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# Ultra-High Pressure Calibration: Influence of Cubic Workpiece Configuration

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This paper reports on two investigations conducted on a large size cubic unit. The first investigation confirms the advantages of large size specimens by disclosing certain phenomena which have not been observed on smaller size units. The second investigation indicates that the efficiency of pressure transmission in a cubic unit depends on the geometry of the specimen assembly. The optimum geometry varies with the pressure level. The investigation reports on general results established experimentally. Since the apparatus and instrumentation were of substantial importance, their description is included in the report.



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# Ultra-High Pressure Calibration: Influence of Cubic Workpiece Configuration

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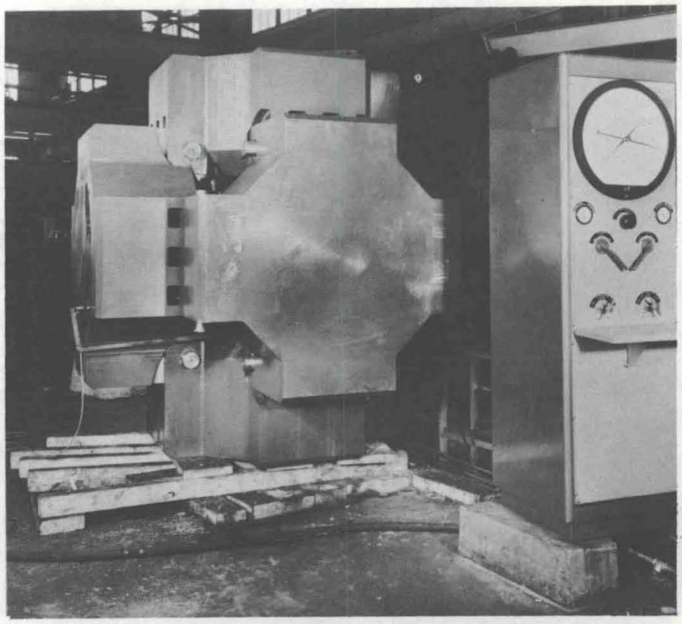

Fig. 1 2700/16, 200-ton cubic hinge unit and operating console

NOMENCLATURE

$L_a$ = length of edge of square anvil tip, in.

$L_w$ = length of edge of square face of specimen assembly, in.

L = length of resistance wire, in.

D = diameter of silver-chloride insert, in.

P = pressure in primary system of machine, Kilobars (kb)

m = magnification factor; ratio between area of primary ram and area of anvil tip

for La = 1.46 m = 196

for La = 1.35 m = 227.5

Pm = p x m

$P_r$ = transition pressure

INTRODUCTION

The use of phase transitions for the calibration of ultra high pressure apparatus was suggested by Bridgman (1,2,3).[1] Further contributions to this problem were made by Bundy (4,5) Vereshchagin et al (11,12) Kennedy and LaMori (13) and Boyd and England (10). All cited investigations were conducted either with the assistance of Bridgman anvils or on cylinder-plunger type apparatus of various design. Hall (6,7) has suggested the use of multiaxial arrangements. A review of various multiaxial arrangements will be found in reference (9).

The testing and calibration of one particularly large cubic unit offered the opportunity to conduct two investigations which are the subject of this paper:

1 The purpose of the first investigation was to determine whether the large size of the specimen offered any increase in observability of ultra high-pressure phenomena.

2 The second investigation concerned the determination of the influence of geometric relations between the workpiece assembly and the anvils of the apparatus on the efficiency of pressure transmission.

The tests were conducted by Messrs. J. Brayman[2] and M. Gabey,[3] under the general supervision of Dr. G. Gerard[4] (8).

APPARATUS

The machine used in the investigations is shown in Fig.1. It consists of six heads or platens connected with each other by means of articulating links. Each head contains a hydraulic system consisting of a cylinder (integral bore within the forged head) and a ram. The total hydraulic force developed by the ram is concentrated by means of a tool assembly attached to each ram

[1] Numbers in parentheses designate References at the end of the paper.

[2] Senior Development Engineer, Barogenics, Inc.

[3] Chief of Field Services, Barogenics, Inc.

[4] Vice-President, Barogenics, Inc.; now Director of Engineering Sciences, Allied Research Associates.

