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Peter W. Montgomery, Harold Stromberg,  
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August 1961

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Calibration of Bridgman Anvils, A Pressure Scale to 125 Kbars

Peter V. Montgomery, Harold Strosberg, George H. Lura, and George Jura

Department of Chemistry and Lawrence Radiation Laboratory  
University of California, Berkeley, California

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ABSTRACT

It is shown that a radial pressure gradient exists in the silver chloride when it is used as the pressure transmitting medium in Bridgman anvils. The gradient can be obviated by the use of circular sections of wire. The center of curvature of the wire hoop is made coincident with the anvil center. When the inner and outer diameters of the pyrophyllite retaining ring are  $1/2$  and  $7/16$ " respectively, the pressure,  $P$ , is

$$P = (0.725 + 0.462R) L$$

where  $R$  is the fractional displacement from the center, and  $L$  is the average pressure as determined from the total load and area of the anvil face. The above appears to be valid to 125 Kbars. The Bismuth 6-8 transition is found to occur at  $88.3$  Kbars.

### INTRODUCTION

The simplicity of construction and ease of operation make the anvils designed by Bridgman<sup>1</sup> very attractive for the investigation of high pressure phenomena. Bridgman used the anvils to determine the pressure coefficient of electrical resistance of 72 substances. Phase transitions for a number of solids were deduced from finite discontinuities in the resistance.

In principle, as the internal friction which complicates the determination of pressure in the piston-cylinder apparatus is absent in the anvils, one would expect the discrepancy to be such that the pressures for fixed points would be higher in the piston-cylinder apparatus. However, the reverse is true. The pressures at which Bridgman found transitions in the anvils did not agree with the corresponding transition pressures found in the conventional cylinder-piston apparatus.<sup>2,3</sup> For example, a barium transition found in the piston-cylinder apparatus at 60 Kbar, did not appear in the anvils until a pressure of 80 Kbars was reached. Kennedy<sup>4</sup> has pointed out that, above 30 Kbars, the pressures for all fixed points in the anvils are high, and that reasonable agreement can be obtained if the computed pressures in the anvils are reduced by 30% of the excess above 30 Kbars.

In this investigation, the causes of this discrepancy were determined. The discrepancy was found to be attributable to the combination of the existence of a radial pressure gradient in the solid used to transmit the pressure to the material under study with the fact that the effective load bearing area is less than the area of the anvil face. Because of the simplicity of design and the lack of internal friction, earlier investigators assumed that the pressure on the silver chloride was the load divided by the face area. This procedure

was found to be incorrect. With respect to the area our results showed that if the load was distributed uniformly the effective area was less than the area of the face. This made the discrepancy between pressure scales even poorer.

The answer to this apparent paradox was to be found in the existence of a radial pressure gradient in the silver chloride. The pressures for fixed points in the anvils appeared to be higher simply because they were located in the region of lowest pressure. When the gradient was considered in the determination of the pressure, the discrepancy between the transition pressures found by the two methods disappeared, at least to pressures of 90 kbars.

As a result of this study, we believe that we have established a pressure scale that is valid to about 125 Kbars. We also found that excellent results can be obtained with the anvils when the existence of the pressure gradient is considered in designing the geometry of the sample to be studied. Thus, it was not possible to obtain good results by using a thin strip of metal mounted across the center of the anvil, instead a circular section of a wire sample was required, with the center of curvature of the specimen coinciding with the center of the anvils. In this way the effect of the pressure gradient across the sample could be made negligible.

#### EXPERIMENTAL

The variations we have made in the construction of the anvils has been described.<sup>5</sup> To pressures of 125 Kbars, the radiographic work indicates that no correction need be made for the increase in face area when G.E. Carboly 999 or Kennametal K-11 carbide inserts are used.

Single crystal Harshaw silver chloride sheet rolled to the desired thickness

was used for the pressure transmitting medium. Other solids were tried, but silver chloride was found to be the most satisfactory both with respect to the size of the pressure gradient and with respect to the sharpness of the transition. A punch was used to obtain a disc of the necessary diameter. The diameters of the disks were 0.172, 0.297, and 0.425 in. for use with anvil faces of  $\frac{1}{8}$ ,  $\frac{3}{8}$ , and  $\frac{1}{2}$  in diameter. In order to prevent reaction between the silver chloride and some metals, the discs are sprayed with an acrylate resin.

The silver chloride was retained with pyrophyllite rings coated with ferric oxide. The outer diameter of the rings was equal to the face diameter, the inner, unless otherwise specified, was  $\frac{1}{16}$  in. less. The thickness of the rings was  $0.010 \pm 0.0005$  in. Rings of these dimensions will be referred to as standard.

Fig. 1 shows a schematic diagram of the method used for resistance measurements. The output of a regulated constant current power supply was connected to backup blocks, B. The output of the power supply was variable from 3 to  $10^{-4}$  amps with a regulation of better than 0.1%. Lower currents can be drawn, but regulation is poorer. The output was shunted through R. R. could be a standard resistor or voltage divider. The voltage drop was fed into a 10 mv channel of a two channel Leeds and Northrup Speedomax, Type G, recorder. This trace acted as a monitor on the constancy of the output of the power supply. Leads were soldered to the shoulders of the anvils, A. The voltage drop between these two points was the input of the other channel of the recorder. This channel was 5 mv full scale. The current output of the power supply and the voltage drop across the anvil shoulders were used to compute the resistance. The resistance through the anvils in direct contact was in the neighborhood of  $30 \times 10^{-6}$  ohms which, for the resistances quoted in this paper, is a negligible quantity.

Two different methods were used for mounting the metal samples. In the first, used only with bismuth, an 0.008 in diameter wire was mounted normal to the face of the silver chloride disc. A hole to receive the wire was made in the silver chloride with an 0.008 in. pivot drill. The length of the wire was adjusted so that contact was made between the anvils and wires. The position of the wire was measured with respect to the center of the disc with a low power binocular microscope. The position from the center could be determined to two significant figures. Freshly flamed platinum and amalgamated copper contacts were used in some of these experiments, but the results did not differ from those in which the bismuth contacted the anvil faces directly.

