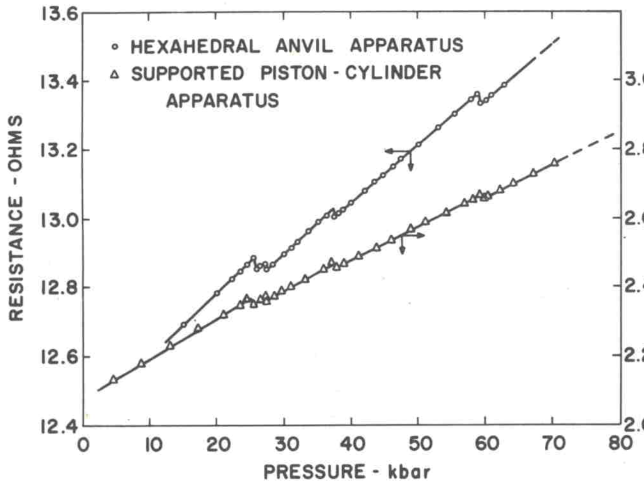

Fig. 1 Typical manganin gage responses in the hexahedral anvil and piston-cylinder apparatuses. The manganin wire was wound on a threaded AgCℓ sleeve enclosing a composite core of Bi, Tℓ and Ba

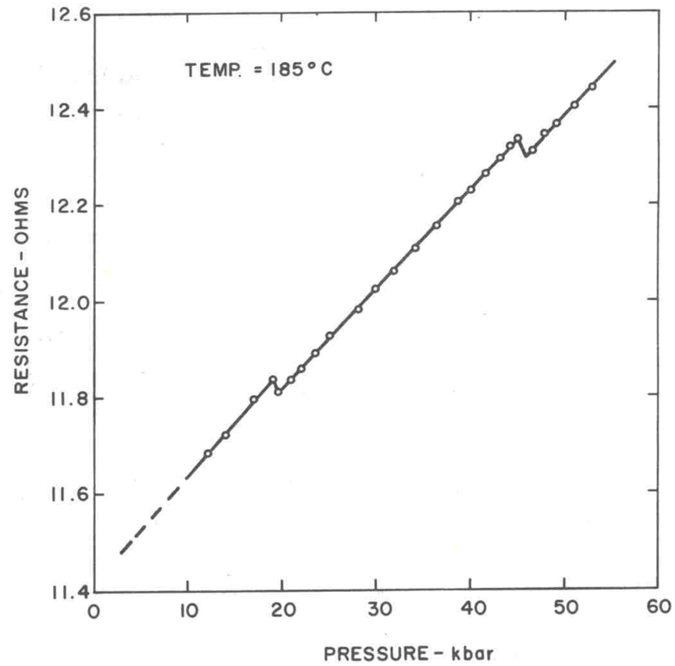

Fig. 2 Manganin gage response at 185 C. The manganin wire was wound on a threaded pyrophyllite sleeve enclosing a Tℓ core

used for pressure measurement in this range have centered around the measurement of discontinuities in electrical resistance associated with the pressure-induced transitions in bismuth, thallium and barium. In between and beyond these reference events, pressure is measured by interpolation and extrapolation, respectively. Pressure values for the transitions were first established by Bridgman (5). Later workers have confirmed Bridgman's values for the low bismuth transitions, commented on the barium transition, and offered some improvement for thallium (6,7). It is now generally believed that these transition pressures are known to about ± 1 percent for Bi and Tℓ and ± 3 percent for Ba.

We have developed two pressure-sensing devices for use in piston-cylinder, multianvil, and other relatively large-volume pressure apparatus. One is a multiple-event calibration cell and the other is a continuous manganin pressure gage with multiple integral calibrants.

Multiple-Event Calibration Cell

We have been using this technique for some time (1,8). The cell consists of small sections of bismuth, thallium, and barium wire cut in lengths inversely proportional to their respective resistivities and encapsulated in series in a silver-chloride sleeve. Pointed spacers of a relatively hard inert wire such as platinum-rhodium are placed on either side of the Ba in order to pierce its usually oxidized surface and insure good ohmic contact. Copper or gold contacts are used between the Bi and Tℓ and the leads. The cell can be made in a variety of sizes and shapes.

Cells such as this provide five fixed points in the 100-kbar range on a sample-pressure versus applied ram-pressure plot, with another point available at around 140 kbar (upper Ba) for possible use to higher pressures. Careful interpolation between points is acceptable; however, extrapolation much beyond any reference event is not recommended unless the operating characteristics of a given apparatus as well as the rheological behavior of the material within the pressure chamber are well understood.