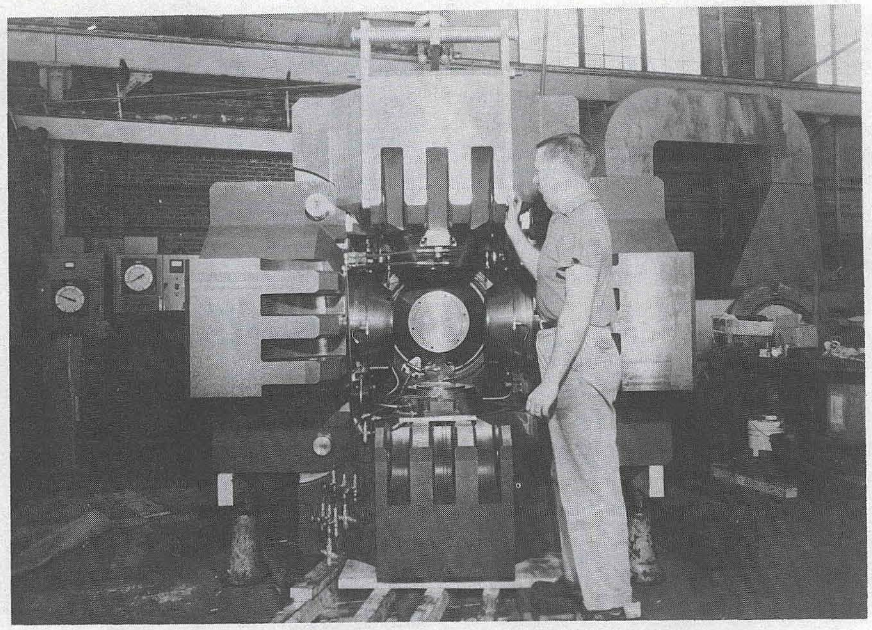

Fig. 2 2700/16, 200-ton cubic hinge unit

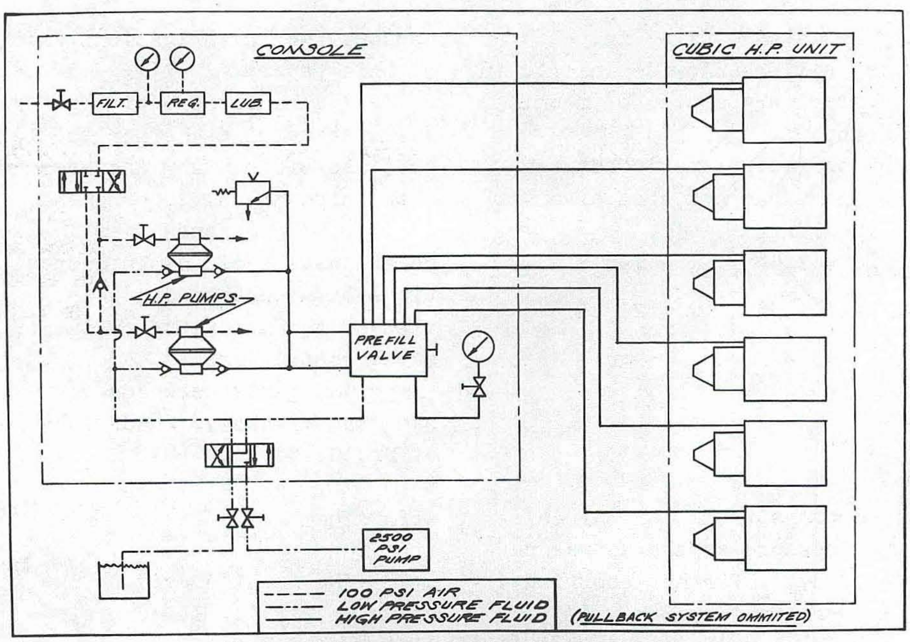


Fig. 3 High pressure hydraulic schematic

on a comparatively small face of a tungsten-carbide anvil.

The six carbide anvils press upon the six faces of a cubic specimen assembly. Fig.2 shows the machine with the front platen removed. The hydraulic circuit is shown in Fig.3. Pump 1 provides the prefilling of the system up to a pressure of 2500 psi, pumps 2 and 2a are used to raise the pressure up to the maximum of 13,000 psi. Each ram is rated at 2700 tons at a primary pressure of 13,000 psi.

The special design of the hydraulic seal, Fig.4, reduces the friction to an amount which can be considered negligible within the accuracy of measurements reported here.

