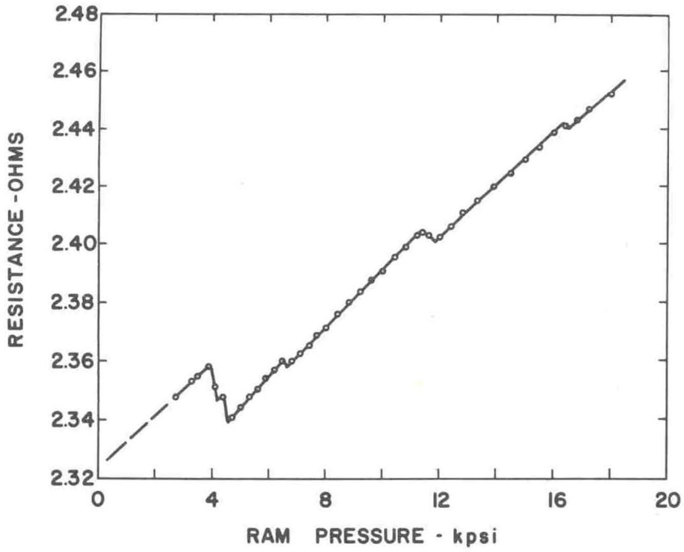

Fig. 3 Response of manganin gage used in reexamining the upper Bi transition. A Bi-Tℓ -Ba core was used

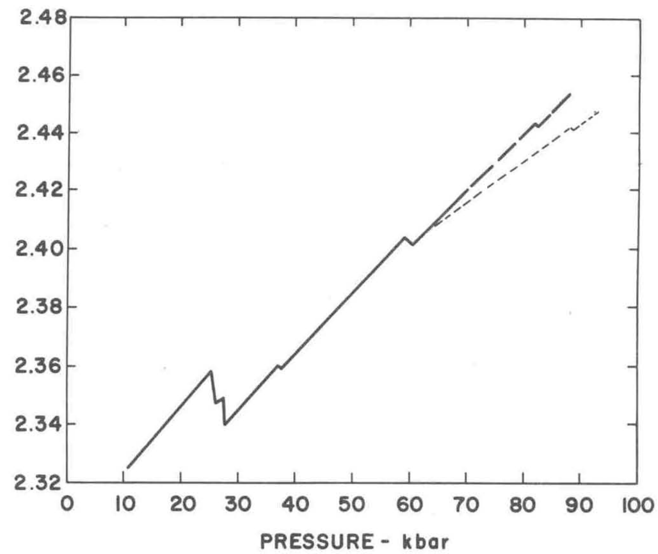

Fig.4 Resistance versus true pressure curve obtained from the data in Fig. 3 and showing the upper Bi transition at 81-82 kbars

Fig.4. The value of the resistance at the upper Bi transition is 2.4420 ohms. Extrapolating the calibration curve in Fig.4 linearly above 70 kbars (dashed line) gives for this value of the resistance a transition pressure of 81-82 kbars. For the transition to be at 88 kbars, the slope of the curve would have to change drastically (dotted line), and this is unlikely.

This new value for the transition is confirmed in another way. We have previously shown (1) that for our hexahedral apparatus using standard size pyrophyllite blocks, the points for the lower Bi, Tℓ and Ba transitions fall on a straight line on a plot of true pressure versus applied ram pressure. Fig.5 shows results from the present experiment. The open triangles represent the points from the manganin gage. The linearity of the calibration up to 60 kbars is clearly exhibited. Extrapolating this response beyond 60 kbars yields a value of 81 kbars for the upper Bi which occurred at an applied ram pressure of 16,400 psi. For the transition to be at a higher pressure than this would indicate an improvement in efficiency which is hardly likely and has never been observed experimentally. The 88-kbar value is indicated in Fig.5 by the closed triangle.

Fig.5 also shows the results from one of the multiple-event cells. The transitions in the calibrants came in at different applied pressures than in the manganin gage,[3] but this is of no consequence. The upper Bi came in at a ram pressure of 18,600 psi. Again, extrapolating the low-pressure results beyond 60 kbars yields a value of 82 kbars for this transition. An 88-kbar value is indicated by the closed circle.

We believe that the present results show that the long accepted 88-kbar value for the upper Bi transition is too high. The obtained value of 81-82 kbars is probably an upper limit. The final value will have to await further determinations, preferably by other techniques.

[3] Reference (1) discusses the pressure homogeneity in the hexahedral apparatus.

COMPRESSIBILITY MEASUREMENTS

We have previously (10) described an a-c inductive technique for simultaneously measuring volume and resistivity changes in solids under pressure and have demonstrated its accuracy and sensitivity in studying polymorphic transformations. We have recently incorporated several improvements into the technique which allow us to measure compressibility with a high degree of sensitivity and reproducibility. In addition, the work has been extended to elevated temperatures.

