Title

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Authors

Vaisnys, Juozas R.
Stromberg, Harold
Jura, George.

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ABSTRACT

A modification of the Bridgman anvils is described. This modification permits studies to be made to a load of 400 kbars, about twice that previously available. The external deformation of the anvils has been studied for 1/4-in. faces of 1-in.-diam. carbides. Thus, a lower limit of the pressure can be stated provided internal deformations are neglected. It is shown that silver chloride is an insulator to the highest loads, and selenium is a semiconductor to a load of 270,000 bars.

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I. INTRODUCTION

The pressure dependence of the electrical resistance has been investigated by Bridgman for many substance.[1] Using the anvil technique, Bridgman was able to reach a pressure of 178,000 kg cm^{-2},[2] but most of the investigations were limited to a maximum of 100 kg cm^{-2}. Recently several investigators have made measurements as high as 200 kbars. By modifying the anvil design, we have successfully reached a calculated pressure of 400 kbars. This is not the ultimate limit of the design, but at present the maximum pressure is restricted by the size of the available press.

The exact pressure attained is uncertain because of mechanical deformation of the carbide inserts. An independent pressure gauge is not available. The absolute pressure scale was extended to 30,000 kg cm^{-2} by Bridgman,[3] but at higher pressures the scale becomes uncertain. However, Bridgman has shown that, below 100,000 kbars, friction is small in the anvil design, and the pressure can be computed from the applied force. In this work, it is assumed that frictional losses are negligible over the entire pressure range.

The mechanical deformation of the anvils under high pressure is of two kinds. First, the anvil faces do not remain flat but become convex, so that the pressure cavity becomes "lens" shaped. If the solid material

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in the cavity were truly hydrostatic, this deformation would have no effect on the computed pressure. Otherwise, it is the average pressure that is quoted. Secondly, the anvils deform so that the area of contact increases and the load is distributed over a larger area. The actual pressure is lowered by this increase in the load-bearing surface. We have been able to measure the change in the area of contact at high pressure by radiographing the anvils, but it has not been possible to determine the extent of "lensing", because the carbide boundaries were very indistinct when X-rays of sufficient energy to pass through the carbides were used. The region of indefiniteness in the shadow was greater than the thickness of the sample.

II. EXPERIMENTAL METHODS

The present anvil design employs a tighter shrink fit of the carbide insert in a high-tensile-strength steel jacket. One-and-a-half percent interference is used, compared to Bridgman's 0.75%, and the initial compression stress in the carbide is of the order of 240,000 psi. This higher compression stress permits the attainment of higher cavity pressures before shear breakage of the carbide occurs. If the carbide insert is polished and lubricated with MoS_2 (Molycote), a total force of about 25 tons will drive the carbide smoothly into the jacket. The steel used for the jacket is Ferrovac 609, a high-strength die steel manufactured by Vacuum Metals Corporation. The steel is heated to 1650°F in a neutral pack, oil quenched, and drawn at 850°F to a hardness of Rc 48 to 50, measured at about 1/16 in. below the surface. If the steel jackets are too hard, their ductility is low, and may open under strain at any time after the carbides are pressed into place. Once, with a hardness of Rc 60, a jacket separated into halves with explosive force.

Because of the limited press capacity, the carbides were ground with a working surface of 1/4-in. diam. rather than the 1/2-in. used by Bridgman. Previous experiments with 3/8-in.-diam. die faces, using the Bridgman design, indicated that breakage under pressure is independent of the face area.

The experimental arrangement is essentially that described by Bridgman. The major difference is found in the technique for low-resistance samples: a 0.008-in.-thick AgCl disc containing a centered hole 1/16-in. diam. was used, rather than the thin strip AgCl sandwich arrangement described by Bridgman. The disc diameters were such that the disc just fitted inside the retaining rings 0.010-in.-thick made of either pipestone or ferric-oxide-coated pyraphalite. Low resistances were measured with a Muller bridge; high resistances below 10^7 ohms, with a Leeds-Northrup Tupe-S No. 5300 bridge. For resistances above 10^7 ohms an RCA Senior Voltohmist WV 98 A was used.