Anvils

Tungsten-carbide anvils used in the investi-

Table 1 Parameters of Test Assemblies

a)

| Design | La (in.) | Lw (in.) | Lw/La | Used for Transitions | | | | |
|---|---|---|---|---|---|---|---|---|
| Fig. 6 (straight wire) | 1.46 | 1.61 | 1.10 | Bi I-II | Bi II-III | - | - | - |
| | | 1.75 | 1.20 | " | " | Th II-III | - | - |
| | | 1.82 | 1.25 | " | " | " | - | - |
| | | 1.90 | 1.30 | " | " | " | Ba II-III | - |
| | | 1.97 | 1.35 | " | " | " | " | - |
| | | 1.98 | 1.36 | - | - | " | " | - |
| | | 2.27 | 1.54 | - | - | " | " | - |
| | | 2.50 | 1.71 | - | - | - | " | - |
| | 1.35 | 2.30 | 1.71 | " | " | - | - | Bi V-VI |
| | | 2.50 | 1.85 | " | " | - | - | " |
| Fig. 7 (coiled wire) | 1.35 | 2.50 | 1.85 | " | " | - | - | " |

b)

| La (in.) | Lw/La | L (in.) | La/L | D (in.) | La/D |
|---|---|---|---|---|---|
| 1.46 | | .615 | 2.4 | .25 | 6 |
| 1.35 | 1.71 | .675 | 2 | .675 | 2 |
| 1.35 | 1.85 | . " | 2 | .56 | 2.4 |
| 1.35 | 1.85 | 1.95 | .75 | " | " |

La - Edge of Anvil Tip

Lw - Edge of Specimen Assembly

L - Length of resistance wire

D - AgCl Diameter

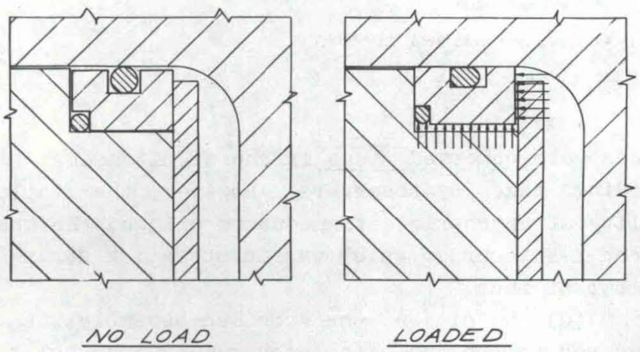


Fig. 4 Deflection compensating seal

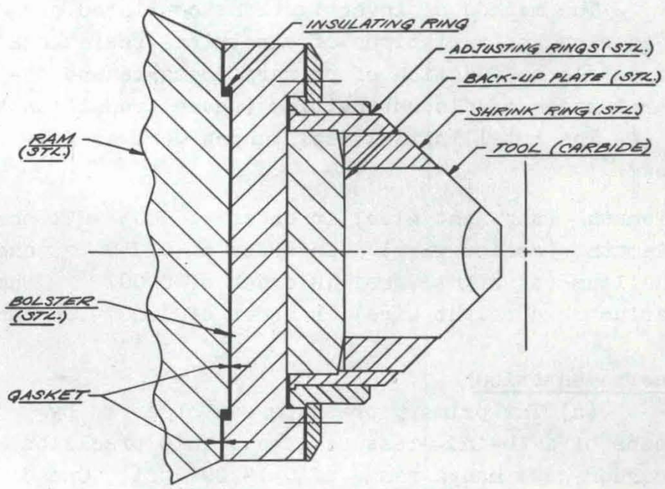


Fig. 5 Anvil assembly

gation are shown in Fig.5. The high-pressure tip is a square. Two sizes were used in this investigation: a square tip with 1.46 in. edge length (2.14 in.$^2$) and a square with 1.35 in. edge length (1.82 in.$^2$).

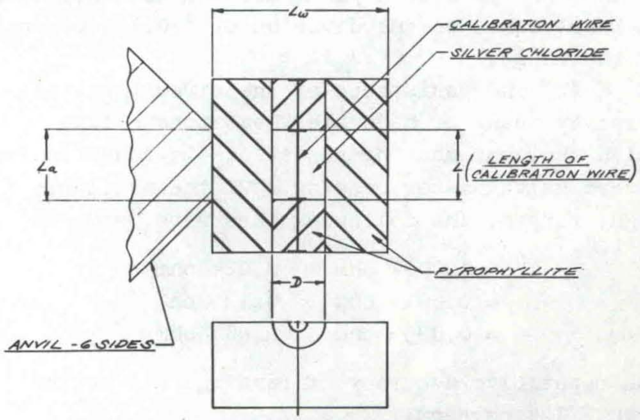


Fig. 6 Conventional sample assembly

Specimen Assemblies

These are shown in Figs.6 and 7. They consist of a cubic outer layer of pyrophyllite, a cylindrical insert of silver chloride and a sensitive-element assembly. The sensitive-element assembly, Fig.6, consists mainly of a straight piece of wire while Fig.7 shows a silver-chloride cylinder with a wire wound on it in the form of a spiral. The contacts between the sensitive element and the outer portion of the electric circuit are provided in a conventional manner by means of tabs abutting against the tips.

