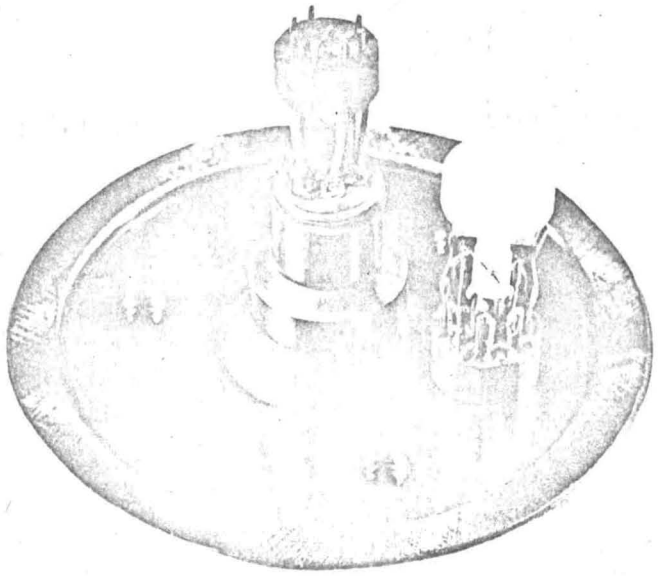

Fig. 1—Specimen holder and bottom closure for hydrostatic pressure system.

high-purity single-crystalline stock having the following detectable impurity levels:

Pb - 0.6 ppm
Fe - 0.9 ppm
Cu - 0.1 ppm
Cd - 0.3 ppm

In order to minimize variations in impurity content between the single and polycrystalline specimens, the polycrystalline samples were produced from the original single crystals by remelting into a 3/4-in. billet and extrusion at 220°C to a 0.17-in. diam. The smaller grain size samples were prepared directly from the as-extruded material. For the larger grain size, the as-extruded material was subjected to an annealing treatment of 220°C for 5 hr. It was attempted to obtain polycrystalline samples by progressive compressive deformation of the single crystals with intermediate annealing. Although moderate grain sizes could be obtained in this manner, this technique was abandoned in favor of the former.

The specimens were 0.16 in. in diam and 0.22 in. long. Prior to pressurization, a plane parallel to the longitudinal axis of the specimen was metallographically prepared by electropolishing using a saturated KI solution with 2 pct by volume of concentrated HCl.

C) Resistance Measurement. Electrical resistance as a function of pressure was measured by means of a conventional potential-drop method using a K-3 type potentiometer and recording oscillograph. Two specimens were connected in series to a current source, and the potential drop across each was measured separately. The arrangement of the two specimens in the sample holder is shown in Fig. 1 along with the bottom pressure closure. For the pressure run, the sample holder was simply connected to the seven-anode insulating block of the

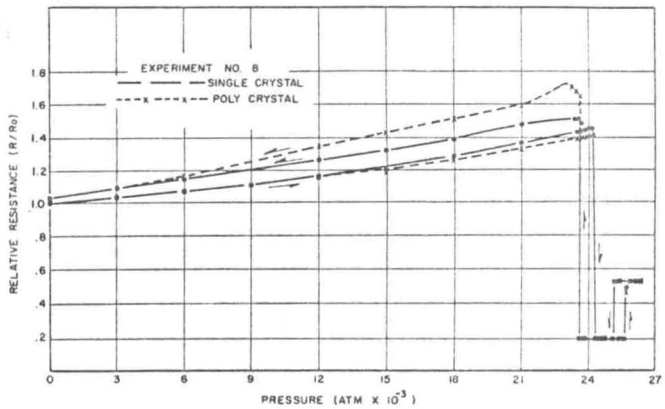

Fig. 2—Relative resistance vs pressure.

closure. There is a total of seven electrical leads emerging through the bottom closure with two being used for the current through the specimens, four for voltage measurements, and the remaining one for the manganin coil inserted inside of the insulating block.

D) Procedure. In all experiments, pressure was increased by 3000-atm increments up to 21,000 atm and a 2000-atm increment to 23,000 atm with a 5- to 10-min hold period between each pressure change to permit stabilization of the pressure and voltage readings due to thermal effects. Beyond 23,000 atm, four different procedures, consisting of *a*) 100-, *b*) 250-, and *c*) 500-atm increments with 5-min stabilization periods, and *d*) continuous pressurization at the rate of 50 atm per min, were utilized. The depressurization rate closely approximated the procedure used for increasing pressure.

RESULTS AND DISCUSSION

A) Transition Pressure. In order to establish the effect of initial structure on the transition variables, a single crystal and a polycrystalline sample, connected in series in the manner previously described, were simultaneously exposed to the pressure. Typical relative resistance vs pressure curves utilizing this procedure are shown in Fig. 2.

As is demonstrated in Fig. 2, the I-II and II-III transitions, upon increasing pressure, occur isobarically with the transformation pressure being independent of initial structure. Similarly, the III-II transition, upon decreasing pressure, is also isobaric and structure-insensitive. However, the II-I transition is not completely isobaric, but exhibits some sluggishness near the completion of transformation. This deviation from isobaric conditions is small, usually not exceeding 300 atm.

Under the condition of this experiment, there is a substantial hysteresis in both the I-II and II-III transitions. The magnitude of this hysteresis is independent of structure, but somewhat dependent upon pressurization rate as is shown in the following tables.

The single value of the pressures and pressure difference shown is the average for all tests (only