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Apparatus

An Automatic Calibration System for Measuring Electrical Resistance at High Pressures

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An improved method for measuring electrical resistance at high pressure is described. Bridgman anvils are employed. Two reference standards, either bismuth and barium or bismuth and tin, are run simultaneously wtih the unknown to obtain pressure calibration. The method is described and typical data obtained with lead and beryllium are shown.

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An Automatic Calibration System for Measuring Electrical Resistance at High Pressures

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Bridgman anvils have proven to be very useful and versatile high-pressure research tools. One of their earliest uses was in the measurement of electrical resistance of materials at high pressures. However, in such systems the anvils were used as resistance leads and thus the contact resistance between the sample and anvils was included. In many cases the contact resistance was very small and could safely be ignored; in other cases it was appreciable compared to the resistance of the sample and had to be estimated.

Stromberg and Jura (1)[1] have published a method whereby the measurement of contact resistance is eliminated. Stromberg and Stephens (2) have recently published a modification wherein a reference standard may be run concurrently with the unknown. Thus by using bismuth as a reference a pressure calibration for the particular anvil set is obtained simultaneously. This is quite convenient as previous experience (2) has shown that each anvil set must be individually calibrated; anvil sets differ in attainable pressures by as much as 20 percent.

We have recently devised a method to run two reference standards with the unknown with a consequent improvement in accuracy of pressure calibration. Measurements are taken at room temperature. The method is described below.

EQUIPMENT

Bridgman-type compression anvils were used to contain the samples to about 150 kb. Shrink interference fit between the carbide and the support ring was 1.2 percent. Fine-grained carbide with 3 percent cobalt binder was used because of its dimensional stability. The anvils usually were cracked after compressing to 150 kb, the cracking generally occurring on decompression. The material used to support the carbide was a shock-resisting tool steel such as vacuum-melted Al 609, hardened to Rockwell C of 46-48. These steel support rings are reusable many times on replacement of the carbide insert. Press load on the anvils was supported by an inserted carbide end block to eliminate jacket creep between the steel and carbide.

Data were recorded on an x, y, y'-plotter,

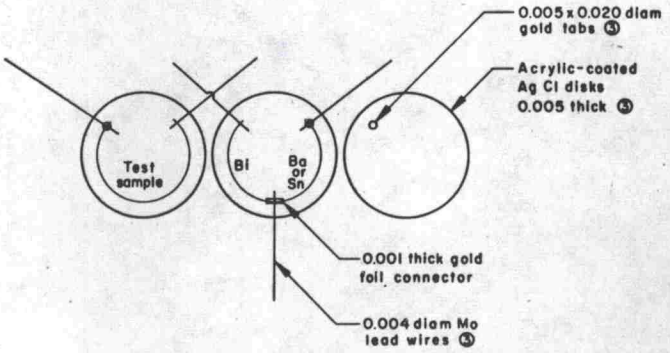


Fig. 1 Bridgman anvils

where the x-coordinate was driven by a strain-gage-operated load cell whose calibration was virtually a straight-line function. Range of pressure on a given size coordinate paper could be changed by varying the constant voltage input to the load-cell bridge circuit from 2 to 15 volts. Output from a constant-current power supply was placed across the samples in series, and voltage drop across each sample was individually taken by means of 0.004-in-dia molybdenum wires inserted through the anvil gaskets, Fig.1. It was found by experience that wires made from high-modulus material were the most successful. Molybdenum was an ideal choice since it is quite ductile, strong, and has a high E. Breaking of these interconnecting wires virtually never occurred. One of the best materials found for interconnecting the samples within the sample cavity and for obtaining good contact with the molybdenum lead-in wire was pure gold. Gold was compatible with all of our samples and was soft enough that local pressure gradients were not detected.

The press used to apply the load was a Lake Erie 500-ton hydraulic press especially designed for high-pressure-die application. Parallelism of the platens was better than 0.001 in. in 10 in. Air-operated hydraulic pumps were used to power the main ram, and ram pressure was controlled by a fine needle valve. Control of the ram pressure was quite sensitive over the entire range of loading speeds. A distinct advantage of the air-operated hydraulic pump is its quiet operation, which is very desirable in a research laboratory.

