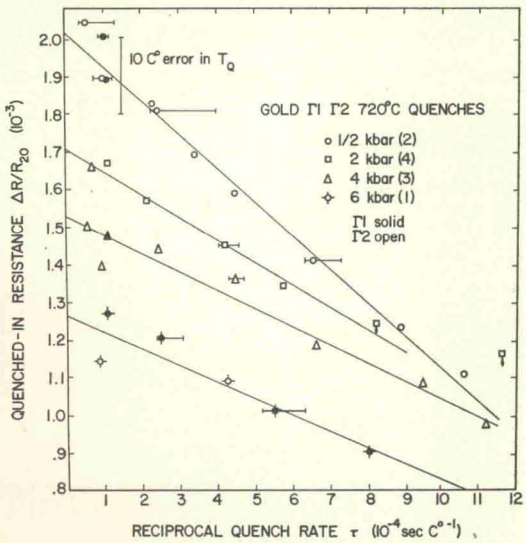

FIG. 2. Isobars of quenched-in resistance versus reciprocal quench rate for high-temperature quenches of gold. Numbers in parentheses indicate sequence in which isobars were made.

three figures from room temperature to beyond the quench temperature.

D. Pressure Apparatus

The specimen frame was inserted in a rectangular slot in a 1-in.-diam aluminum cylinder. The cylinder minimized the dead volume in the pressure vessel and helped minimize temperature gradients. The pressure system was a standard gas system with a Harwood intensifier operated with dry piston packings and a 1:1 separator in the priming line to minimize the possibility of oil entering the system. Pressure was measured by a manganin gauge calibrated against the freezing pressure of mercury. The pressure medium was ultra-high purity argon for most series of quenches. However, no differences in results could be attributed to the use of commercial grade argon on several runs.

E. Experimental Procedure

Since it was difficult and undesirable to immerse the specimen plug in a temperature bath, the specimen resistance was measured in the pressure vessel at atmospheric pressure and temperature. Specimen temperature was determined by the thermocouple on the plug (Fig. 1) which had been calibrated against an ASTM thermometer. This value of resistance was then used as the base for the temperature scale. (The thermocouple was not used to determine the quench temperature.) Since the thermocouple was not in direct contact with the specimen it is possible that the temperature scale from one specimen to the next varied by ± 5°C at T_Q. Thus direct comparison of values of ΔR for a given P and indicated T_Q from one specimen with those of another is difficult. Fortunately, the specimen remains in place with no further handling once it is in the vessel. An entire series of quenches at different rates and pressures can be performed on a single specimen. It is therefore possible to do the entire series without affecting dislocation densities due to mechanical strains. Thermal strains are also minimized since the maximum quench rate was 2×10^4 C°/sec.

After annealing, the resistance base δR_b[4] was measured at bottle gas pressure with both current directions to minimize the effect of thermal emf's. The system was then pumped to the quench pressure. The specimen was heated slowly to T_Q by use of the temperature controller. T_Q was determined by measuring the specimen voltage with an L&N K3 potentiometer and specimen current with a Weston Model 931 ammeter which was calibrated against a 1-Ω resistance standard. Specimen resistance at T_Q was calculated using the BLB data. A Tektronix 555 Dual-Beam oscilloscope was triggered after the K3 potentiometer output indicated a stable temperature had been attained. The scope-delayed trigger started the linear ramp which quenched the specimen while the specimen voltage and current were recorded on Polaroid film. Trace intensity was modulated by pulses from a Tektronix Type 184 Time-Mark generator operating into the z-axis inputs of the 555. This procedure eliminated errors due to variations in sweep speed between the two beams. The photographic traces could then be analyzed to determine quench rates.[11]

Variations in T_Q due to turbulence of the pressure gas are the chief source of error. It is necessary for the controller to sense the resistance of the entire specimen length. The gauge length can therefore fluctuate as much as ± 15°C while the *average* temperature of the

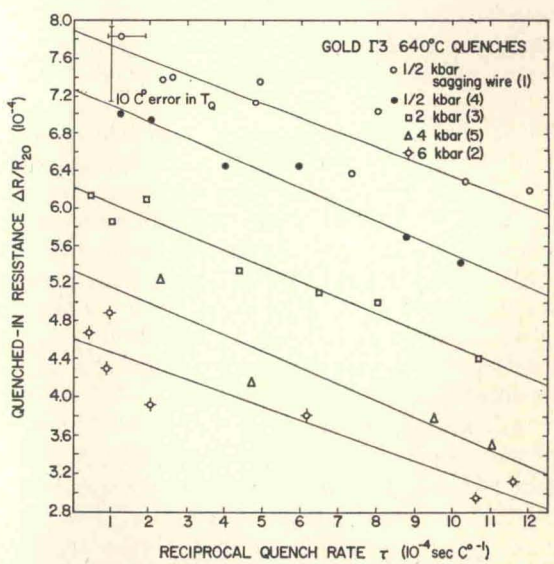

FIG. 3. Isobars of quenched-in resistance versus reciprocal quench rate for low-temperature gold quenches. Numbers in parentheses indicate sequence in which isobars were made. After taking isobar (1), it was discovered that the specimen sagged during the interquench anneals.

entire specimen remains constant. Small pressure leaks had a dramatic effect on temperature stability, presumably because they introduced currents in the high-pressure gas. Errors in reading the films led to an uncertainty of ± 10°C in measuring temperature. When the quench temperature indicated by the film was more than 10°C away from the desired value, the results of the quench were discarded.