III. EXPERIMENTAL RESULTS

Anvils, made with inserts of Kennametal K96 and of GE Carboloy Grade-999 carbides, were radiographed. The radiographs were taken by J. L. Hile and E. M. Placas of Lawrence Radiation Laboratory at Livermore with a portable model-MG-100 Norelco 100-kv X-ray machine. The increase in area was calculated from the change in face diameter. With the K96 inserts, radiographs were taken both with the dies directly in contact and with a sample of polyethylene contained in a pipestone ring.

Within the error of measurement, the results are identical. The Carboloy-999 anvils were photographed only with the faces in direct contact. The results of these measurements are shown in Fig. 1, where the gauge pressure is plotted against the "true" pressure. The gauge pressure is the pressure calculated from the applied force under the assumption that no change occurs in the area of contact. The "true" pressure is obtained by assuming that the entire experimentally determined area of contact is uniformly loaded. Since part of the deformation is elastic, as shown by the partial recovery of the anvils when pressure is released, the "true" pressure is probably best interpreted as the minimum average pressure that is attained, provided friction losses can be neglected.

At 200 kbars the deformation of the K96 carbides increases in such a manner that "true" pressure appears to decrease with an increase in applied forces. That this is not so is shown by the fact that the resistance of several substances continues to decrease as the applied force is increased, and these measurements indicate that the "true" pressure is about 225 kbars at gauge pressure of 350 kbars.

The Kennametal K96 carbide, containing 6% cobalt binder, is softer and thus deforms more readily at the high pressures than the GE Carboloy 999, which contains only 3% cobalt binder. However it has the advantage of a longer life at lower pressures because it has a higher transverse rupture strength. The average life of an anvil with this carbide is two compressions to a pressure of 200 kbars. Breakage always occurred after the maximum pressure had been reached, and in most cases after the pressure had been completely released. The most common failure was in the form of shear cracks in the surface of the carbide around the pressure cavity. Because high residual strain is present, spalling can occur for a considerable time after the release of pressure, and therefore eye protection should be worn whenever anvils that have been subjected to high pressure are handled. Pieces have struck eye glasses without damage to the glasses.) In most cases the carbide breakage is quite shallow, and the anvil assembly can be ground down and re-used several times.

Figure 2 shows the resistance of an AgCl disc as a function of pressure. It is questioned whether the initial increase in resistance for pressures below 30 kbars is real, since no careful study was made of this region. What is important is that the resistance remains over 10^6 ohms at the highest pressures obtained.

Figure 3 gives the measured resistance, uncorrected for end effects, of selenium as a function of pressure. The data of Bridgman are also shown for comparison. One hundred kbars was taken as the fiducial point, and the agreement to this pressure is satisfactory. The results indicate that selenium is a semiconductor over the entire pressure range investigated. A transition to the metallic state of the sort found by Bridgman for tellurium at 45 kbars has not occurred with selenium. Indeed, it appears that the specific resistance of selenium begins to increase at the very highest pressures that were attained. For this reason it is probable that the conductance observed in sulfur by David and Hamann is semi- rather than metallic conductance.[4]

