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Recovery of Pure Platinum and Dilute Platinum-Gold Alloy Electron-Irradiated at Liquid-Nitrogen Temperature*

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Pure platinum, platinum–0.1-at.%-gold alloy and platinum with various supersatuations of vacancies were irradiated at approximately 90 °K by 3-MeV electrons. The production of damage for these samples did not follow the simple power law widely used. Isothermal annealing in 10 C° temperature intervals in the range − 150 to 100 °C revealed annealing peaks at − 75, 0, and 25 °C. The activation energies of these peaks were determined to be 0.48 ±0.04, 0.74 ±0.06, and 0.74 ±0.06 eV. In addition, the annealing was continued to higher temperatures in 25 °C intervals to investigate the stage at 325 °C. From the dependence of the various peaks upon the preirradiation vacancy concentration, dose, and the results for the alloy samples along with the activation energies, it is suggested that the annealing process at 325 °C (activation energy of 1.45 ± 0.06 eV) associated with the migration of vacancies should be called stage III. In this way the physical phenomenon associated with this stage will correspond to that in other metals, such as Al and Au. The stage around − 75 °C is strongly impurity dependent, being completely suppressed in the 0.1-at.% alloy.

I. INTRODUCTION

The recovery of the physical properties (for example, electrical resistivity, change of length, and stored-energy release) of metals irradiated at liquid-helium temperature shows annealing stages when the sample is heated to higher temperatures. Most of the pure metals (Al, Ag, Cu, Pt, Fe, etc.) have a distinct stage below 100 °K, which is called stage I. Above stage I, there is some annealing spread over a wide temperature range often labeled stage II. Next, a rather distinct stage, stage III, follows. Stage III in Al, Au, Ag, and Cu occurs at about −40 °C, 30 °C, − 30 °C, and − 30 °C, respectively. We believe that the physical phenomenon occurring in stage III in Al and Au, namely the migration of vacancies, is really occurring in what is usually called stage IV in platinum, and furthermore that the stage labeled stage III in platinum is in reality a part of stage II. Hereafter this assignment is used in the present paper. Confusion often arises from the differences in labeling used among investigators. Although authors do not always agree on the reasons for relabeling stages IV in favor of stage III such relabeling has been proposed independently by Doyama,[1] Schilling,[2] and Van den Beukel.[3]

There is a considerable amount of literature available on platinum irradiated at temperatures below stage I (~ 25 °K). Neutron irradiations have

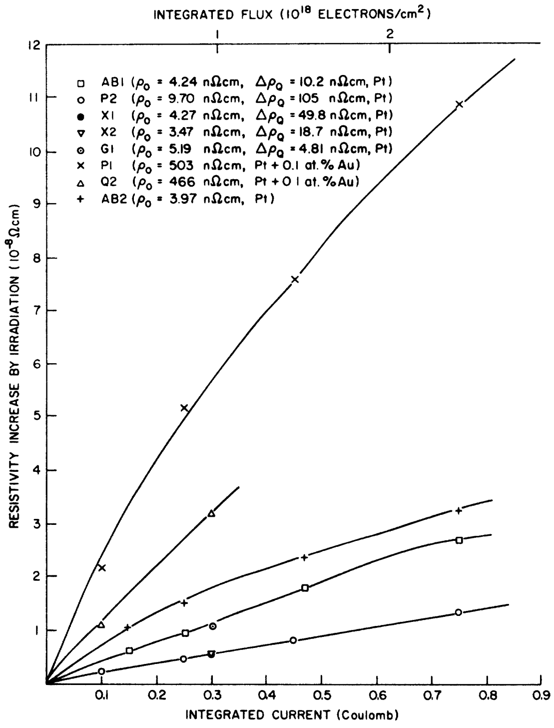

FIG. 1. Resistivity increase as a function of integrated electron flux for samples bombarded by 3-MeV electrons at 80 °K.

TABLE I. Summary of data for irradiated samples.