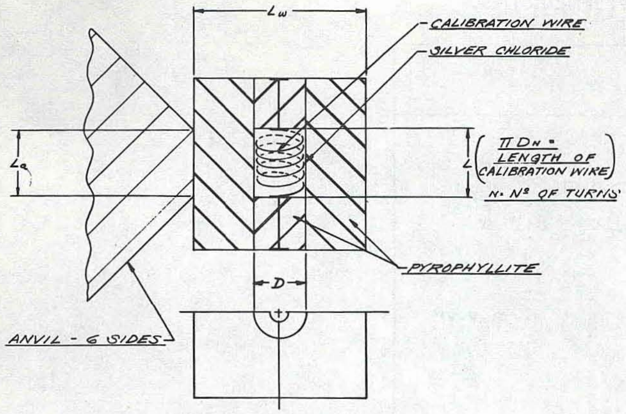


Fig. 7 Coiled sample assembly

Table 2

| Pressure Range | Residual Lw, in. | Residual Volume, cu in. |
|---|---|---|
| Tl II-III and lower........ | 1.75 | 5.4 |
| Ba II-III and Bi V-VI........... | 1.71 | 5.0 |

Table 3 Volumetric Transitions of Bismuth

| Transition | Pressure* kg/cm2 | kb** | $\frac{\Delta V}{Vo}$ ** |
|---|---|---|---|
| I-II | 25,300 | 24.8 | 4.60% |
| II-III | 27,000 | 26.5 | 2.95% |
| III-IV | 44,800 | 44.0 | .6% |
| IV-V | 65,000 | 63.8 | .5% |
| V-VI | 89,800 | 88.1 | 1.2% |

* Bridgman data

** Computed from Bridgman data.

Table 1 indicates the construction and the dimensions of specimen assemblies used in various test runs covered in this paper.

## METHOD AND INSTRUMENTATION

### Method

The method of investigations consisted of measuring the variations of electrical resistance of metals as function of primary pressure and determination of discontinuities (phase transitions).

The total initial resistances were as follows:

| | | | |
|---|---|---|---|
| Bismuth | (straight wire) | in order of 0.05 -0.1 | ohm |
| Bismuth | (coiled wire) | in order of 0.250 | ohm |
| Thallium | (straight wire) | in order of 0.002 | ohm |
| Barium | (straight wire) | in order of 0.007-.04 | ohm |

### Instrumentation

(a) The primary pressure was observed by means of a 16-in. pressure gage. This precision Bourdon gage had a range of 0-15,000 psi. One division of the scale was equivalent to 10 psi. The gage was equipped with a knife-edge pointer so that parallax was eliminated. Careful calibration in the manufacturer's plant assured accuracy being at least equal to one division or ± 0.133 percent of the range.

(b) The resistance of the circuit was measured by means of a double Wheatstone bridge in which the compensating resistance is adjusted for a zero galvanometer reading. Of the available eight ranges, the following ones were used:

| | |
|---|---|
| $1^o$ = 0.0002 ohm | 0.024 ohm |
| = 0.001 ohm | 0.12 ohm |
| = 0.005 ohm | 0.60 ohm |

The repetitive accuracy of readings was better than ± 1/4 percent.

(c) In general, the pressure and resistance gages were observed visually and simultaneous readings taken by observers. However, the availability of appropriate transducers allowed the use of an X-Y recorder which was in operation during a number of runs.

(d) The size of the specimen assembly was measured before and after each run by means of a micrometer. It is interesting to note that for a given structure of the specimen assembly the residual size (after being subjected to pressure) was primarily the function of the anvil size. It depended to a small degree only on the initial size of the specimen assembly.

For instance, for La = 1.35 the residual length Lw is given in Table 2.

## FIRST INVESTIGATION (BISMUTH DISCONTINUITIES)

Bridgman observed five volumetric transitions of bismuth shown in Table 3. Previous electrical measurements by Bridgman and Bundy disclosed only three discontinuities. Of these, the first two occurred at pressures identical with those for the first two volumetric transitions, while the last one occurred at pressures in the neighborhood of 125 kb.

