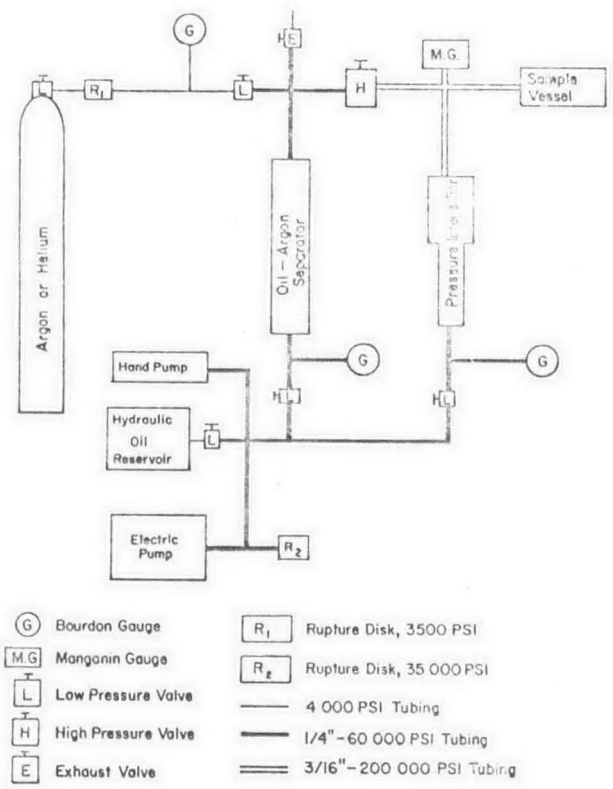

FIG. 1. Schematic drawing of the pressure system.

many of the experiments are difficult to perform because of oxidation problems. Gold has been the most extensively studied fcc metal in the experimental investigations, and it is the only one of the noble metals for which independent measurements have been made of the volumes of formation and motion. Emrick[14] determined the volume of motion ΔV_m by measuring the effect of pressure on the annealing rate of quenched-in resistivity. DeSorbo[15] derived a value of the volume of formation ΔV_f by combining his calorimetric measurement of the total energy released during the annealing out of quenched-in vacancies with the resistivity data of Bauerle and Koehler.[16] Simmons and Balluffi[3] likewise derived a value of ΔV_f by combining their vacancy-concentration data with the data of Bauerle and Koehler, and also with the data of Takamura,[17] both combinations yielding the same value, but differing from DeSorbo's value. A more direct determination of ΔV_f was accomplished by Huebener and Homan,[18] who measured the effect of pressure on the quenched-in resistivity of gold. The purpose of the present investigation is to provide an independent determination of the total activation volume, $\Delta V = \Delta V_f + \Delta V_m$, by measuring the effect of hydrostatic pressure on the rate of self-diffusion in gold.

II. EXPERIMENTAL PROCEDURE

A. Preparation of Specimens

A single crystal of 99.99% pure gold (Sigmund Cohn Corporation), approximately $\frac{3}{8}$ in. in diameter and 4 in. in length, was grown under a vacuum of at least 5×10^{-6} mm of mercury in a furnace similar to that described by Lazarus and Chipman.[19] The usual precautions were taken to clean and bake out the high-purity graphite crucible used for the crystal growth.

The single crystal was sectioned with a jeweler's saw, and the standard metallographic procedure was used to polish, etch, and anneal the cylindrical specimens. The final size of the specimens was 0.410 in. in diameter and 0.210 in. in thickness. A light etch after annealing showed that some recrystallization had occurred in some of the specimens, but the stray grains were few and large enough to preclude competitive grain-boundary diffusion.

The Au^{198} isotope was obtained from Oak Ridge National Laboratory in the form of a very high-specific-activity solution of $AuCl_3$ in a mixture of HCl and HNO_3. An active layer of Au^{198} was electroplated on each specimen just prior to the diffusion anneal. It was estimated that the thickness of the plated layer could not have exceeded 100 Å.

B. Apparatus and General Procedure

A gas pressure system similar to the one described by Goldsmith and Heard[20] was used in the present experiment. A schematic drawing appears in Fig. 1. A mixture of argon and helium was used, the helium being added in a small amount to make possible the use of a mass spectrometer leak detector tuned for helium. Gas leaking from connections or other possible leakage sites was trapped and carried outside the safety barricade by a set of polyethylene tubes. A sniffer attachment to the leak detector was then used to find the locations and magnitudes of any leaks that existed.

Pressures up to 2 kbar could be reached with the separator alone. Higher pressures were attanied with the Harwood intensifier. Inevitable leaks during the diffusion anneals limited the runs to 9 kbar.

The sample vessel itself was machined from Bethlehem "Omega" tool steel and hardened to Rockwell C-52. The volume available for the internal furnace

[14] R. M. Emrick, Phys. Rev. 122, 1720 (1961).
[15] W. DeSorbo, Phys. Rev. 117, 444 (1960).
[16] J. E. Bauerle and J. S. Koehler, Phys. Rev. 107, 1493 (1957).
[17] J. Takamura, Acta Met. 9, 547 (1961).
[18] R. P. Huebener and C. G. Homan, Phys. Rev. 129, 1162 (1963).
[19] D. Lazarus and D. R. Chipman, Rev. Sci. Instr. 22, 211 (1951).
[20] J. R. Goldsmith and H. C. Heard, J. Geol. 69, 45 (1961).