One of the major improvements in the technique is our development of a high-strength coaxial cable and connector for use at high pressure and temperature. In our earlier work two parallel copper wires were used as leads to the coil. In the initial stages of compression and gasket formation, these leads suffer a considerable amount of distortion and motion relative to each other, and result in a fairly large change in lead inductance. The effect of this distortion is oftentimes of the same order of magnitude or even larger than the desired change due to the sample. As a first approximation, one can measure the change

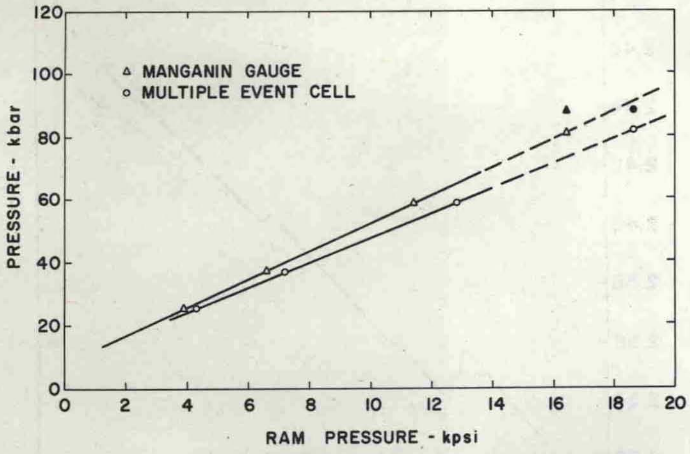

Fig. 5 True pressure versus applied ram pressure calibration curves showing the upper Bi transition at~81 kbars. The long accepted 88-kbar value is indicated by the dark symbols

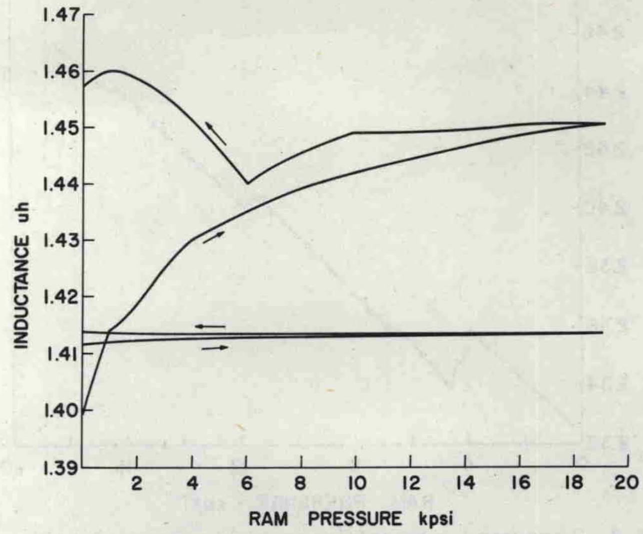

Fig. 7 Comparison of the change in lead inductance with applied pressure between the high pressure coaxial cable and the previously used two parallel copper leads. The inductance of the coax is essentially independent of pressure

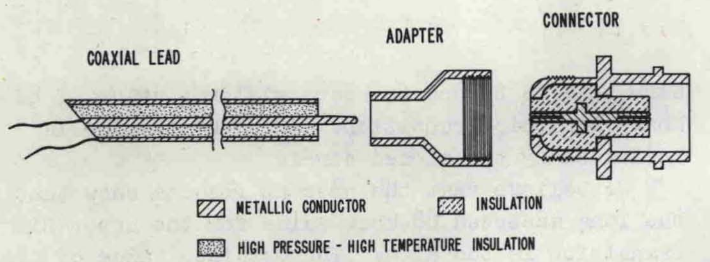

Fig. 6 Schematic diagram of high pressure coaxial cable and connector. The two thin wires at the end are soldered on to facilitate connection to the sample inside the high pressure chamber

in the inductance of the leads in one experiment and subtract it from the total inductance measured in another experiment. However, this procedure is not too satisfactory since the change in the inductance of the leads can vary in an unpredictable manner from one experiment to another. This is the reason why some of our published curves (10) showed considerable scatter, and sometimes anomalous behavior, in the low pressure region (below ~20 kb).

The use of the coaxial cable mentioned previously remedies this difficulty. Here the deformation of the cable is tolerable and there is practically no motion of the two conductors relative to each other. The inductance of the cable changes only slightly with pressure and the behavior is reversible and reproducible. Fig.6 is a schematic of the cable and connector. The cable is made of a copper center conductor and a stainless steel sheath separated by an insulator such as pyrophyllite or MgO. Commerical cable of this type has been found to be satisfactory and is currently in routine use. The end connection is designed to fit standard coaxial connectors. Fig.7 compares the change in lead inductance with pressure for the two methods. This remarkable improvement allows us to measure compressibility from the initial stages of compression.

In addition to the aforementioned usage, this coaxial-cable technique is useful for a variety of other pressure experiments using a-c techniques where the sensitivity is such that the noise level of the leads becomes an important factor. This is the case in experiments dealing with the measurement of certain dielectric and magnetic properties. It has also proven useful for multiple thermocouple and other d-c circuitry in the multianvil type apparatus.

Another improvement in the coil technique is the winding of the coil directly on a threaded sample. This eliminates the need for making a correction for the filling factor and eliminates any uncertainties that may be introduced due to differences in the compressibilities and rheological properties between the sample core and enclosing sleeve. With some effort one can thread most materials. We have threaded (usually 80 threads/in.) samples of Bi, W, CdS and a variety of other materials. With metallic samples it is important to have clean threads in order to prevent insulation breakdown of the formvar coating on the copper wire of the coil. We have experienced this breakdown in a few cases.

Improvements in techniques previously described (1,10) practically eliminate any sample deformation. With relatively soft samples such