Materials and techniques for pressure calibration by resistance-jump transitions up to 500 kilobars

K. J. Dunn and F. P. Bundy

General Electric Corporate Research and Development, Schenectady, New York 12301

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The resistance jump of $\alpha \rightarrow \epsilon$ transitions for Fe–V alloys up to 20-wt.% V is observed by static compression. It is used along with $\alpha \rightarrow \epsilon$ transitions for Fe–Co alloys and some other transitions as the basis for pressure calibration up to 500 kilobar.

INTRODUCTION

In the early days of research in the field of high pressure the apparatus and pressure magnitudes were such that the pressures attained could be determined by direct force over area methods. As techniques and designs for higher pressures were developed, using pistons of truncated cone or truncated-pyramid geometry with pressurized gasketing on the flanks, the cell pressure could no longer be determined directly from the force applied to the piston bases because an undetermined fraction of the applied force was borne by the gasket. In such apparatus in which the electrical resistance of the specimen could be monitored pressure calibration points could be established by observing resistance jumps associated with known first-order phase transitions in the specimen material.

As the pressure capability of various kinds of static ultrahigh pressure apparatus was increased still more by development, the problem of determining the true values of the pressures at which various first-order phase transitions occur at the higher pressure levels became more difficult. In general, two or three approaches were taken. One was to observe shock compression experiments, in which very high pressures could be reached transiently, for discontinuities in volume or density associated with phase transitions.[1] The second was to study in detail the P, V, T behavior of some simple-structured substance (like NaCl or Al) and work out a theoretical model which could be extrapolated rather reliably beyond the region of actual measurements.[2] Then, in pressure cells amenable to x-ray diffraction monitoring, the pressure could be gauged by the lattice compression of the model substance. If a resistance-monitored substance with a phase transition could be observed simultaneously in the same cell, the pressure of the transition could be established by the lattice compression of the surrounding model substance. A third approach was to insert a material having some

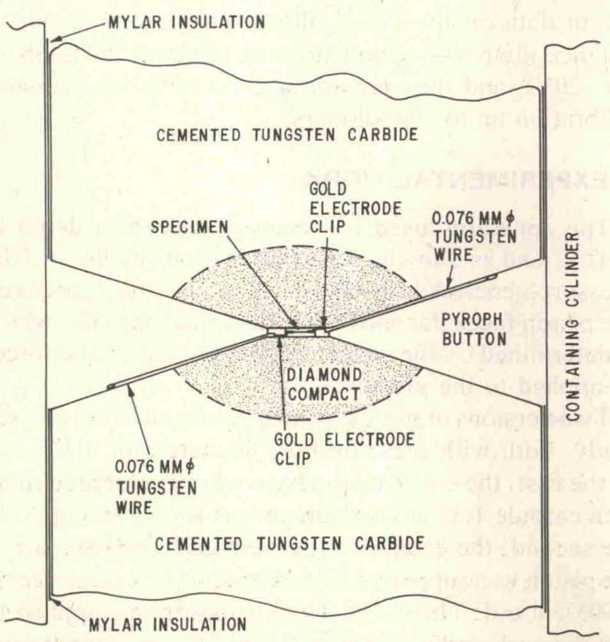

FIG. 1. Cross section of diamond-tipped opposed anvil apparatus used in the calibration experiments.

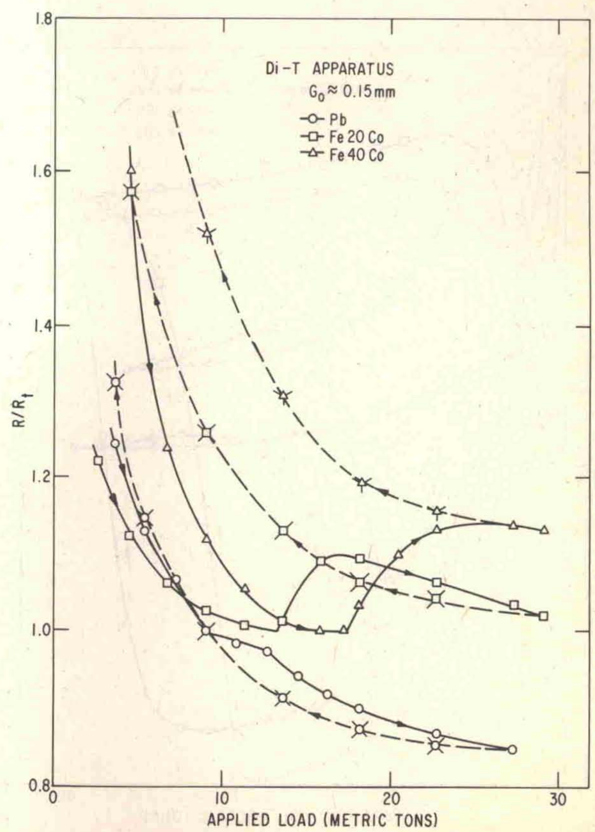

FIG. 2. Resistance versus force loading of the apparatus for specimens of Pb, Fe–20Co, and Fe–40Co in the 1.37-cm-diam apparatus.

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kind of optical spectrum shift with pressure along with a model compressibility material (such as ruby and NaCl) and monitor the spectral shift of the first against the lattice compression of the second.[3] Then a resistance-jump substance could be run against either the spectral-shift substance or the lattice compression substance to establish the pressure values of the resistance-jump transitions.