In the second method, the metal wire was bent into a circular arc of measured diameter. The wire was mounted between two discs of silver chloride each of which was 0.0035 in. thick. The center of the wire arc was made to coincide with the center of the silver chloride. Contact was made by either platinum or gold plugs 0.020 in. in diameter. These plugs were punched from 0.005 in. foil of the metal. One hole was drilled in each of the silver chloride discs for the contact plug. The discs, wire, and plugs were pressed together by a small hand press to compact the entire assembly. When properly made, a wire was mounted between two chloride discs, one contact plug facing up, the other down. The center of curvature of the wire arc, that of the anvils.

The two materials used in this paper are bismuth and manganese. The former came in pellets, purity greater than 99.9%. It was made into 0.008 in. wire by back extrusion through a die of this diameter. The pellets drew very easily. Our present efforts to make 0.003 in. bismuth wire have not as yet

been successful. The manganin wire was double covered 40 gauge, about 0.003 in. in diameter.

Pressures were generated by placing the anvils in a hydraulic press of 100, 200, or 500 ton capacity. The total load on the anvils was obtained from the ram diameter and the pressure on the oil. The total loads were calibrated by steel load cells which in turn had been compared with Bureau of Standards certified Baldwin test stands.

#### RESULTS AND DISCUSSION

Fig. 2 shows a pressure resistance curve for bismuth as a short wire placed in the center of a  $3/8$  in. anvil. The characteristic increase of resistance below the 1-2 transition is lacking.<sup>6</sup> This is due to takeup in the system. Actually, the anvils are not reliable until a pressure of 25 to 30 Kbars. However, the reliability is sufficient to note the load at which the transition starts. The curve shows the three transitions.<sup>7,8</sup> Because of end effects, this curve cannot be used to determine the relative change of resistance with pressure. The only significant electrical properties are the changes that indicate transitions.

As it was known that the results obtained with the anvils depended on the amount of silver chloride used in an experiment, one of our first investigations concerned the effect of the silver chloride thickness on the loads at which transitions occur. The work was done in  $3/8$  in. anvils using bismuth wire mounted in the center of the silver chloride disk. Three transitions could be followed, the 1-2, 2-3, and 6-8. Of these the 1-2 and 6-8 were quite sharp and easily located. The 2-3 was sluggish and, at times, could not be found. The results are shown in Table I.

Table I  
Effect of Silver Chloride Thickness on Transition Loads  
in Bismuth Transition

Transition	1-2	2-3	6-8
Silver Chloride Thickness in.	Load-Rbars		
0.009	26	29	103
0.0085	26	29	102
0.008	27	30	102
0.007	27	30	106
0.006	34	38	127

It can be seen that as long as the silver chloride thickness was equal to or greater than 0.007 in., the transition load was not effectual within the error of these experiments. The results for the two greatest thicknesses were the results of single determinations. As these thicknesses overloaded the cavity, about 15 attempts at each thickness were required before a successful determination could be made. In all of the later experiments the thickness of the chloride disk was at least 0.007 in.

Another variable that we studied was the face diameter of the anvils. We used three sizes, 1/2, 3/8, and 1/4 in. in diameter. The results of a set of experiments in which a short bismuth wire was mounted in the center of the silver chloride disk are summarized in Table II.



Table II

Effect of Anvil Face Diameter on Bismuth Transitions

Diam. - in.	Transition Kbars	
	1-2	6-8
0.250	23.5	82
0.375	27	100
0.500	35	120

The 1-2 transition was found to occur at loads of 23.5, 27, and 35 Kbars in 1/4, 3/8, and 1/2 in. anvils respectively. This transition is known to occur at 25.5 Kbars. It is one of the best established fixed points.<sup>4</sup> Thus, we know that the pressure of 25.5 Kbars was reached at loads varying from 23.5 to 35 Kbars depending on the anvil size. The variation in the "6"-8 transition was even more striking. This was presumably the 88 Kbar transition found by Bridgman in the piston-cylinder apparatus; throughout the remainder of this paper, the identity is so assumed. These results suggested to us that a radial pressure gradient existed in the silver chloride.

The pressure gradient was established by varying the position of the bismuth wire. Figs. 3 and 4 show the loads required for the 1-2 and "6"-8 transitions in 3/8 in. anvils as a function of the position of the wire. R is the distance from the center of the anvil divided by the radius. These results showed that the pressure was the lowest in the center of the anvil and increased towards the edge. Also, it should be noted that the gradient increased with the total load. However, these data cannot be used to determine the pressure gradient. The method for this determination is the subject of the latter part of this paper. Within the error of these experiments, the

load required for a given transition is a linear function of its distance from the center of the system.

Analogous results were obtained when the anvil face diameter was  $1/4$  and  $1/2$  in. The only difference is that the slope of the line changed markedly with the absolute diameter. For the  $1/2$  in. anvils the 1-2 transition varied from 35 Kbars at  $R = 0$  to 23 Kbars at  $R = 0.84$  while for the upper transition, the corresponding figures are 120 and 82 Kbars. In the  $1/4$  in. anvils, the variation in the 1-2 transition was not determined, the upper was 82 Kbars at the center and 62 Kbars at  $R = 0.7$ .

The pressure gradient can be easily understood in a qualitative fashion. The pressure at the edge of the anvil cannot be greater than the shear strength of the pyrophyllite. That is, at the edge of the anvil face the pressure is of the order of a few Kbars, irrespective of the total load on the anvils. Once the anvils are loaded above the shear strength of the pyrophyllite, the carbide anvil faces deform by a downward motion over the edge of the ring. This leads to the lensing observed and discussed by Bridgman.<sup>1</sup> As the lensing of the faces occurs, the silver chloride, or any other solid, will not follow the change in shape of the faces, at least in the time usually taken for an experiment. The lensing brings about the greatest separation at the center of the face. The internal friction of the chloride and its own tensile properties prevent the chloride from flowing to the center. Thus the pressure will be low at the center of the anvil, increase outward, and then at some point decrease to the very low value at the edge of the face.