IV. REFERENCES

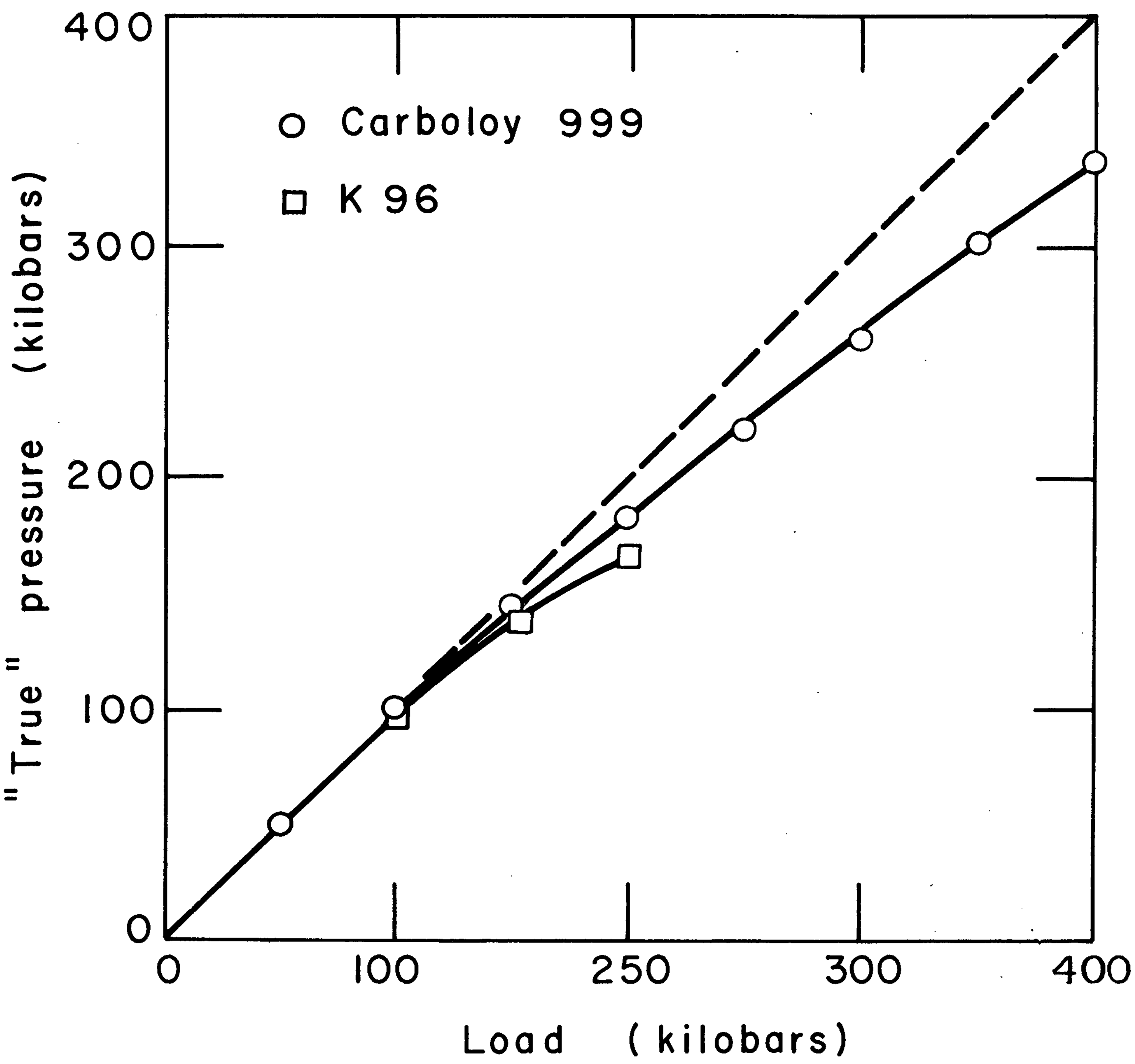
1. P. W. Bridgman, Proc. Amer. Acad. Sci. 8, 165 (1952).
2. P. W. Bridgman, J. Appl. Phys. 27, 659 (1956).
3. P. W. Bridgman, Phys. Rev. 57, 235 (1940).
4. H. G. David and S. D. Hamann, J. Chem. Phys. 28, 1006 (1958).

LEGENDS

Fig. 1. Effect of load on increase in area of contact of carbides. These data are for 1/4-in. surfaces on nominal 1-in. carbides.