Sample name	After preparation nΩ cm ρ_0	After quench nΩ cm ρ_Q	After irradiation of dose nΩ cm ρ	Coulomb	Annealing treatment normal anneal = NA[a]
*Q*1	6.307	···	14.379	0.10	−120 °C ~ 100 °C
			22.019	0.30	
52	4.431	···	11.993	0.17	−150 °C ~ 100 °C NA
*G*2	4.989	···	18.908	0.30	−150 °C ~ 100 °C NA
					100 °C → 400 °C[b]
*AB*1	4.238	14.397	19.973	0.15	−120 °C ~ 100 °C NA
			23.839	0.25	
			32.655	0.47	
			41.358	0.75	
*AB*2	3.97	···	14.623	0.15	−120 °C ~ 100 °C NA
			19.125	0.25	100 °C ~ 550 °C[b]
			27.712	0.47	
			37.177	0.75	
*Q*2 alloy	465.869	···	476.772	0.10	−120 °C ~ 100 °C NA
			497.615	0.30	100 °C ~ 475 °C[b]
*G*1	5.190	10.047	20.469	0.30	−150 °C ~ 100 °C NA
*X*1	4.268	53.042	58.974	0.30	−120 °C ~ 100 °C NA
					+ 100 °C ~ 550 °C[b]
*D*2	6.24	···	34.12	0.65	10 min every 10 °C
					−120 °C ~ 100 °C
					10 min every 25 °C
					100 °C ~ 275 °C
*P*2	9.700	114.359	116.194	0.10	−150 °C
			119.117	0.25	−85 °C Isothermals
			122.828	0.45	−55 °C
			128.334	0.75	−15 °C
*H*2	3.979	···	24.512	0.5	−150 °C −55 °C Isothermals
					−85 °C −15 °C
*P*1 alloy	503.319	···	524.974	0.10	−150 °C
			555.156	0.25	−85 °C Isothermals
			578.891	0.45	−55 °C
			612.824	0.75	−15 °C
*X*2	3.474	22.171	28.100	0.30	−120 °C ~ 100 °C
					100 °C → 450 °C

[a] NA, normal anneal, consisting of measurements after 4–, 8–, 16–, and 30–min total time anneals at each temperature. Temperature intervals are 10° C.

[b] Above 100° C temperature intervals are 25° C.

been carried out by the Oak Ridge group[4] and the Munich group.[5] Using deuteron irradiation, Herschbach and Jackson[6] studied extensively the effects resulting from the presence of quenched-in vacancies and changes in sink density. Previous work with electron-irradiated platinum near 4.2 °K by Bauer and Goeppinger[7] used the Meechan-Brinkman analysis[8] of the recovery to determine the activation energies of the stage-I annealing peaks. The model of interstitial-vacancy close-pair annihilation followed by long-range interstitial migration around 28 °K is generally accepted for stage I.

Recently Duesing and Schilling[9] irradiated platinum near 4.2 °K with 3-MeV electrons. They also suggest the relabeling of stage-III annealing in platinum. In that work, along with the only previous electron irradiation near 80 °K,[10] platinum shows two large annealing peaks, one near 0 °C and the other near 300 °C. The purpose of the present research is to study the annealing in detail above 80 °K after electron irradiation at liquid-nitrogen temperatures to determine the activation energies associated with annealing peaks. An additional annealing peak near − 75 °C and improved resolution of the 0 °C peak provide further information to test various models used to explain annealing processes in this region. One hopes to clarify the relation between the temperatures associated with stage II and stage III in platinum and in other metals (Al, Au, Ag, and Cu). The effects of quenching and alloying on the recovery features and the production of damage are used to assist in the understanding.

II. EXPERIMENTAL PROCEDURE

A. Specimen Preparation and Mounting

The specimens in the form of wire 0.005 cm diam supplied by Sigmund Cohn Corp. of nominal 99.999% pure platinum were mounted in a manner similar to that described by Dworschak, Herschbach, and Koehler.[11] The alloy samples were also supplied by Sigmund Cohn Corporation. Since annealing temperatures higher than those at which Mylar insulation is useful are needed to investigate stage III in platinum, specimens were mounted on removable aluminum disks with Sauereisen. These disks permit removal of the specimens from the sample block without deformation during a transfer to a high-temperature sample holder. To achieve the necessary electrical insulation, a thin cleaved mica sheet was used. After mounting, the samples were an-

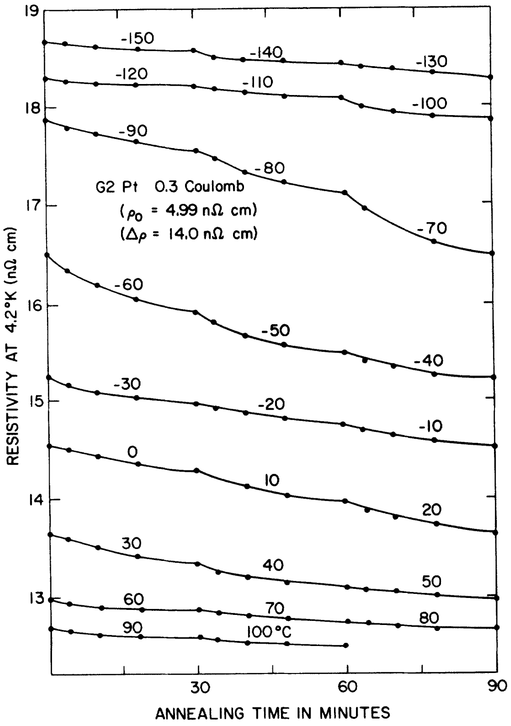

FIG. 2. Isothermal annealing of the electrical resistivity ($\Delta\rho$) between − 150 and 100 °C for specimen G2(Pt) following electron irradiation near 80 °K.