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

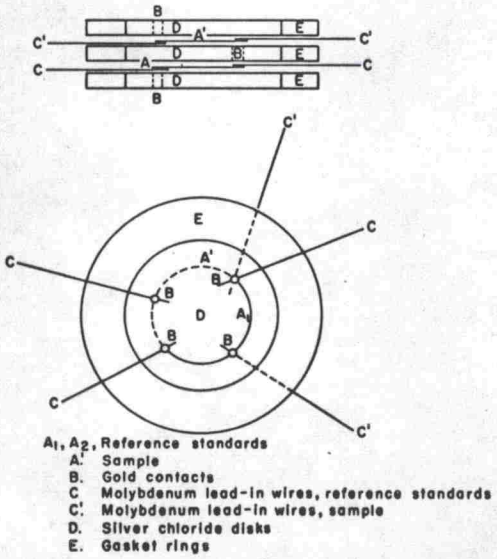


Fig. 2 Sample subassembly, dimensions in inches

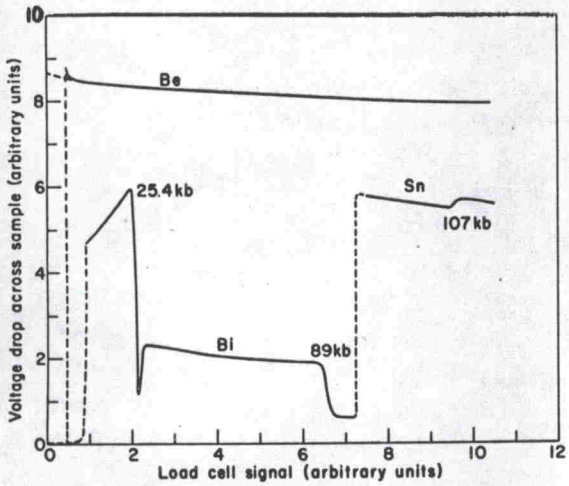


Fig. 3 Completed sample assembly

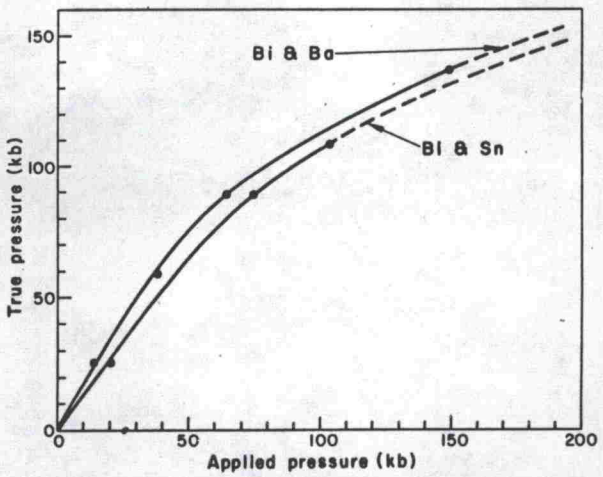


Fig. 4 Typical data trace for beryllium, bismuth and tin

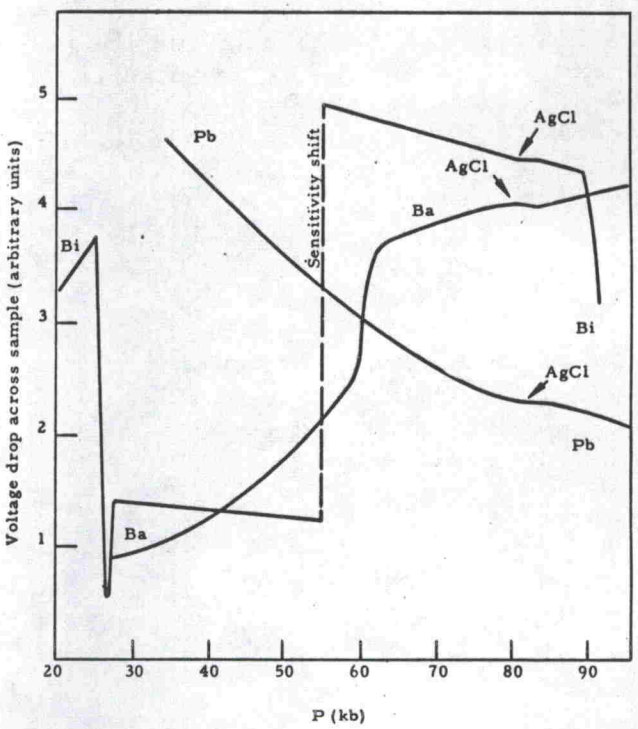


Fig. 5 Typical calibration curves

PROCEDURE

Procedure for loading the sample was similar to that described by Stromberg and Stephens (2) where split gaskets 3/16 in. wide with a total thickness of 0.002 to 0.026 in. were used to contain three disks of single-crystal silver chloride each 0.005 in. thick. These gaskets were carefully controlled to a total thickness of 0.020 in. The gasket material used in most cases was pyrophyllite. A few of the runs were made using gaskets of mica-filled epoxy, which were much less fragile than the pyrophyllite gaskets and held at least as well. In fact, a thicker silver-chloride stack could be held without blowout. Sample strips of the materials to be tested were bent into hoops 0.20 in. dia and placed between the silver-chloride disks concentric with the center of the anvil face. This silver chloride was coated with a film of clear acrylic spray to eliminate corrosion of the samples by the silver chloride. When working with barium strips, it was necessary to keep the barium coated with DP oil under a flowing stream of argon in order to produce satisfactory samples. Assembly was done using a 10-power stereoscopic microscope.

The samples were placed between the silver-chloride disks in such a manner that continuous current flow from one anvil face to the other tra-

