ELECTRICAL RESISTANCE MEASUREMENTS OF METALS TO 40 GPa IN THE DIAMOND CELL

R. Reichlin

To cite this version:
R. Reichlin. ELECTRICAL RESISTANCE MEASUREMENTS OF METALS TO 40 GPa IN THE DIAMOND CELL. Journal de Physique Colloques, 1984, 45 (C8), pp.C8-399-C8-402. 10.1051/jphyscol:1984871 . jpa-00224373

HAL Id: jpa-00224373
https://hal.archives-ouvertes.fr/jpa-00224373
Submitted on 1 Jan 1984

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L’archive ouverte pluridisciplinaire HAL, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d’enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.
ELECTRICAL RESISTANCE MEASUREMENTS OF METALS TO 40 GPa IN THE DIAMOND CELL

R. Reichlin

University of California, Lawrence Livermore National Laboratory, Livermore, CA 94550, U.S.A.

Abstract - A four-probe technique has been developed for measuring electrical resistances of metals to 40.0 GPa in a diamond-anvil cell. Several gasketing techniques are presented which provide the necessary support for the diamonds and the electrical leads at the diamond edges. Electrical resistance data are presented for iron and bismuth. A comparison is made between electrical resistance measurements made on bismuth under hydrostatic and non-hydrostatic conditions.

A number of new techniques have recently been developed for measuring the electrical resistance of samples under high pressures in diamond-anvil cells (DAC). The advantages of the DAC over conventional Bridgman anvils or sintered diamond techniques include the ability to visually observe the sample during the experiment and to directly measure pressure using the ruby pressure scale /1/.

Block and Piermarini /2/ and Block et al. /3/ developed a two-probe resistance technique using a gasketed cell and liquid pressure medium. They reported resistance measurements to 20 GPa. Walling and Ferraro /4/ developed a four-probe technique using a Mylar gasket capable of measuring resistances up to 10 GPa. Mao and Bell have designed a number of modifications of both two-probe and four-probe resistance techniques using MgO gaskets /5/. They have reported data from a series of two-probe conductivity measurements of minerals up to 30 GPa. Their four-probe design /6/ was used to observe the α-to-ε transition in iron at about 13 GPa, although no resistivity data have been published. Sakai et al. /7/ have measured resistances at low temperature (1.7 K) and pressures of up to 25 GPa using a modified two-probe technique. Reichlin /8/ has developed a four-probe technique utilizing several gasketing modifications for measuring resistances to 40.0 GPa.

Electrical resistance measurements of metallic conductors must be made using a four probe technique to alleviate large errors associated with contact and lead-wire resistances. A four-lead wiring arrangement has been utilized in our experimental design in which electrical resistences can be measured to pressures of at least 40.0 GPa. This report briefly discusses the experimental technique and some examples of electrical resistance measurements made on metal wires of iron and bismuth. A detailed discussion of our four probe technique can be found in /8/.

Résumé - On a mis au point une technique à quatre sondes pour mesurer la résistance électrique des métaux jusqu'à 40.0 GPa dans une presse de diamant à haute pression. On présente plusieurs méthodes de fermeture étanche qui fournissent le soutien nécessaire aux diamants et aux contacts électriques pour les bordent. Les données de résistances électriques pour le fer et pour le bismuth sont présentées. On compare les mesures de résistance électrique faites sur le bismuth sous les conditions hydrostatiques et sous les conditions non-hydrostatiques.

Article published online by EDP Sciences and available at http://dx.doi.org/10.1051/jphyscol:1984871
A "megabar" DAC based on the design of Mao and Bell /9/ was used for the resistance experiments. Figure 1 illustrates the relationships between the cell, gasket, and electrical components. The gasket (discussed in detail below) is placed on spacers so that it lies flush with the diamond culet and is attached firmly to the piston with screws. The lead-wires and sample are laid directly on the gasket and attached to the surface. Figure 1 shows the details of the wiring arrangement on the gasket. Resistance measurements are made by passing a small, constant dc current (2 to 20 mA) through the sample wire (A to A') and measuring the voltage drop across electrodes B and C in the very center of the sample wire. By measuring the voltage drop on a very small segment of the sample wire in the center of the diamonds, the effect of pressure inhomogeneities can be minimized. Ruby chips for pressure determination are placed in the cell around the sample wire.