This very nicely explains the discrepancy between the transition pressures obtained in the piston-cylinder apparatus and the anvils. Bridgman and later workers loaded the sample in the center of the anvil, where the pressure was lower than the average. Indeed, if the samples had been located at the edge of

the silver chloride, the pressures also would have deviated from the piston-cylinder scale, but in the opposite direction.

The explanation that the internal forces in the solid did not permit it to flow with the distortion of the anvils to obtain uniform pressure suggests that temperature would have a marked influence on the size of the pressure gradient; the higher the temperature, the lower the gradient. Experiments at  $140^{\circ}\text{C}$  confirmed this prediction. The only transition studied was the bismuth 6-8 transition. This transition occurred at 76 Kbars in the center of  $3/8$  in. anvils and at a load of 68 Kbars at  $R = 0.74$ . This difference is about 0.5 of that found at room temperature. Because of the nature of the phase diagram of bismuth<sup>7</sup> a higher temperature could not be used on this solid.

The size of the gradient could also be changed by changing the ring dimensions. In a small number of experiments, the outer dimension and thickness were kept the same, while the inside diameter was reduced to  $1/8$  in. less than the outer diameter. The pressure gradient was greatly decreased. At the Bismuth 1-2 transition there was no gradient in the silver chloride. The results are shown in Fig. 5. These results are to be compared with those in Fig. 3. A pressure gradient was found when the transition was studied at the higher pressures; however, it is considerably less than that shown in Fig. 4 for the "6"-8 transition.

The absence of the pressure gradient in the last series of determinations gave an estimate of the fraction of the ring that was not load bearing. The transition shown occurs at a pressure of 25.5 Kbars, while the average load of transitions in Fig. 5 was 21.8 Kbars. To convert 21.8 to 25.5 Kbars, it was necessary to assume that the "load-bearing" diameter was 0.036 in less than the face diameter. This figure was found to be independent of the face diameter,

of the ratio of the inside to outside diameter of the ring, and of the total load.

This decrease in effective diameter accounted for a number of observations. First, it will be noted that we have listed the 1-2 transition in the  $1/4$  in. anvils at 23 Kbars when the sample was in the center of the face. Since this was the position of lowest pressure, it was apparent that the effective face diameter cannot have been so large as the actual diameter of the face. This was especially true since we constructed an apparatus to make sure that the anvil faces were lined up within 0.001 in.. A second observation in agreement with this conclusion was that when we attempted to measure the bismuth transitions when the wire was set in the standard ring, the wire was always swept out. It was never possible to find the 1-2 transition even when the total load was up to 125 Kbars. The standard rings were 0.032 in. across. Since we would have expected the pressure to increase from its minimum to its maximum value over a region on the order of twice 0.013 in., and the wire is 0.008 in. in diameter, it is not surprising that the wire was swept out. In the rings that were 0.064 in. across, it was possible to determine these transitions when the wire was set in pyrophyllite, provided that there was more than 0.027 in between the edge of the anvil and the center of the wire. When the wire was in this position the 1-2 transition ran from a load of 56 to 72 Kbars, and the "6"-8 transition was found to start at 121 Kbars. From this, it seems reasonable to assume that the pressure rises from its low value at the edge of the anvil to its maximum about 0.030 in from the edge.

So far the existence of the pressure gradient in the silver chloride has been demonstrated. However, no information has been obtained as to the magnitude of this gradient. Because of the design of the anvils we assumed that the pressure gradient was radial. This means that if a wire were to be bent into a circular arc, and its center of curvature made to coincide with the center of

the anvil, and if the diameter of the wire was sufficiently small, we could say that the wire was at constant pressure. Actually, the gradient across the wire was sufficiently small and it may be neglected.

For this type of experiment, the larger diameter anvils were advantageous. Another advantage of this geometry was the long length of wire that can be used. This minimized the effects of contact resistances. Because of its ready availability and because it has been used in the past for pressure gauges<sup>9</sup> we chose manganin wire as the basic conductor for our studies. To obtain circular sections, the wire was wound around an appropriate sized drill shank, then cut to the desired arc length.

The basic properties were studied in  $1/2$  in. anvils with the standard sized rings. The thickness of each of the silver chloride discs was 0.0035 in..

This manner of mounting the samples required an investigation of the effects of this geometry. We were able to show that the results obtained with this mounting were highly reproducible, and to a large extent showed a lack of hysteresis except in the immediate region of a polymorphic transition. This was important since it tended to indicate that the observed values were probably those which would have been obtained at equilibrium. Since the sample is under essential isobaric conditions, the corrections made by Bridgman<sup>1</sup> for the difference in compressibility of the sample and silver chloride need not be considered.

In what follows with respect to the determination of pressure the only assumption that we were forced to make was that the pressure was proportional to the load. It will be shown that this assumption was self checking, if the pressure was not proportional to the load, an analysis of the pressure gradient would have revealed the failure of this tenet.

Fig. 6 illustrates one of the unexpected dividends of this geometry.

In the past, it has been assumed that only the first determination with rising pressure had any validity when anvils were used. In this determination, the manganin wire was  $1/4$  in. in diameter, the anvils  $1/2$  in.. The circles are the results of the first compression, the squares the second on the same sample. The pressure was lowered to 24.7 Kbars, and then the first reading on recompression was made at 34 Kbars. The two determinations agreed to the extreme limit of the recorder. This has been found to be true with other materials.

The above experiment suggested that we study the reversibility of compression and decompression on manganin wire. The results are shown in Fig. 7. The down pressures have been corrected for the 2% hysteresis in the press. The geometry was identical with that of the previous paragraph. There was no detectable hysteresis. Of the few studies made so far with this geometry, hysteresis was found only when a transition occurred.

To show the reproducibility of the technique, reference is made to Fig. 8. To make the comparison between different samples it was assumed that the resistance of each sample was unity at a load of 58.5 Kbars. The two determinations agreed within experimental error.

It was also easy to show that the presence of the wire does not disturb the radial distribution of pressure. This is illustrated in Fig. 9 where the relative resistance is the basis of comparison. For this purpose the resistance was taken as unity at 58.5 Kbars. One of the wires was approximately  $3/4$  of a complete circle, the other  $1/4$  of a circle. Again there was no discrepancy between the two.