The resistance after quench was measured immediately after the pressure had been released, typically within 8 min after quench and never more than 15 min. A single measurement (the average over two current directions) could be taken within 6 min after quench. Occasionally measurements were taken over a period of $\frac{1}{2}$ h. No resistance annealing could be detected. For the gold specimens the vessel was at ambient (22–25°C), while for aluminum the vessel was packed in dry ice (about -80°C). Although there was a diurnal variation in internal vessel temperature of about 3°C, no effects could be attributed to it.

Quenches were made at various rates at a given pressure. The sequence of quenches mixed fast and slow rates to reduce the effects of a possible change in specimen purity with use. The sequence of pressures was also varied for the same reason.

IV. RESULTS

A. Gold

Figures 2–4 are isobars of $\Delta R/R_{20}$ versus τ for gold specimens Γ2, Γ3, and Γ4. Four isobars were also made with specimen Γ2 for 640°C quenches. Note that τ is the reciprocal quench rate $\dot{T}^{-1}$ and not τ_Q as discussed in the theory section. They are related by $\tau_Q = T_Q \tau$. Measurement errors in $\Delta R/R_{20}$ are smaller than the symbols. An error of 10°C in quench temperature is indicated by the vertical bars for $E_F = 0.98$ eV, $P = \frac{1}{2}$ kbar, and $\tau = 1\times10^{-4}$ sec/°C. That is, a 10°C error in quench temperature produces a change of about 13% in the equilibrium concentration. Except where noted by error bars, the quench rates are quite linear to at least 500°C below the quench temperature. Occasionally the quench rate would change after the first 100 to 200°C drop in temperature. The range in slopes is indicated by error bars but the point is weighted in favor of the initial slope. In Fig. 3, two $\frac{1}{2}$-kbar isobars are shown. During the first $\frac{1}{2}$-kbar run it was noted that a large (2.5-$\mu\Omega$) baseline shift occurred after each anneal. The specimen was removed from the vessel and found to have sagged excessively. A 0.0006-in. gold wire support was looped under the specimen to lift it back to a nearly horizontal position. It was then given another 25-min 900°C air anneal before replacing in the vessel. The other four isobars were then taken in the order indicated. Specimen Γ2 had no such support, but it neither sagged nor showed the large baseline shift. As a precaution, Γ4 was also made with a support. The support had no visible effect on temperature uniformity at 900°C in air on either Γ3 or Γ4. Despite the baseline shift, the first $\frac{1}{2}$-kbar isobar is a straight line. Thus, a straight line alone cannot be taken as evidence of good data. The baseline shift was presumably caused by the sag of the specimen during anneal. Whether the resistance change was due to a change in dislocation density or specimen dimensions could not be determined.

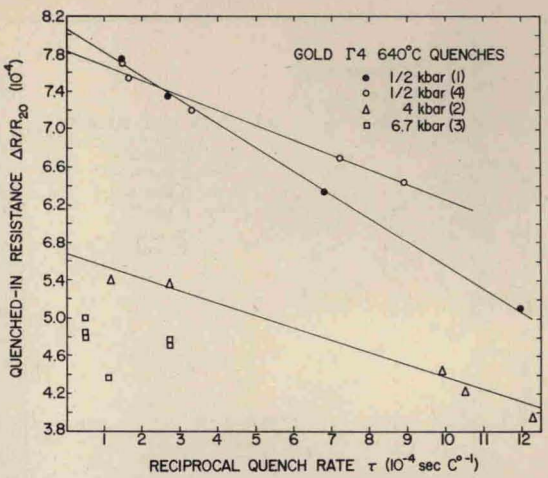

FIG. 4. Isobars of quenched-in resistance versus reciprocal quench rate. Numbers in parentheses indicate sequence in which isobars were made.

At 6 kbar it was possible to get quench rates in excess of 1×10^{4}°C sec^{-1}. Specimen Γ4 was used to determine whether or not the slope for the 1×10^{4} to 1×10^{3}°C sec^{-1} isobars continued to the higher quench rates. These rates, $\dot{T} > 10^{4}$°C sec^{-1}, are the ones typical for quenches into liquids. The heating current decreased so rapidly to produce these high quench rates that the voltage signals were too small to be measured with any degree of accuracy. The ramp speed was found to be linear in the time-constant resistor over a wide range, so the fast quench rates were inferred from the ramp control potentiometer setting. The pressure system was leaking badly at 6.7 kbar, so the full isobar was not traced out. Nonetheless, it is seen to be consistent with the 4-kbar isobar, even for the fastest quenches.

Another interesting result from this specimen is the decrease in slope of the $\frac{1}{2}$-kbar isobar after the 6.7-kbar run. The intercepts agree within 3% but the slope decreases by a factor of 1.6. Further experiments are being made in an attempt to determine the behavior of the sinks under pressure. This result does indicate that our extrapolated values are nearly independent of sink concentration. This is as expected unless the dominant sink is an impurity which has a large vacancy-impurity binding energy.

In Fig. 5 the intercepts are plotted as a function of pressure. The 640°C data do not superpose because of the variation in absolute temperature scale from one specimen to the next as discussed in Sec. III E. The