Historically, Bridgman suggested the first resistance-jump calibration points of Bi, Tl, Cs, Ba, etc., in his paper on resistance behavior of materials based on experiments in his opposed anvil apparatus.[4] The pressure numbers associated with these transitions were revised by Kennedy in 1960.[5] A new higher range of resistance-jump calibration points was added in 1961 by Balchan and Drickamer[6] (Fe, 131; Ba, 144; Pb, 161; Rb, 190 kilobars). As a result of the development of the NaCl compression scale by Decker[2] and others, Drickamer[7] revised the numbers for Fe, Ba, Pb, and Rb downward in 1970. In 1975 Bundy reported some newly observed resistance-jump data associated with the $\alpha \rightarrow \epsilon$ phase transitions in the Fe–Co alloys first reported by Loree *et al.*[1] as density transformations observed in shock compression experiments. These Fe–Co resistance-jump transitions were observed in a new opposed piston apparatus[8] in which the piston tips were of strongly sintered diamond powder. According to the shock compression data the Fe–40Co ($\alpha \rightarrow \epsilon$) transition occurred at about 290 kilobars. Using a sample of the same material and a Bassett-type "diamond squeezer" apparatus

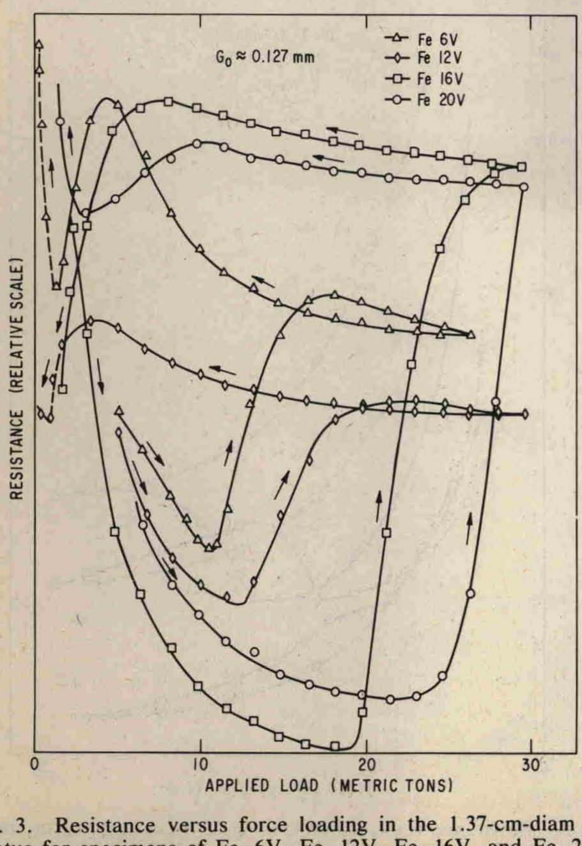

FIG. 3. Resistance versus force loading in the 1.37-cm-diam apparatus for specimens of Fe–6V, Fe–12V, Fe–16V, and Fe–20V, all at about the same G_0 in the same apparatus.

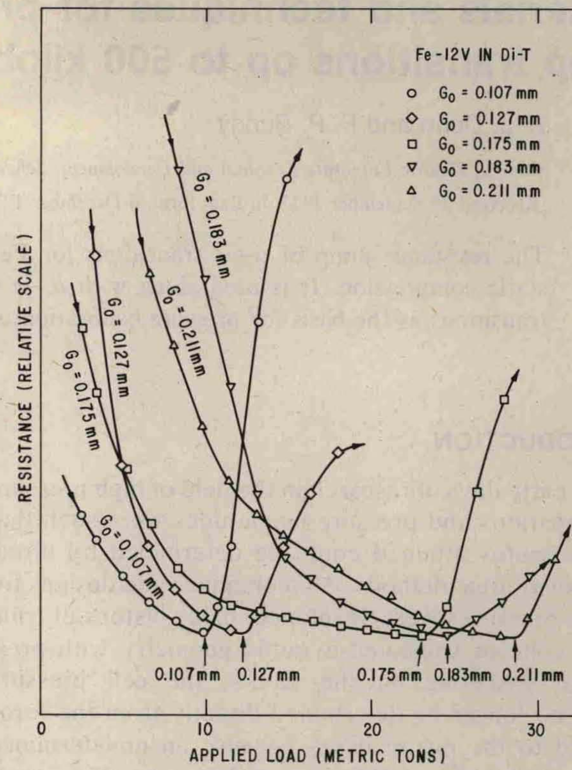

FIG. 4. Resistance versus loading for Fe–12V in the 1.37-cm-diam apparatus for a range of initial gaps, G_0.

Papantonis and Bassett[9] checked it by x-ray diffraction monitoring against NaCl. The $\alpha \rightarrow \epsilon$ transition in the Fe–40Co occurred at about 290 kilobars by the NaCl compression scale, which is about the same pressure at which NaCl starts to transform over to the CsCl structure. Thus the resistance-jumps of the $\alpha \rightarrow \epsilon$ transition in the Fe–Co alloys could be used reliably for calibration up to about 300 kilobars.

The purpose of the present paper is to present some recent data on the Fe–V alloys which show good resistance-jump $\alpha \rightarrow \epsilon$ transitions up to about 500 kilobars (Fe–20V), and thus provide a good basis for pressure calibration up to 500 kilobars.

I. EXPERIMENTAL WORK

The apparatus used has been described in detail in Ref. 8, and is shown in partial section in Fig. 1. The pressure generated at the center of the space between the piston faces depends upon the initial gap, G_0, which is determined by the gasket thickness, and by the force, L, applied to the pistons.

Two versions of the apparatus were used in the present study, both with pressure-face diameters of 0.127 cm. In the first, the entire piston base was of cemented tungsten carbide 1.37 cm in diameter, as shown in Fig. 1. In the second, the cemented tungsten carbide base part of the piston was supported by a shrunk-on tool steel sleeve, 1.905-cm o.d. This steel sleeve support was applied to put the carbide base part into initial hoop compression and thus prevent the development of hoop tension stress in the carbide during heavy axial loading. The pistons