Kennedy and LaMori pointed out that the high electrical bismuth transition probably corresponds

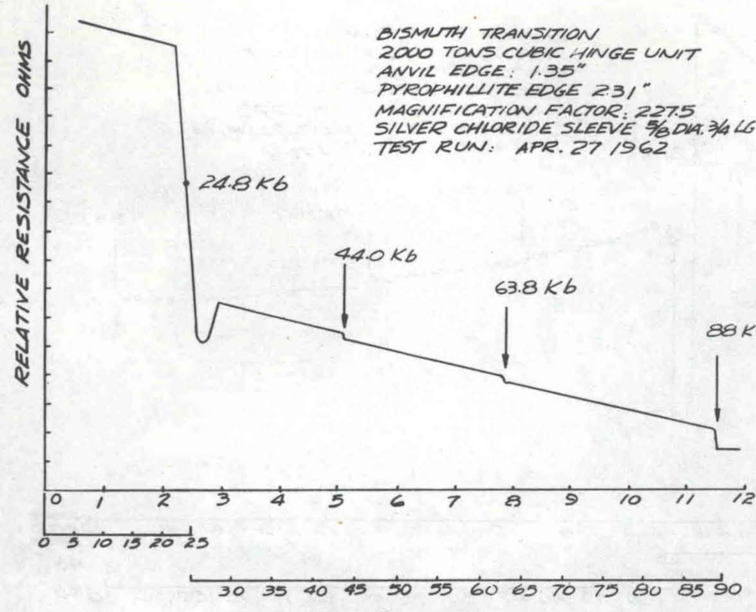


Fig. 8 Bismuth transition curve

Table 4 Bismuth Transitions

**Data used in the determination of Bismuth Transitions.**

| Transition | Reference Pressure kb | | Pm* during experimental runs — Experimental Runs kb | | |
|---|---|---|---|---|---|
| | Volumetric (Bridgman)(2) | Resistance (Bundy)(4) | I | II | III |
| I-II | 24.8 | 25.4 | 36.8 | 43.4 | 37.7 |
| II-III | 26.5 | 27.9 | 42.2 | 47.9 | 42.5 |
| III-IV | 44.0 | -- | 82.0 | 82.4 | 79.4 |
| IV-V | 63.8 | -- | 119.2 | 119.5 | 117.0 |
| V-VI | 88.1 | | 176 | 176.0 | 180.0 |
| VI-VIII | | 89.0 | | | |

* Pm = p x m - where

p = primary pressure in kb

m = magnification factor = $\frac{\text{area of main ram}}{\text{area of anvil tip}}$

Table 5 Volume and Resistance Changes for Bismuth Transitions

| | Pressure kb (Bridgman) | $\frac{V}{V_o}$ * | $\frac{R}{R_o}$ |
|---|---|---|---|
| I-II | 24.8 | 4.60% | 75-88% |
| II-III | 26.5 | 2.95% | 12-25% |
| III-IV | 44 | .6% | 1-3% |
| IV-V | 63.8 | .5% | 1-2% |
| V-VI | 88 | 1.2% | 4-6% |

* Calculated from Bridgman data.

to the volumetric transition designated by Bridgman as V-VI and that the difference in observed pressures is probably caused by substantial friction losses occurring only during electrical runs and not occurring during the measuring of volumetric changes.

Accepting Kennedy's explanation, Bundy found three of the volumetric transitions accompanied by discontinuities in electrical resistivity,[5] while no such correlation was observed for the volumetric transitions Bi III-IV and Bi IV-V.

In analyzing this apparent discrepancy, we have come to the conclusion that, as Bridgman surmised, the reasons for it lie in the comparatively small volumetric and electrical changes accompanying these transitions. The total initial resistance of specimens used by Bundy were only on the order of 0.060 ohm. One per cent of change in resistivity would amount to less than one milliohm and, most probably, be too small to be observed with the instrumentation used in those investigations.

The available cubic machine offered the opportunity to use rather large specimens with a resistance substantially higher than that used in

[5] After the correction described in test is applied, Bundy's VI-VIII electrical transition becomes identical with Bridgman's volumetric V-VI.