Manganin Gage With Integral Calibrants

This gage is designed for continuous pressure measurement at room and moderately elevated temperatures (3). The principle of its operation is based on (a) the nearly linear dependence of the resistance of manganin on pressure, (b) the volume discontinuities accompanying pressure-induced polymorphic transitions in the calibrants and (c) the appreciable strength and rigidity at high pressure of the commonly used solid pressure transmitting media.

Basically, the gage consists of a manganin coil wound on a threaded insulating sleeve which encloses a core of primary calibrants. Bi, Tℓ and Ba are used for calibrants and they are proportioned so as to give small and approximately equivalent volume changes at their respective transitions.

Briefly, the operation of the gage is as follows: The gage is embedded in the pressure-transmitting medium and its d-c resistance is measured as a function of applied pressure. The resistance of the manganin increases up to the point where one of the calibrants undergoes a phase transition. At this point the volume within the coil decreases discontinuously. The pressure-transmitting medium does not flow fast enough to follow the sudden volume change. As a result, a "cave effect" develops (1). The manganin coil "feels" a sudden lowering of the local pressure and its resistance decreases. Additional force must be applied in order to collapse the cave and reestablish the pressure intensity on the coil. Once this collapse is completed, the resistance of the manganin rises again with increasing pressure until another phase transition in the calibrants is encountered, and the same behavior is repeated. The overall response, therefore, consists of segments showing a smooth increase in resistance versus applied pressure separated by very small regions where the resistance decreases slightly. On a resistance versus actual pressure plot the response consists of nearly linear segments of increasing resistance separated by small decreases. If the reference events are not separated by a large pressure range (say not more than 20-30 kb), then, to a very good first-order approximation, the change in the resistance of manganin between neighboring reference points can be taken as linear.

Fig.1 shows typical gage responses at room temperature. It will be noted that in the two pressure apparatuses the pressure coefficients are quite different even though the same stock of manganin was used.[2] However, this is no longer of any consequence since internal calibrants were used. Here we have in effect a continuous pressure-measuring technique with built-in check points. The linear response between adjacent check points allows one to measure pressure fairly accurately at any point.

Fig.2 shows the response of the gage at 185 C. For this experiment the manganin coil was wound on a threaded pyrophyllite sleeve enclosing a Tℓ core. At 185 C, Tℓ has two transitions in the 50-kb range; hcp → bcc at 19kb and bcc → fcc? at 45 kb.

It should be mentioned in passing that the gage can be used for determining unknown volume changes associated with phase transformations. The method assumes that the decrease in the resistance of the coil at the transition is proportional to the decrease in the volume of the core. Then, by enclosing a core made up of the unknown along with one or two calibrants and comparing the responses, a fairly good estimate of the volume change of the unknown can be obtained. The technique turns out to be fairly quantitative (3).

Of course the accuracy of the gage is limited to the accuracy to which transition pressures in the calibrants are known. As mentioned earlier, in the range up to 60 kbars, the Bi and Tℓ transitions are known to about ± 1 percent and the Ba transition to about ± 3 percent. Above this range, pressures are much less accurately known. We have suggested that the present gage can be very useful in establishing more accurate pressure values for transitions in this higher range (3). The technique assumes that extrapolation of the response of the gage beyond the highest well-established fixed point can be carried out with a higher degree of certainty than with presently used techniques, provided that such an extrapolation does not extend far beyond the reference event. We have applied this in reexamining the upper Bi transition.

At present, the highest reasonably well-established fixed point is the 59 kbar Ba transition. The next best higher point is the Bi transition long believed to be around 88 kbars; however, some recent results suggest that it is more nearly at around 80 kbars (9). We designed an experiment utilizing the two pressure-sensing devices described in the foregoing in order to check this discrepancy. To do this, the range of our equipment was extended beyond the 65-70 kbar limit by the use of carbide intensifier plates in the manner described previously (1). The plates were in the form of truncated pyramids and were 0.340 in. thick. The manganin gage consisted of manganin wire (0.003 in. dia) wound on a 0.015-in-thick threaded (80 threads/in.) AgCℓ sleeve enclosing a Bi-Tℓ-Ba core. The core consisted of concentric cylindrical sleeves of Bi and Tℓ with a Ba inner core. The overall dimensions of the assembly were 0.173 in. dia x 0.280 in. long. All sensors were monitored simultaneously and gave sharp responses at the transitions. The results show that the upper Bi transition is indeed below the 88-kbar value.

The response of the manganin gage is shown in Fig.3. The derived calibration curve is shown in

[2] In a given setup the slopes between calibration points change somewhat, probably due to altered strain conditions brought about by the cave event. This effect is small in gages where the calibrants are proportioned correctly. The amount of Bi should particularly be kept small because of the large volume change at its first two transitions. Part of the drop in the resistance of the manganin at the transitions may be due to induced strains.