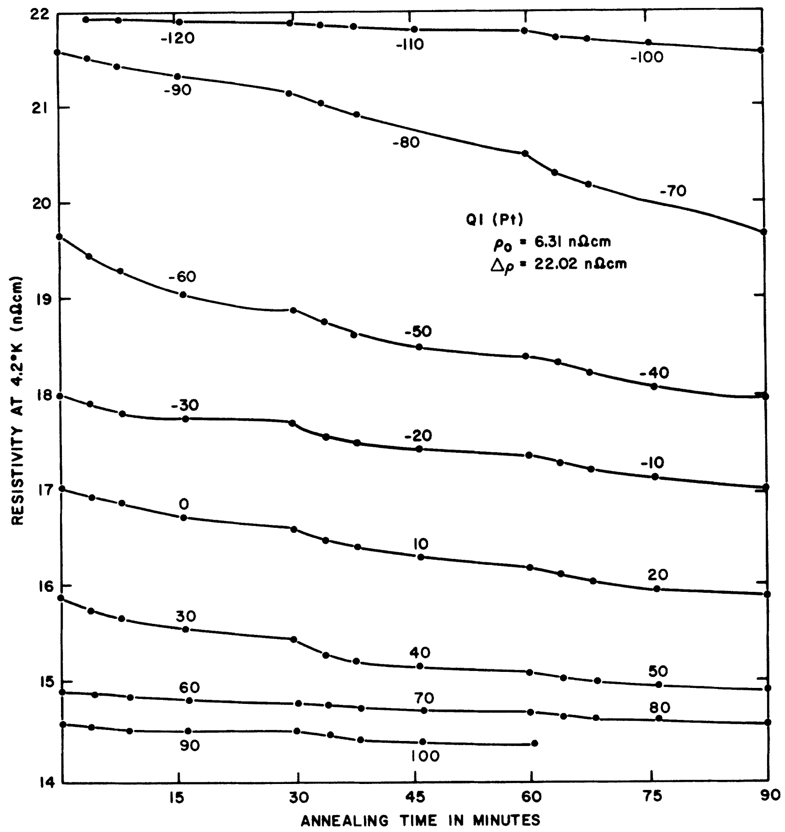

FIG. 3. Isothermal annealing of the electrical resistivity ($\Delta\rho$) between −150 and 100 °C for specimen Q1(Pt) following electron irradiation near 80 °K.

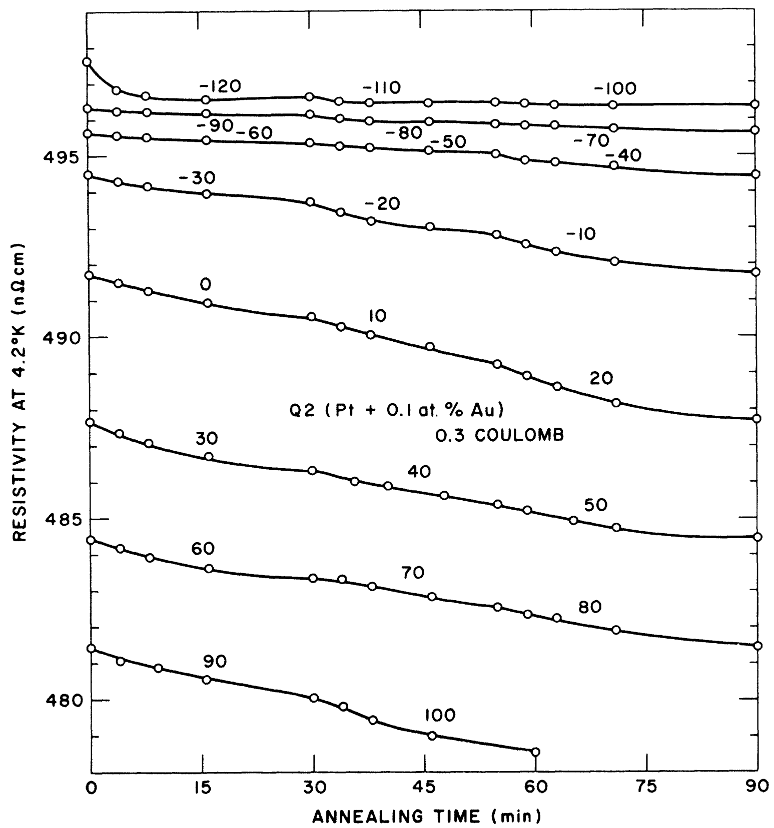

FIG. 4. Isothermal annealing of the electrical resistivity (Δρ) between − 150 and 100 °C for specimen Q2(Pt + 0.1 at.% Au) following electron irradiation near 80 °K.