The previous has shown that the geometry is a highly reproducible technique. Indeed, the results indicated that the voltage drop across the sample could have been more accurately determined than with the recorder.

Another check on the total method was to vary the diameter of the hoop of manganin wire to find the load at which the bismuth transitions occur at the various diameters. The ratio of the resistance for the 6-8 transition to the 1-2 transition should have been independent of the hoop diameter. This was true within the experimental error of the four determinations needed.

Before discussing this point further, we will attempt to set up a pressure scale that is consistent with all of the data. To do this, we will take one fixed point, the bismuth 1-2 transition. We assume that the resistance of the manganin hoop is unity at this pressure. Further, we must assume the manner in which the resistance of manganin changes with pressure. To hope that it would increase linearly with pressure would be naive. The assumption that we make is that the pressure is proportional to the load. This seems far reaching, but later we will show that it is consistent with all of the data available at the present time. The major implication of the assumption that the pressure is proportional to the load is that if one fixed point is chosen, then the relative resistances as a function of the relative load should be independent of the diameter of the hoop of manganin wire.

The results for four different diameters in  $1/2$  in. anvils are shown in Fig. 10. The uncertainty in the load at which the 1-2 transition occurs was about 1 Kbar; thus, the expected difference in the curves was as high as 4%. The agreement was outstandingly better than this. The maximum difference between the points was less than 3%. The deviation from the average was less. Thus it appeared on this basis that this method gave a pressure scale.

Much better concordance between the four sets of data was obtained if the 6-8 transition was used as the fiducial point.

Several tests of this scale were made. The simplest was the determination of the pressure of the bismuth 6-8 transition. When hoops were made of bismuth wire, the 6-8 transition was found at 88 and 89 Kbars with 1/4 and 3/8 in. diameter hoops respectively. The value given by Bridgman in the piston-cylinder apparatus was 88 Kbars. This agreement was remarkable.

A second test was to use these curves to obtain the pressure gradient in the silver chloride under constant load. If the pressure vs distance from center was correct, then the average observed pressure must be equal to average pressure computed from the total face area and the applied load. The gradient was obtained simply from the original resistance-pressure curves, taking the bismuth 1-2 transition as the fiducial points; several such curves are shown in Fig. 11.

There were some difficulties in drawing the entire curve. The four manganese resistance curves permit drawing the curve from  $R = 0.153$  to  $0.750$ . These points in each case were linear. Not much was lost when the extrapolation of these four points was made to  $R = 0$ . This was incorrect since it places a cusp at the center. A more logical solution would have been to set the derivative to be equal to 0 at the center. The data were too sparse for this. Near the edge there also were difficulties. It was known that the pressure must fall to a small value at the edge. Earlier it was pointed out that about 0.013 in. of the ring could be considered non-load bearing. This was based on a deduction from the bismuth 1-2 transition when there was no pressure gradient in the silver chloride, and second the experiment that no bismuth wire could be held under a pressure approaching the load unless it was over 0.027 in from the edge of



the anvil. Thus we assumed that a reasonable representation of the gradient near the edge was that the pressure rose linearly to within 0.030 in. of the edge of the anvil, then fell linearly to 5 Kbars at the edge. This certainly oversimplified a complex situation.

The weakest point in the above description is that of the behavior near the edge. However, since this region contributed only a relatively small part to the total, a relatively large error could be made without obviously affecting the final result. The area under the pressure distribution curve where the pressure starts to fall to its low value at the edge was only about 3% of the total area. Thus a 100% error in its estimation could lead to an error of only 3%. The average pressure obtained from the curves in Fig. 11 agreed to within 1% of the computed average pressure from the load. Thus when the applied load is 117 Kbars, the average under the curve gave 116 Kbars.

All of the previous results can be collated simply (in 1/2 in. diameter anvils) by the following,

$$P = (0.725 + 0.468 R) L \quad (1)$$

where  $P$  is the pressure,  $L$  the load computed in the standard manner, and  $R$  the distance of the sample from the center of the anvil divided by the anvil radius. This formula is applicable only when the dimensions of the specimen are so small that the pressure gradient across it is negligible. Bridgman's setup was such that it is not possible to compare results directly. His samples were about 0.18 in. long, and the anvil faces were 1/2 in. in diameter. This would mean that the transition pressures he observed should be multiplied by some number between 0.725 and 0.893. The method of obtaining the correct transition pressure with this geometry is complex, and will be the subject of a forthcoming paper. However, Table III gives a comparison

Table III

	Bridgman Piston-Cylinder	Anvils	Calculated from Eq. (1)
Ba	60	80	58-71.5
Tl	40	45	33-40
Te	45	54	39-48
Cs	45	54	39-48

of pressures observed by Bridgman in the piston-cylinder and anvils. The third column gives upper and lower bounds for the resistance as calculated by Eq. (1).

More precise values for cesium and thallium are 42 and 37 Kbars respectively.<sup>4</sup> If these values are used for comparative purposes, then the corrected values are about at the midpoint of the calculated limits except for barium. The midpoint value for barium is high by about 7% from the quoted value.

It should be emphasized at this point that this correction is valid only with what we have called our standard ring dimensions. Preliminary work with the  $3/8$  and  $1/4$  in. anvils is in agreement with the results of the  $1/2$  in. anvils. The bismuth 6-8 transition occurs at 86 and 90 Kbars in the  $3/8$  and  $1/4$  in. anvils when the resistance change is used as the criteria. The average of all of our results for the bismuth 6-8 transition is 88 Kbars.