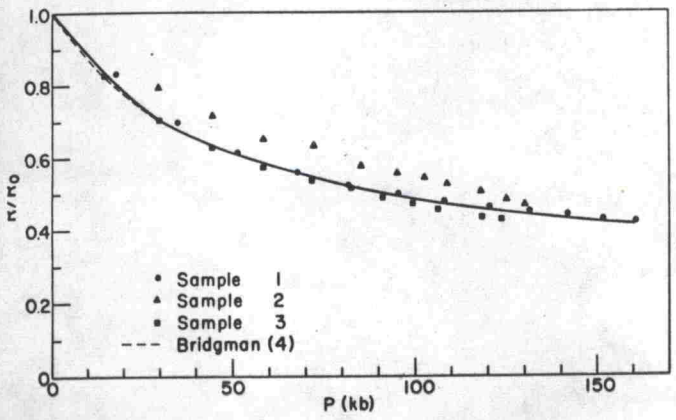


Fig. 6 Resistance of lead versus pressure

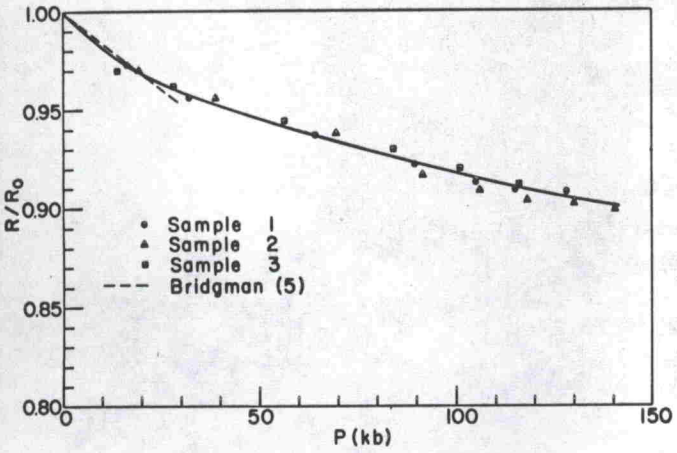


Fig. 7 Resistance of beryllium versus pressure

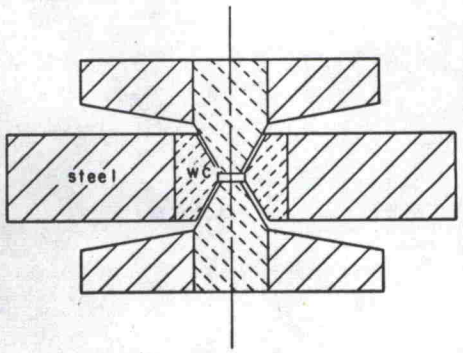


Fig. 8 Girdle anvil system, 1/4-in. anvil faces

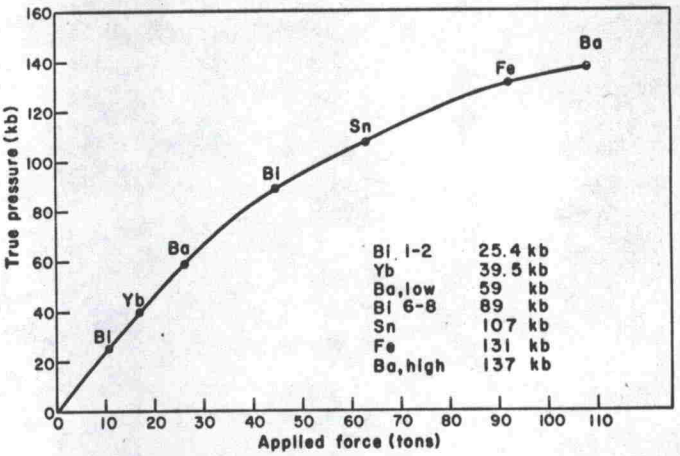


Fig. 9 Calibration of 1/4-in. face girdle anvil

versed all three samples in series. Appropriate gold tabs were used to interconnect the samples, Fig.1. At each junction of the three samples 0.004-in-dia molybdenum wires were brought out through the gaskets to measure voltage drop across each sample separately. This method eliminated contact resistance problems and gave a true R/R_0 reading, Fig.2.

The only sample requiring accurate R/R_0 analysis was the unknown, since the Bi-Ba or the Bi-Sn samples were used only to indicate transition pressures. This gave an automatic calibration on each run. Since the load cell driving the x leg of the x, y, y' recorder gave a precise straight-line function of pressure versus millivolt output, relatively accurate pressure calibration was obtained, providing the transitions obtained were accurately known.

RESULTS AND CONCLUSIONS

A typical data trace is shown in Fig.3. This was made with beryllium, bismuth, and tin. Thus three pressure points were used as the pressure calibration of this run: Bi 1-2 at 25.4 kb, Bi 6-8 at 89 kb, and tin at 107 kb. Bismuth and barium also were used as the two reference standards. A typical curve of applied pressure (on a 1/2-in-face anvil) versus true pressure is shown in Fig.4. Pressures higher than the reference standard transitions were estimated by extrapolation. Also a short extrapolation of the resistance trace was necessary to obtain R_0. The convenience of this system for pressure calibration is quite apparent.

Silver chloride was used as the pressure-transmitting medium. In the majority of runs the transition in silver chloride reported by Bridgman could be observed. The increase in electrical conductivity over this transition apparently allowed a very small fraction of the current to bypass the sample and cause a small but discernible anomaly in the resistance curve. By the use of our pressure calibration as reported in this and a previous paper (2), we have determined this phe-