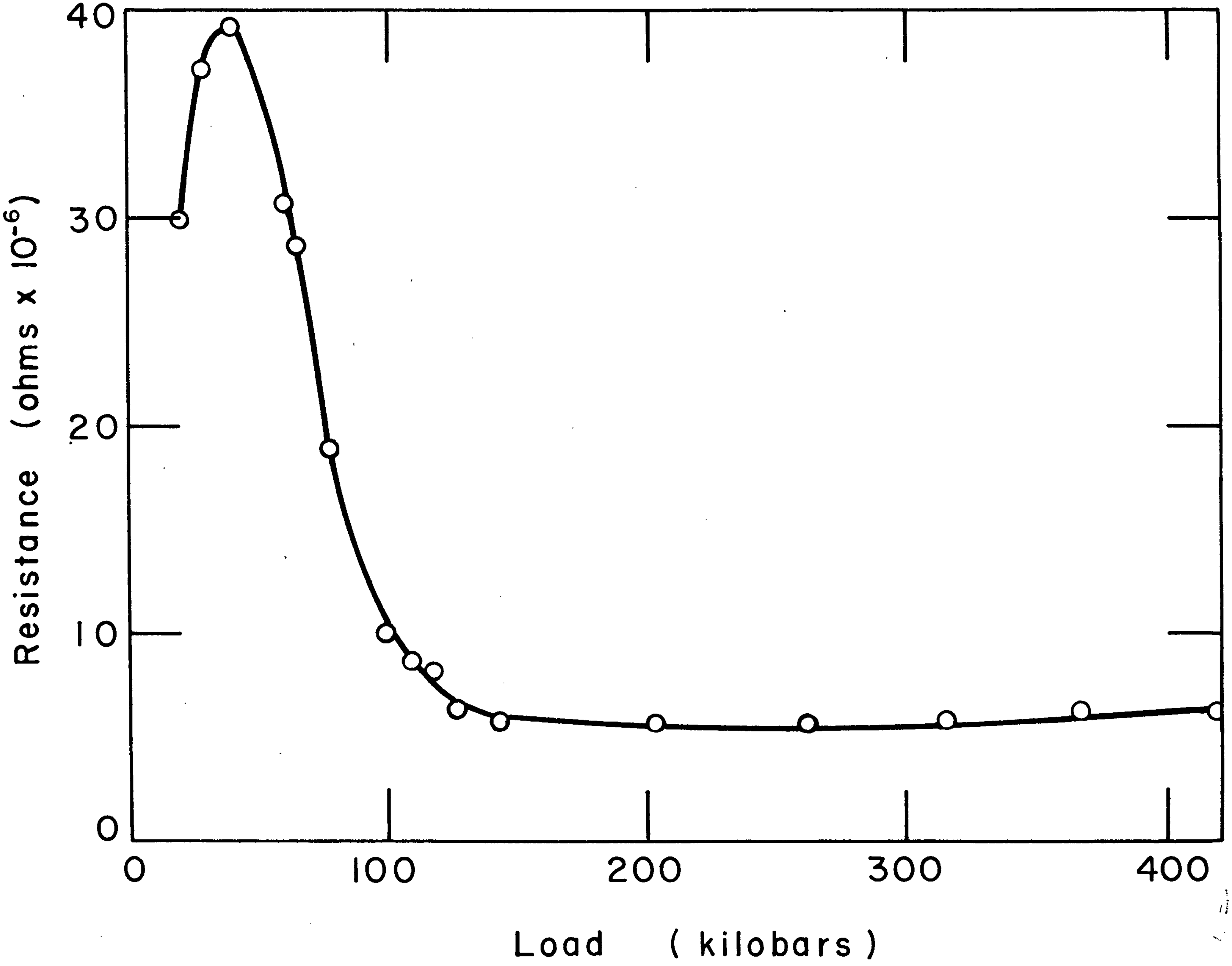
Fig. 2. Resistance of AgCl as a function of load. Carboloy-999 carbides were used in these experiments.

Fig. 3. Resistance of selenium as a function of load. These results were obtained using K96 carbides.