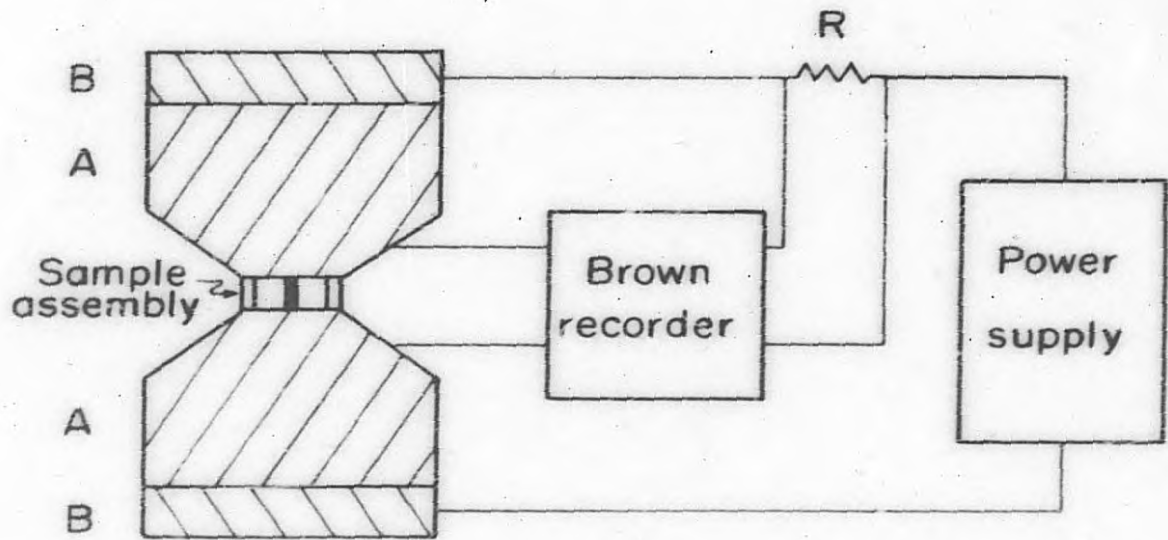
One surprising feature of the present work is the magnitude of the pressure gradient that exists in the silver chloride disk. When the average load is 117 Kbars, the gradient is of the order 45 Kbars. Actually, Eq. (1) yields a gradient that is 44% of the load. These figures are valid only for this particular geometry. Our present work is extending this same treatment to the  $3/8$  and  $1/4$  in. anvils, and different ring thicknesses.

Legends

- Fig. 1. Schematic diagram of instrumentation for resistance measurement.
- Fig. 2. Load-resistance curve of short bismuth wire mounted in the center of silver chloride in  $3/8$  in. anvils.
- Fig. 3. Load at which bismuth 1-2 transition occurs of  $3/8$  in. diameter anvils.
- Fig. 4. Load at which bismuth 6-8 transition occurs of  $3/8$  in. anvils.
- Fig. 5. Load at which 1-2 transition occurs in  $1/2$  in. anvils when the inner ring diameter is  $3/8$  in.
- Fig. 6. The reproducibility on recompression of the same sample of manganin wire bent to the shape of hoops.
- Fig. 7. Shows the lack of hysteresis on compression and decompression of manganin wire hoops.
- Fig. 8. The reproducibility of the pressure coefficient of resistance of different manganin wire hoops.
- Fig. 9. The relative resistance of manganin hoops of different arc lengths as a function of pressure.
- Fig. 10. The relative resistance of manganin wire hoops as a function of hoop diameter. Unity is taken at the bismuth 1-2 transition.  $1/2$  in. anvils.
- Fig. 11. The pressure gradient in silver chloride at various loads.  $1/2$  in. diameter anvils.
- Fig. 12. Pressure resistance curve of bismuth hoop. The hoop was 0.002 in. thick and 0.022 in. across. There is a pressure gradient across the sample.

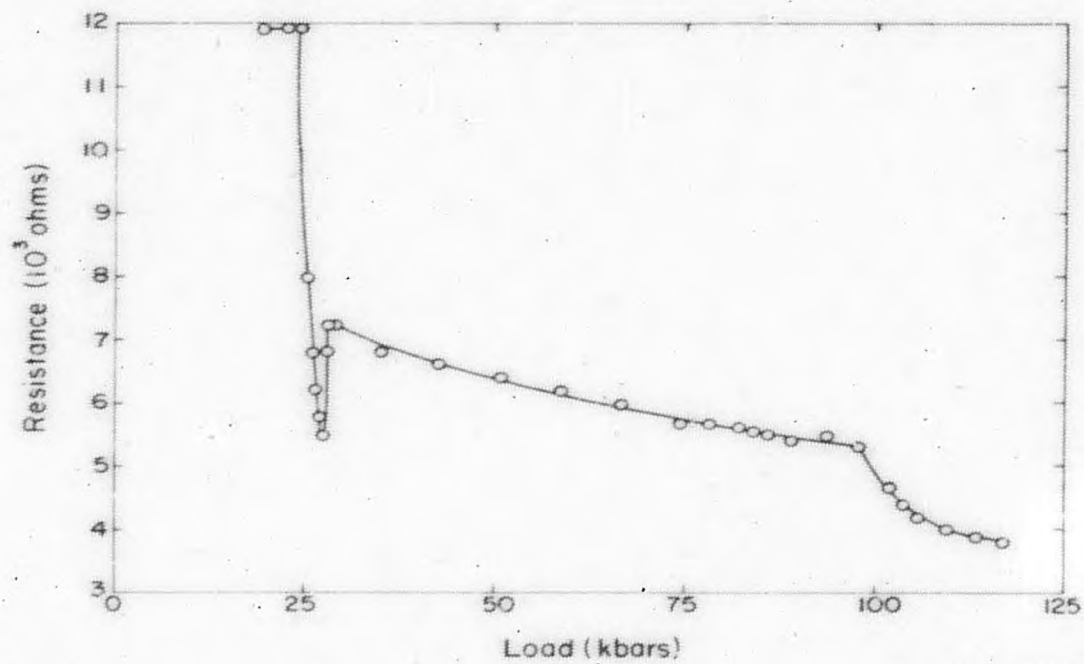
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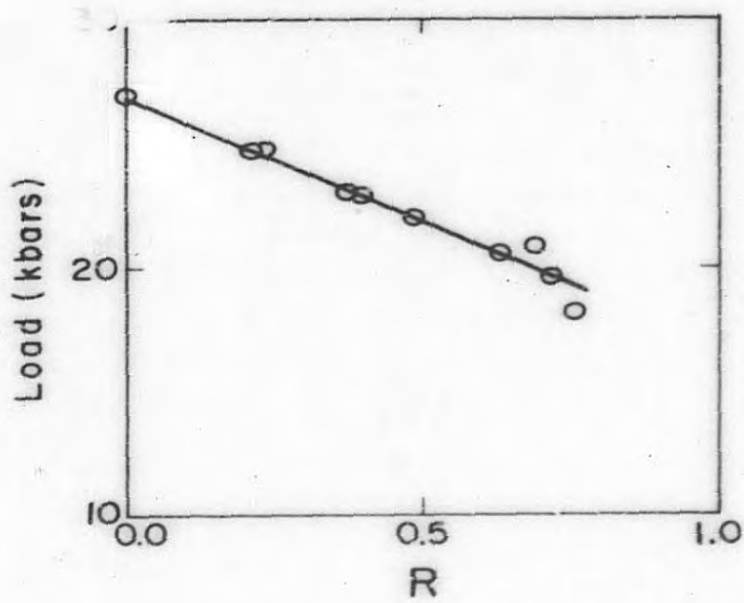
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Fig. 1