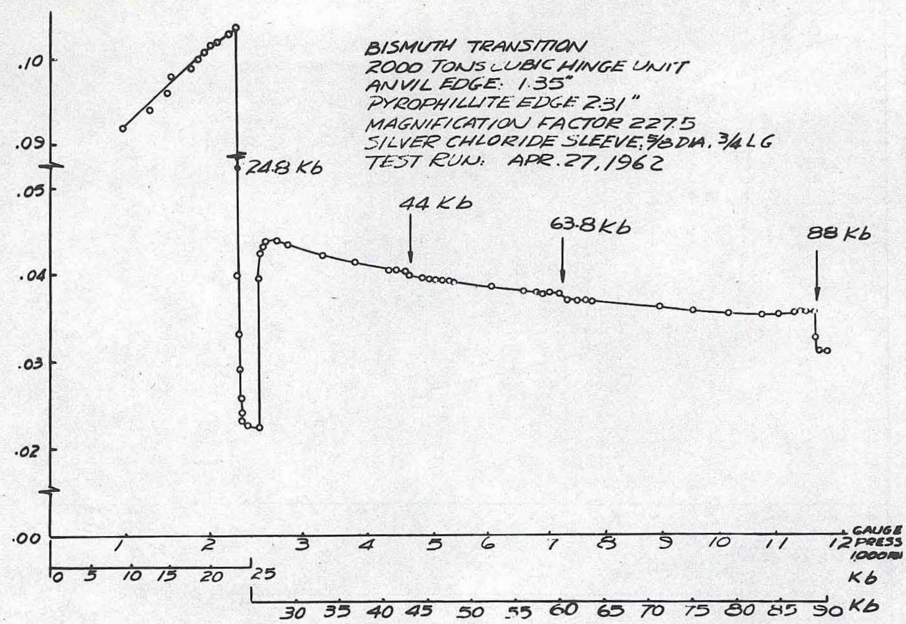

Fig. 9 Bismuth transition curve

previous investigations. Table 4 provides a summary of runs considered here.

The next two figures show resistances of bismuth specimens as functions of pressure at room temperatures. Fig.8 shows a run monitored on the X-Y recorder. The total initial resistance of the bismuth wire (actually of the entire circuit) was approximately 0.25 ohm. In addition to the expected discontinuities I-II, II-III and V-VI, the curve shows two more discontinuities corresponding to transitions III-IV and IV-V. The upper pressure scale corresponds to the indications on the primary pressure gage. The pressure scale at the bottom is a composite scale based on the assumption that the middle point of the I-II transition corresponds to 24.8 kb and that the high transition occurs at 88 kb. Each of the two intervals 0-25 kb and 25-90 kb is subdivided on a linear basis.

Fig.9 shows a similar run monitored by direct readings on the pressure gage and the Wheatstone bridge. It shows again the pressure of discontinuities corresponding to transitions III-IV and IV-V.

Table 5 presents a comparison of Bridgman's volumetric changes with resistance changes as observed by us.[6] The rather wide spread of our figures for $\Delta R/R_0$ as observed during various runs is due, probably, to the fact that the bismuth material may have been slightly contaminated by impurities.

Fig.10 is a correlation between Bridgman's pressures of volumetric transitions and our measurements of electric discontinuities. The III-IV transition is off the straight correlation by about 3 kb. Following a suggestion made in reference (4), the dotted line represents an alternative (curved) calibration line. Of course, these calibration lines apply only to specific dimensions of anvils and specimen assemblies.

Table 6 Transition calibration Values

| | | kg* |
|---|---|---|
| Bismuth | I-II | 25.4 |
| Bismuth | II-III | 27.9 |
| Thallium | II-III | 37.0 |
| Barium | II-III | 59.0 |
| Bismuth | V-VI | 89. |
| | (Bridgman)<br>( VI-VIII)<br>( Bundy ) | |

* Data of Bundy.

SECOND INVESTIGATION:
OPTIMIZATION OF GEOMETRIC RELATIONS

Purpose. The purpose of the second investigation was to explore the relations between the

[6] In addition to runs shown in Table 4, data available on other runs were used in preparing Table 5.