![Diagram of DAC assembly for electrical resistance measurements](image)

The major difficulty in making electrical resistance measurements at high pressures in the DAC is in developing a suitably strong insulating gasket. A number of potential gasket materials were investigated during the development of our resistance technique /8/. We found several combinations of materials which were suitable for making measurements up to 40 GPa. The best gaskets we found for high pressure experiments are illustrated in figure 1. For the lower pressure bismuth experiments we used a drilled metal gasket, plasma sprayed with Al₂O₃, similar to the girdles illustrated in Fig 1d.

We measured the electrical resistance of iron, so that direct comparison could be made with extensive data already existing on iron. Figure 2 plots relative resistance vs pressure for a sample of iron wire. The discontinuity in the relative resistance at about 15 GPa is assumed to mark the initiation of the
α-c transition in iron. This transition pressure agrees well with that observed by Zou et al. (10/1 (-15.3 GPa) in a recent x-ray diffraction study of iron in the DAC. They reported a sluggish transition ranging over an approximately 10 GPa pressure range, which is consistent with our resistance data.

Fig. 2 - Relative resistance, R/R_{1.8}, of iron vs pressure (R_{1.8} is resistance of iron at 1.8 GPa and 22°C). Data of Balchan and Drickamer (11) are inserted for comparison.

Tozer (1984, personal communication), have utilized the metal gaskets plasma-sprayed with Al₂O₃ to make hydrostatic measurements of electrical resistance in the diamond-anvil cell. They have used glycerol as the pressure medium and report measurements of the resistance of bismuth to 7 GPa. Figure 3 is a plot of relative resistance of bismuth as a function of pressure and includes the data of Tozer as well as our non-hydrostatic measurements using an MgO pressure medium in a similar gasket. Both sets of data are normalized to the resistance of bismuth at 9.3 GPa.

Overall, the data are in very good agreement. In the bismuth I phase (up to about 2.6 GPa), the two sets of data are in perfect agreement. The initiation of the Bi(I-II) phase transition agrees within 0.1 GPa, which is less than the estimated error in the precision of pressure measurements. The main difference in the hydrostatic and non-hydrostatic data is in the sharpness of phase transitions. The Bi (I-II) transition is very sharp in the hydrostatic data, whereas in the non-hydrostatic data the transition is somewhat smeared out over a pressure range of .4 or .5 GPa, as indicated by intermediate values of relative resistance in the transition region. The very small phase field (~ .2 GPa) of Bi II observed in Tozer's data is not seen in the non-hydrostatic data, probably due to the coexistence in this region of all three of these phases as a result of pressure gradients around the sample wire.

In the high pressure region (above 3 GPa) the two data sets are in reasonably good agreement. The data follow the same trend, though the two curves are displaced by as much as five to ten percent. The Bi III to V phase transition at about 7 GPa is observed in both sets of data, but again is less clearly defined in the non-hydrostatic case.

From this comparison of hydrostatic and non-hydrostatic measurements of electrical resistance of bismuth in a DAC it is reasonable to conclude that non-hydrostatic measurements can be very useful in accurately determining the pressures of the onset of phase transitions in metals. Furthermore relative resistance data measured under hydrostatic and non-hydrostatic conditions are in very good agreement with each other. Very small phase fields may be missed in...
non-hydrostatic experiments, presumably because of pressure gradients around the sample wire which cause some smearing out of resistance data at transition boundaries.

Up to now hydrostatic measurements of electrical resistance of metals in the DAC are limited to about 7 GPa because of the problems of containing a liquid in the special gaskets needed to meet the other requirements of electrical resistance techniques. Our non-hydrostatic techniques described here and in /8/ can be used up to at least 40 GPa.

Fig. 3 - Relative resistance of bismuth, $R/R_{g,3}$ vs pressure ($R_{g,3}$ is resistance of bismuth at 9.3 GPa and 22°C). Hydrostatic data of Tozer are indicated by A, our data using MgO pressure medium are indicated by 0.

ACKNOWLEDGMENTS

I would like to thank Stan Tozer for sending me his bismuth data prior to publication (has been submitted to Rev. Sci Instr.). I also gratefully acknowledge the technical support of S. Martin. The work was done under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory, under Contract No. W-7405-Eng-48.

REFERENCES