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Fig. 2



MU24480

Fig. 3

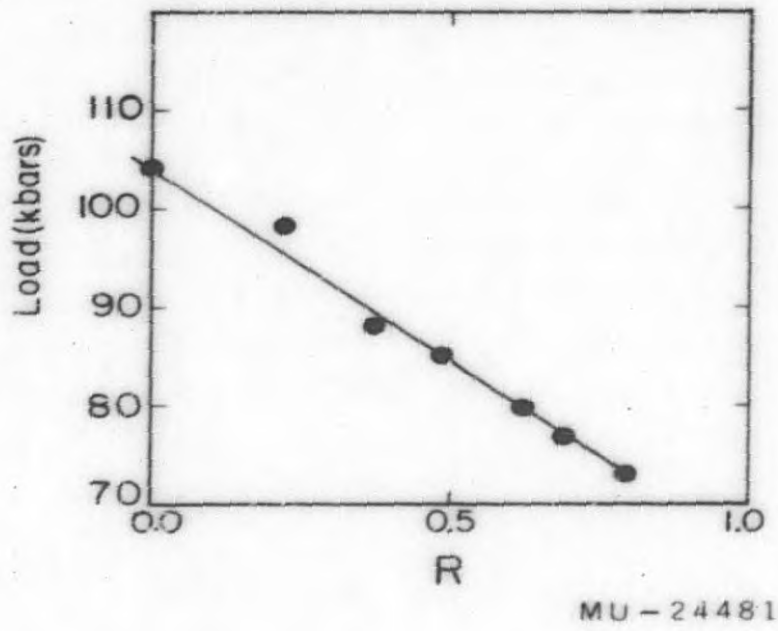
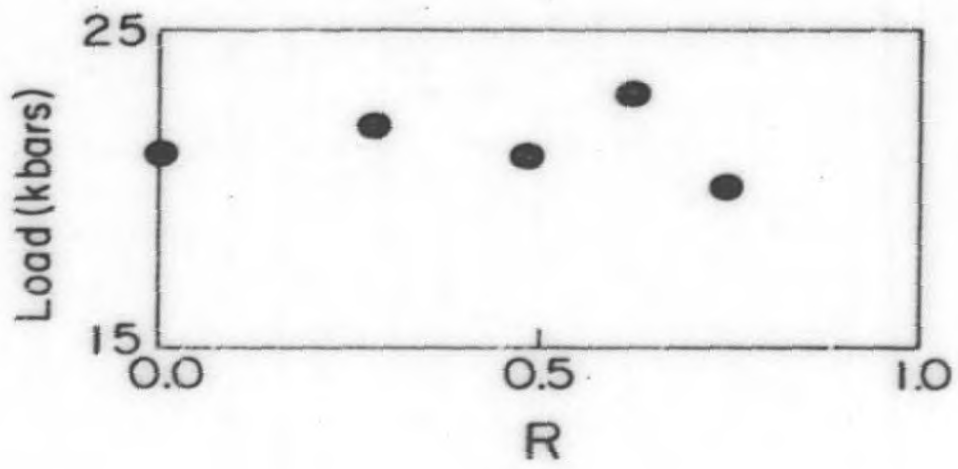