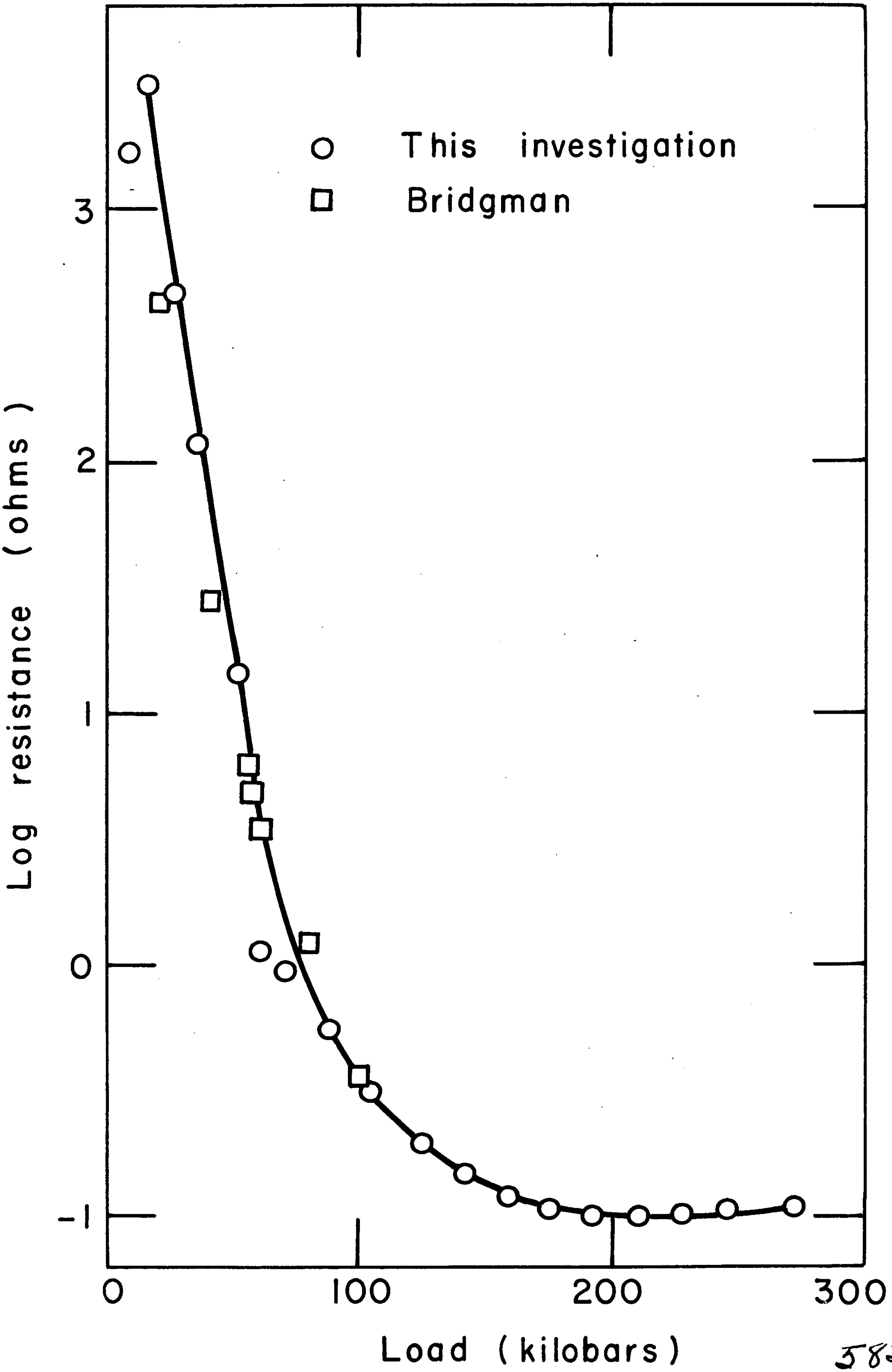

400
300
200
100
0
"True" pressure (kilobars)
Carboloy 999
K 96
0
100
250
300
400
Load (kilobars)

2 3

282271-1


Resistance (ohms x 10^{-6})
40
30
20
10
0
0
100
200
300
400
Load (kilobars)

58525-1


This investigation
Bridgman
3
2
1
0
-1
Log resistance (ohms)
0
100
200
300
Load (kilobars)

58526-1