1 percent (5). The scatter in the data indicated that with precautions, this lower limit would be near 0.01 percent. Resistivity can be read to three figures, and the estimated sensitivity to sudden changes is 1 percent.

The errors and difficulties which can properly be called purely electrical are quite minor. One such minor trouble was the slight influence of the magnetically permeable press anvils. The additional inductance from this source was about 0.01 μh, or about 0.3 percent of total inductance. Since this additional inductance is so small and should remain essentially constant, no correction was applied to the data.

The principal source of error in the measurements, distortion of the coil and connecting leads, is in reality a mechanical difficulty rather than electrical. Compressibility measurements are particularly susceptible to interference of this sort. A small stretching of the heavy copper connecting leads, or an increase in their mutual distance, can change lead inductance by several percent. This could completely mask compressibility effects in hard substances like tungsten or diamond. Further, distortion of the core and coil can change inductance in a manner not readily corrected. On the other hand, resistivity measurements are largely unaffected by any of these difficulties. The only requirement is in selection of a frequency sufficiently high to make coil impedance much greater than lead impedance.

Noise and drift arising from thermal and motional disturbance of the coaxial cable were minimized by enclosing the cable in a rigid conduit.

No difficulty was experienced with the "Formvar" enameled wire used for the coil, either with regard to insulation breakdown or breakage. The smallest size used was 41-gage.

Mechanical Components. Most of the mechanical difficulties arise from two sources; nonhydrostatic stress near the sample, and pyrophyllite flow at the edges of the container during initial gasket formation.

In a study of polymorphic transitions, nonhydrostatic stress will often retard the completion of a sudden volume change. All parts of the sample core do not feel the same pressure, and the transition commences at different ram pressures in different regions of the core. As a result, the transition is "smeared out."

Another effect of nonhydrostatic stress is a transient "negative" pressure which has been apparent on occasion. In this case, a volume reduction commences in the sample core, but internal friction prevents the surrounding medium from maintaining pressure throughout the sample core. Certain portions of the sample core actually feel a slight reduction in pressure. This effect is a major cause of the broadening of the range of ram pressure required to complete a transition.

For long-term observations, such as a compressibility determination, nonhydrostatic stress causes both temporary and permanent core and coil deformation which can invalidate the results. While temporary deformation can only be inferred, any permanent deformation which is comparable to sample compressibility strain is cause for rejection of the data. To reduce both types of distortion, the sample core should occupy only a small part of the sample container. Besides being small, the sample core should approximate the cubical symmetry of the sample container. We found that, with hard materials, our elongated cylindrical cores, $\frac{1}{4}$ in. dia by $\frac{1}{2}$ in. long, were entirely unsuitable for compressibility measurements. The sample cores suffered barrel distortion from the higher stress on their ends which were closer to the anvils. The short core, $\frac{1}{4}$ in. dia by $\frac{1}{4}$ in. long, remedied this ailment. A standard short core discussed previously made of titanium and encased in silver chloride, experienced a "blowout" on pressure reduction from 50 kb. Despite severe