nomenon to occur at approximately 80 kb. This is lower than Bridgman's (3) value of 88 kb; the reason for the discrepancy is not known. A data trace indicating the magnitude of the silver chloride transition is shown in Fig.5.

This system was used to measure the resistance of lead and of beryllium with pressure; these curves are shown in Figs.6 and 7. The lead was 99.995 w/o pure; it was obtained from Johnson & Mattheny, Ltd. The beryllium was 99.7 w/o pure and was obtained from Aeroprojects, Inc.

The transition in lead was not obtained, since our system is not capable of pressures higher than 150-160 kb. Data of Bridgman (4,5) also are shown.

The transition in beryllium reported by Marder (6) was not confirmed. As reported previously, pressures in a Bridgman anvil system using hoop-shaped samples are nearly hydrostatic. In previous work on iron (2) the 130-kb transition was not obtained although data were obtained to 150 kb. This apparent overshooting of the transition can be attributed to the lack of shear forces. There is some evidence that nonhydrostatic forces tend to initiate sluggish reactions, and it is possible that the case for beryllium might be similar to iron. This does not seem likely, however, as we have repeatedly observed the iron transition in another, less hydrostatic system, the girdle anvils shown in Fig.8. The calibration for this system is shown in Fig.9. However, we have not observed any phase change in beryllium with the girdle anvil system. Our beryllium samples were 4 mils dia, compared to 20 mils for that of Marder, and were of comparable or higher purity. Neither of his reported phenomena, the 93-kb phase change nor the 55-kb anomaly, were observed.

CONCLUSION

We have described a simple and accurate system for measuring electrical resistance at high pressure and room temperature. The data are about as accurate as the degree to which the pressures of transition of the reference standards are known. Thus the accuracy may be estimated as $\pm$ 2 percent to 90 kb and $\pm$ 4 percent at the higher pressures.

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