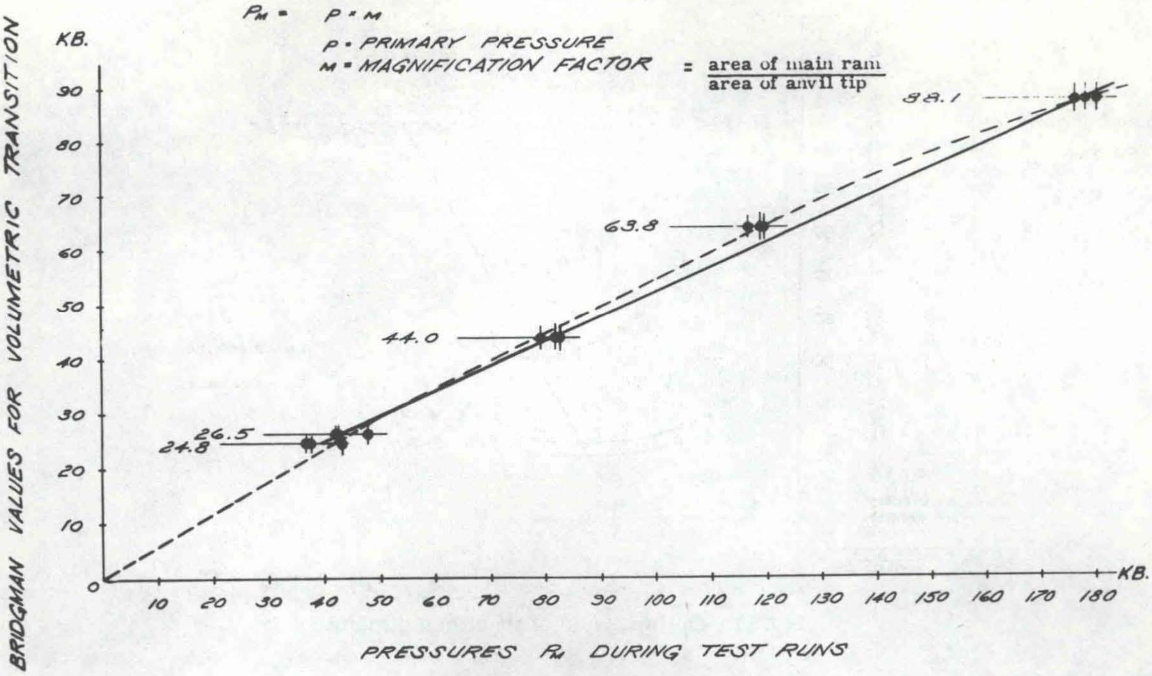


Fig. 10 Calibration curve

Table 7 Transition Parameters

| Transition | La (in.) | Lw/La | La/D | La/L | Pm/kb | $P_r$/kb | Pm/$P_r$ |
|---|---|---|---|---|---|---|---|
| Bismuth I-II | 1.46 | 1.10 | 6 | 2.4 | 33.5 | 25.4 | 1.32 |
| | | 1.20 | | | 28.1 | | 1.11 |
| | | 1.25 | | | 28.1 | | 1.11 |
| | | 1.30 | | | 28.5 | | 1.12 |
| | | 1.35 | | | 31.2 | | 1.23 |
| Bismuth II-III | 1.46 | 1.10 | 6 | 2.4 | 37.2 | 27.9 | 1.33 |
| | | 1.20 | | | 31.3 | | 1.12 |
| | | 1.25 | | | 31.3 | | 1.12 |
| | | 1.30 | | | 31.6 | | 1.13 |
| | | 1.35 | | | 37.6 | | 1.35 |
| Thallium II-III | 1.46 | 1.20 | 6 | 2.4 | 57.3 | 37. | 1.55 |
| | | 1.25 | | | 53.1 | | 1.44 |
| | | 1.30 | | | 50.5 | | 1.35 |
| | | 1.35 | | | 54.0 | | 1.46 |
| Barium II-III | 1.46 | 1.30 | 6 | 2.4 | 128. | 59. | 2.17 |
| | | 1.36 | 5 | 2.4 | 111. | | 1.98 |

La - Anvil edge

Lw - Pryophyllite edge

D - Diameter of silver chloride insert.

L - Length of resistance wire

Pm = p x m

p - primary pressure

m = ram area / anvil tip area

$P_r$ = Transition pressure (Bundy).

dimensions La, Lw and D, Fig.6, in order to determine the influence of these dimensions on the primary pressures corresponding to various transitions.

Program. The investigation was subdivided into two parts. The first part concerned itself with the outer dimensions of the pyrophyllite. In it, the anvil dimension La was kept constant with 1.46; specimen assembly dimensions L and D also were kept constant; dimension Lw was varied. The observed pressures were evaluated as functions of the parameter Lw/La.

The second part concerned itself with the diameter of the silver-chloride insert. Dimensions Lw, L, and D were varied and the parameters La/D, La/L, and Lw/La were calculated. The optim-

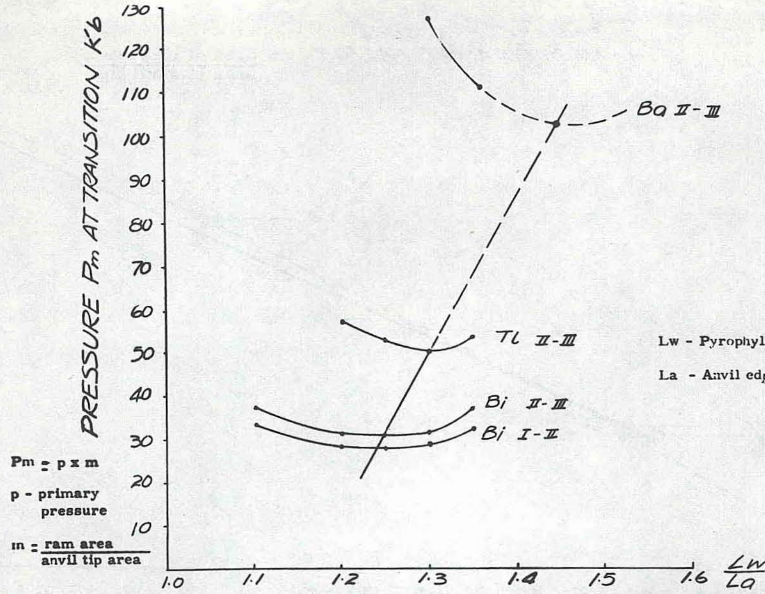


Fig. 11 Optimization of specimen dimensions

al values of these parameters in terms of lowest transition pressures were searched for.

