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Techniques for Physical Measurements in Quasi-Hydrostatic Pressure Systems

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A number of techniques for physical measurements in large volume, quasi-hydrostatic pressure apparatus have been developed: (a) Two pressure-measuring techniques, namely a multiple event calibration cell and a manganin gage with multiple integral calibrants, are described. These two methods were used in reexamining the upper Bi transition. The results indicate that the upper limit for the pressure value of this transition is 81-82 kbars. (b) Improvements in the previously described induction-coil technique for compressibility measurements are discussed, and results on Bi at 20 and 170 C presented. (c) Methods for measuring the shift of ferromagnetic Curie points and changes in magnetization are given and typical data on Ni and 70 pct Fe-30 pct Ni alloy shown. (d) The problems of measuring the pressure coefficient of dielectric constants are considered, and tentative data on $SrTiO_3$ are presented. In addition, a novel approach for measuring dielectric constants, which does away with lead-wire difficulties, is described. (e) Finally, a solid ionic cell for possible use in studying the effects of pressure on ionic conductivity is briefly discussed and a typical response curve given.

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Techniques for Physical Measurements in Quasi-Hydrostatic Pressure Systems

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In this paper we shall describe some of the techniques that we have been using for making a number of physical measurements in apparatus where pressures, although not truly hydrostatic, have no preferred directional characteristics. The paper is divided into five sections.

In the first section we review two methods for the measurement of pressure in the 100-kbar range. The first is a multiple-event calibration cell which provides five fixed points in this range. The second is a manganin gage with multiple integral calibrants for continuous pressure measurement. The use of these two methods in reexamining the value of the upper bismuth ("88 kbar") transition is described.

The second section describes a method for the measurement of compressibilities at both room and elevated temperatures. The technique incorporates improvements in the inductive coil method that we have used previously in studying polymorphic transitions. Important among these improvements is the development of a high-strength coaxial cable for use at high pressures. Data on the compressibility of bismuth at 20 and 170 C are presented.

In the third section we present methods for studying the effects of pressure on the Curie point and saturation magnetization of ferromagnetic materials. Some observations on nickel and on iron-nickel alloy (70 pct Fe -30 pct Ni) are given and comparisons made with similar data obtained under purely hydrostatic conditions.

In the fourth section, the problem of measuring the dielectric constant of high dielectric-constant materials in nonhydrostatic apparatus is considered. Some preliminary results on strontium and barium titanates are discussed. A novel resonant-coil technique for possible use in measuring dielectric constants is briefly described.

Finally, a solid ionic cell for possible use in studying ionic conductivity under pressure is described and a typical response given.

The pressure apparatus used in most of this work is a 2000-ton hexahedral (or cubic) multi-anvil unit (1).[1] The anvil faces are 1.46 in. on edge. Pyrophyllite was used for specimen containers (or blocks) and served as the solid pressure-transmitting medium. Standard blocks (2.26 in. between opposite faces) with integral preformed gaskets were used throughout (1). The large size of the sample chamber allowed us to develop techniques whereby sample deformation and distortion are reduced to negligible proportions. This eliminates a major uncertainty in the interpretation of the data. Even with fairly compressible materials, samples as large as $\frac{1}{4}$ in dia x $\frac{1}{2}$ in. long are recovered with no more than $\pm$ 2 percent change in dimensions after compression to 60 kbars.

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

MEASUREMENT OF HIGH PRESSURE

In working with pressures above 30 kbar, the problem of absolute and continuous measurement of pressure remains as one of the principal challenges. In hydrostatic systems, i.e., below 30 kbar, pressure is usually measured by monitoring the change in the resistance of manganin wire. The manganin is given a pressure-temperature "seasoning" treatment and is calibrated against a free-piston gage and/or against certain well-established fixed pressure points (usually the freezing point of Hg and $Bi_{I} \rightarrow_{II}$ transition). This yields a nearly linear relationship between resistance and pressure with excellent day to day reproducibility.

Above 30 kb most fluids solidify at room temperature, and one resorts to solids such as pyrophyllite, boron nitride, talc, and so on, for use as pressure-transmitting media. Some attempts have been made to extend the use of the manganin gage to higher pressures. Static (2,3) as well as dynamic (4) measurements have shown that the pressure coefficient of the resistance of manganin is not far from constant up to around 300 kbar. Unfortunately, however, manganin is very sensitive to shear stresses, and, even in hydrostatic usage, the slightest strains can change its pressure coefficient by several percent. Such stresses and strains are present, to varying degrees, in all types of apparatus utilizing solid pressure transmitting media and, furthermore, they usually vary from one run to another in the same apparatus. The result is poor reproducibility, and any precalibration of the manganin would have little significance.

The techniques which have been most commonly