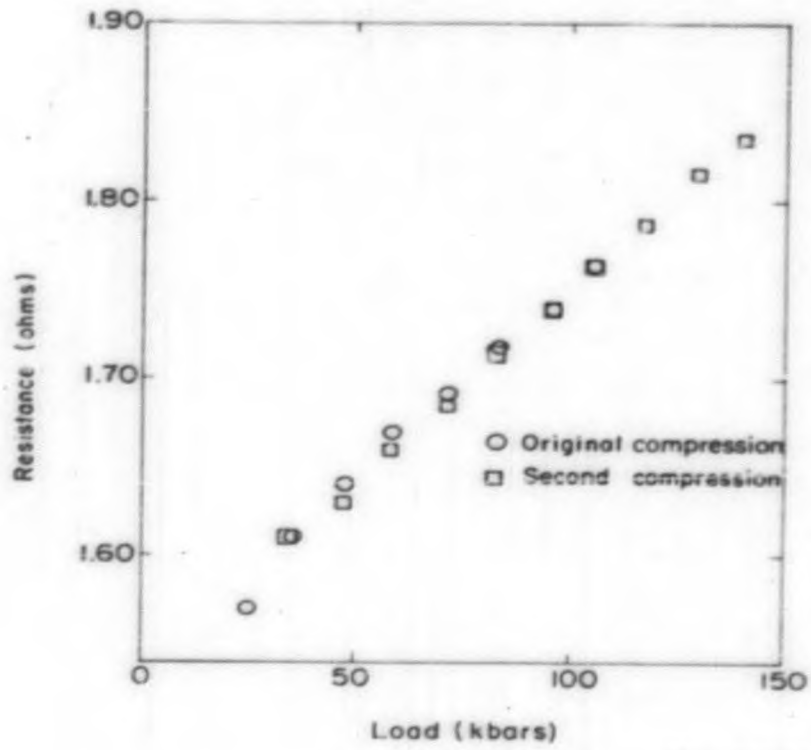
Fig. 4





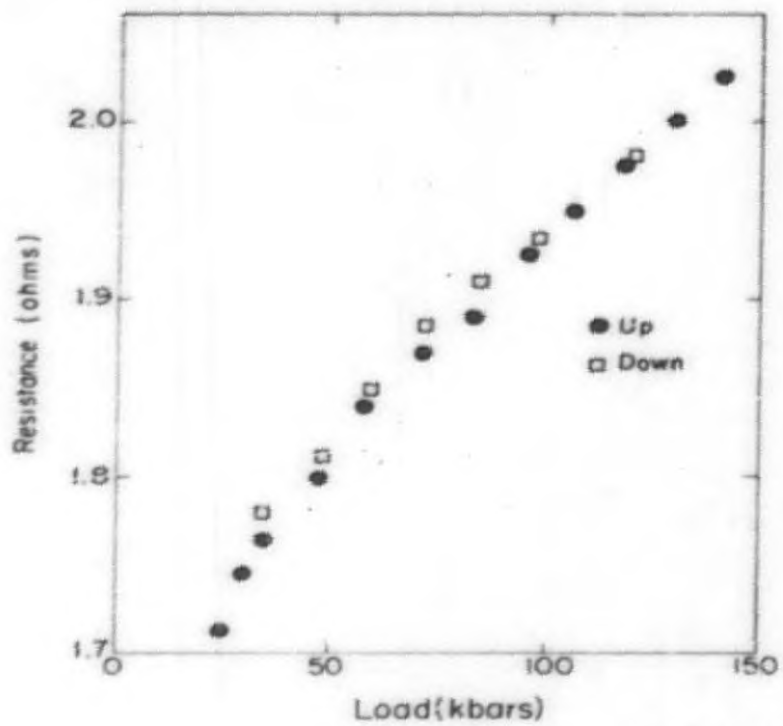
MU - 24482

Fig. 5



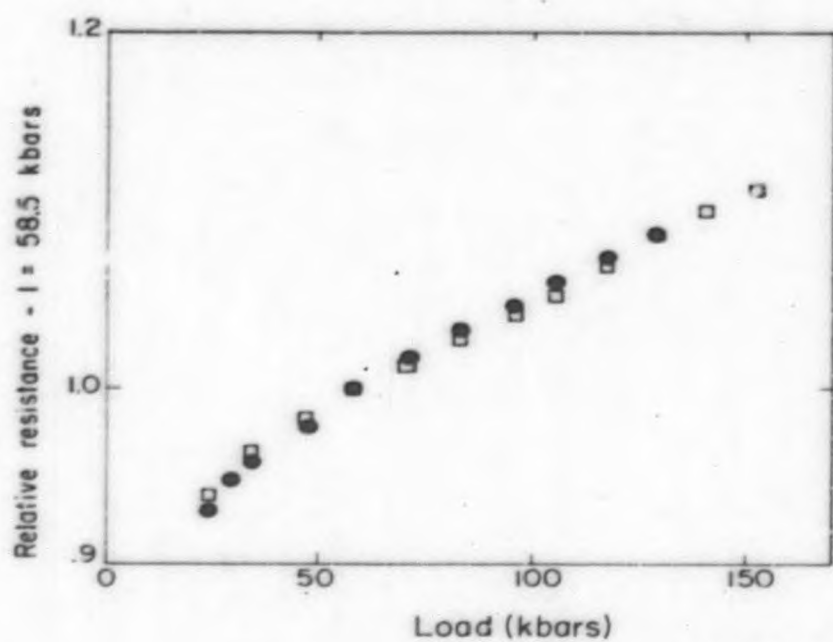
MU-24483

Fig. 6



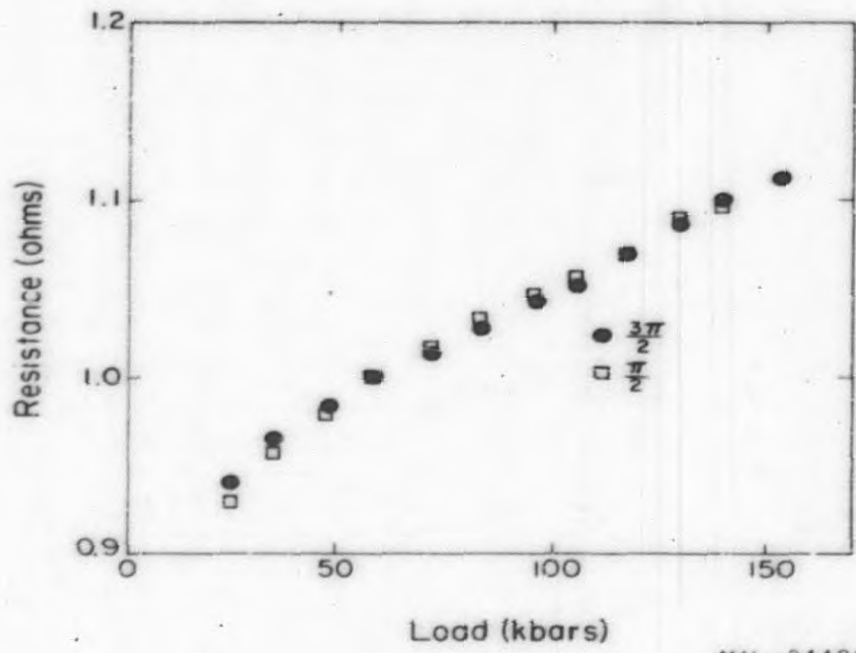
MU - 24484

Fig. 7