Additional data were obtained from test runs made for other purposes. (These data are of a limited use only because they do not fully match the pattern of the evaluation setup.)

Pressure Calibration. The values of electrical transitions cited by Bundy (partly suggested by Kennedy and LaMori) were used as calibration points for the primary pressures in this portion of the investigation Table 6. In this investigation the midpoint of the transition was assumed to correspond to the cited pressure values.

Influence of Pyrophyllite Size. The resume of measurements is shown in Table 7. A review of the data presented in this table shows that an optimum ratio Lw/La exists, but that the optimal values are different for each pressure level. In general, the optimal value of Lw/La gets larger as the pressure level increases.

Fig.11 shows the results of the investigation in graphic terms which confirm the conclusions stated in the preceding paragraph.

Influence of Size of Silver-Chloride Insert. High friction between the specimen assembly and the anvils is desirable in order to limit the gasket extrusion. However, within the assembly, low friction is preferable so as to reduce the transmission losses. Since the silver chloride has a lower internal coefficient of friction than pyrophyllite, it stands to reason that replacing a larger portion of pyrophyllite by silver chloride would be beneficial in terms of covering the transition pressures.

Table 8 Transition Parameters

| | La in. | Lw/La | La/D | La/L | Lw/D | Pm kb | $P_r$ (Bundy) | $Pm/P_r$ |
|---|---|---|---|---|---|---|---|---|
| Tl II-III | 1.46 | 1.30 | 6 | 2.4 | 7.8 | 50.5 | 37 | 1.36 |
| | | 1.30 | 2 | 2.0 | 2.6 | 50.7 | | 1.37 |
| | | 1.30 | 2 | 2.0 | 1.95 | 50.1 | | 1.35 |
| Ba II-III | 1.46 | 1.30 | 6 | 2.4 | 7.8 | 128. | 59 | 2.17 |
| | | 1.36 | 5 | 2.4 | 6.8 | 117. | | 1.98 |
| | | 1.36 | 1.5 | 2. | 2.04 | 111. | | 1.88 |
| | | 1.30 | 1.5 | 2. | 1.95 | 111. | | 1.88 |
| | | 1.54 | 2. | 2. | 3.08 | 106 | | 1.80 |
| | | 1.71 | 2. | 2. | 3.42 | 90.8 | | 1.54 |
| | | 1.71 | 2. | 2. | 3.42 | 84.5 | | 1.43 |

La - Anvil edge
Lw - Pyrophyllite edge
D - Diameter of AgCl insert
L - Length of resistance wire

Pm = p x m
p = primary pressure

m = ram area / anvil tip area

$P_r$ = Transition pressure.

Optimal Dimensions of AgCl Insert. Table 8 summarizes some data pertaining to this question. The data for the thallium transition (as well as the data for the two lower bismuth transitions presented in Tables 4 and 7 do not easily lend themselves to interpretation in terms of dimension D (diameter of AgCl insert) and the primary pressure at which transition occurs. The only conclusion that can be drawn is that seemingly the internal friction contributes only a minor portion of the losses and that the gasket is the main contributor within the Bi I-II - Tl II-III range.

As to barium transition, the first four lines in Table 8 show a definite trend of pressure reduction caused by increase in the size of the AgCl insert. This would indicate a reduction in internal losses due to lower friction coefficient of the silver chloride. For all that, the last three values indicate the existence of an optimal condition at some intermediate size.

It seems, therefore, that the question of optimal relative dimensions of the AgCl insert will require some additional research.

CONCLUSION

The first investigation confirmed the usefulness of large specimens. A suspected but never observed correlation between volumetric and electric discontinuities for Bi III-IV and Bi IV-V was confirmed.

The second investigation showed that the geometry of the specimen (particularly the dimensional relations between anvil tips and specimen assembly) greatly influences the efficiency of the setup. A proper selection of the geometry can substantially lower the primary pressure corresponding to transition phenomena. However, the optimum geometry is a function of the pressure level which is being investigated.

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