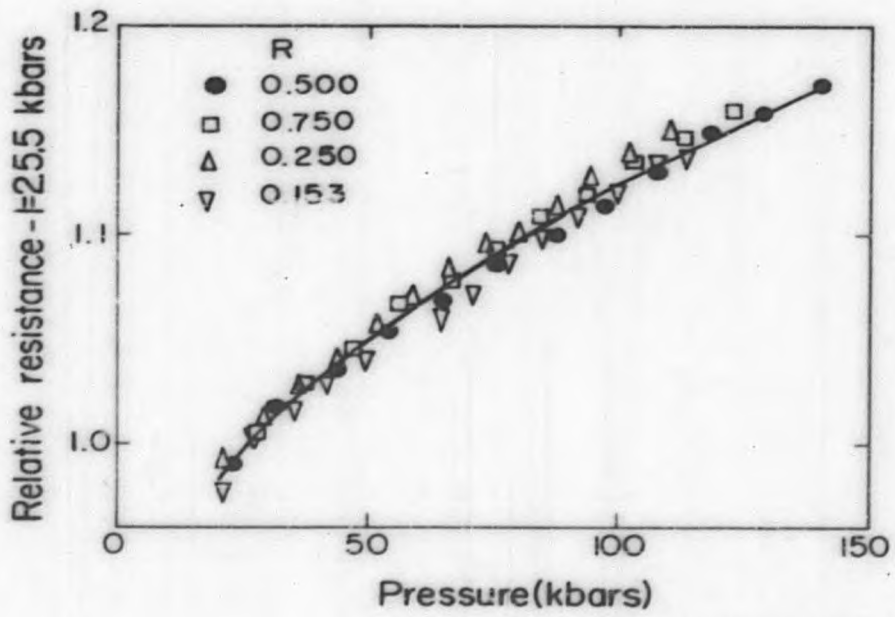
MU - 24485

Fig. 8



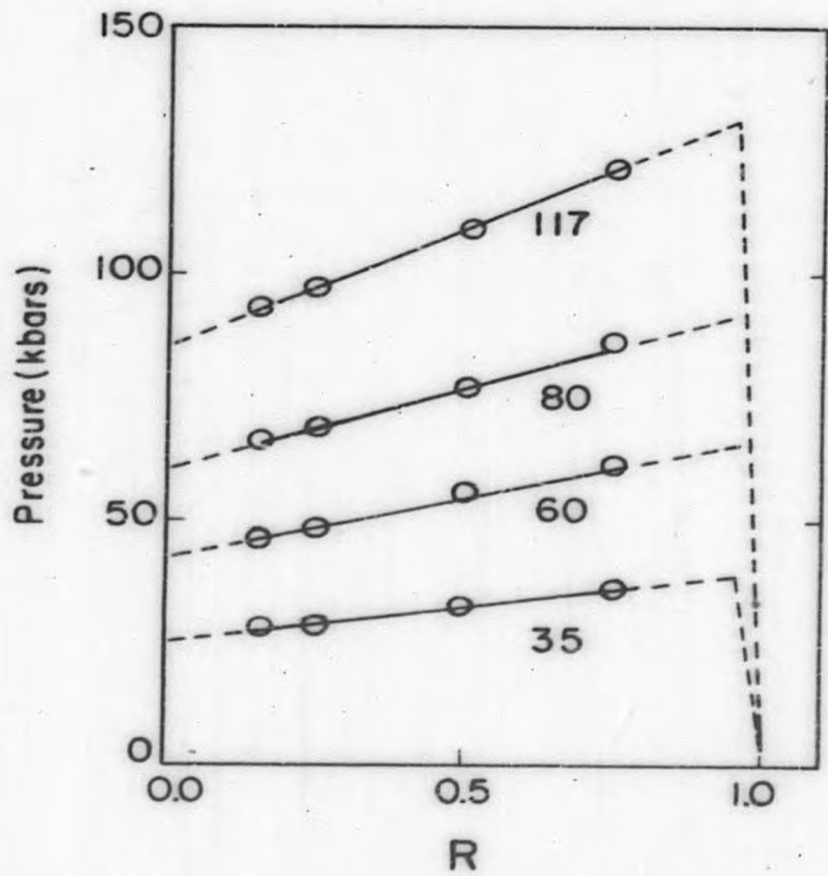
MU-24486

Fig. 9



MU-24487

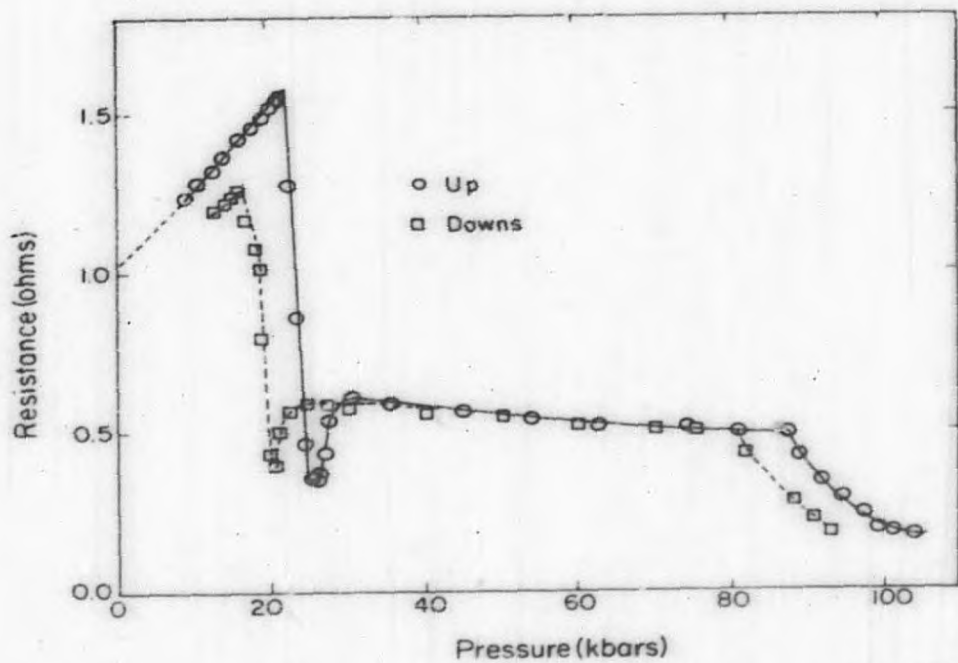
Fig. 10



MU-24488

Fig. 11





MU-24489

Fig. 12.

**END**