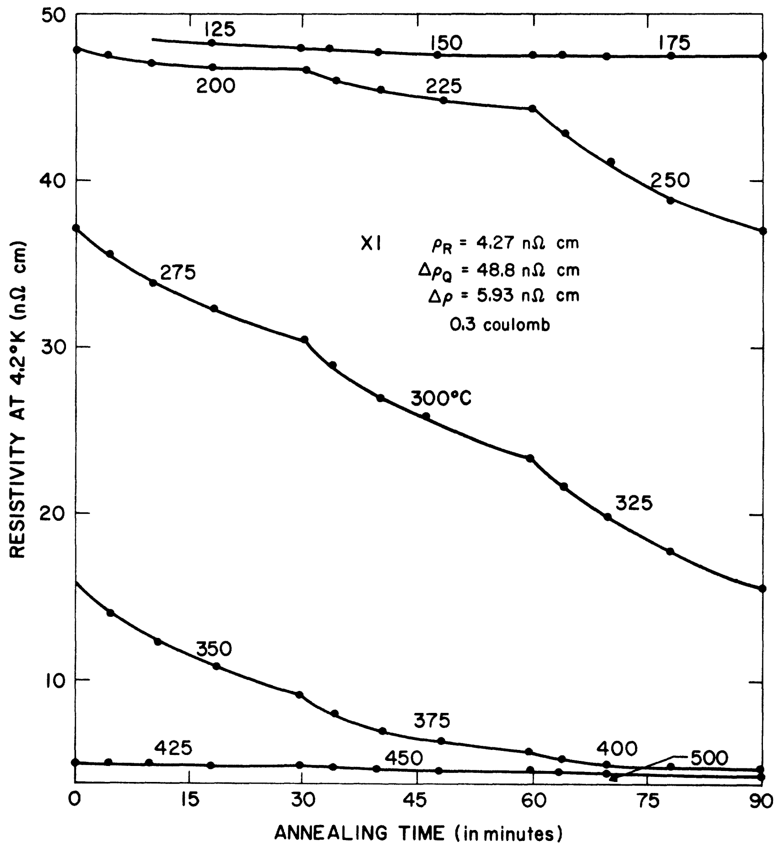

FIG. 5. Isothermal annealing of the electrical resistivity (Δρ) between 125 and 500 °C for specimen X1(pure Pt) following electron irradiation near 80 °K.

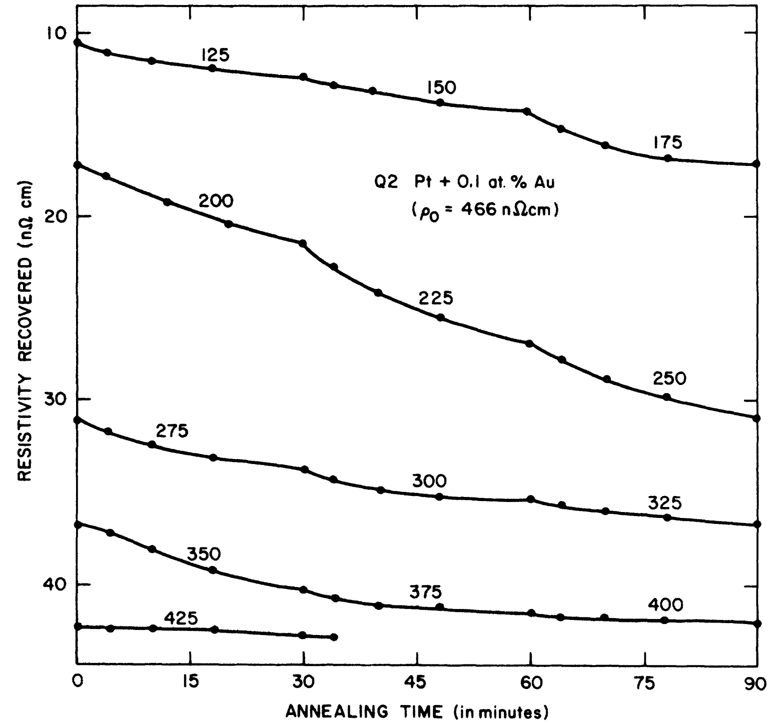

FIG. 6. Isothermal annealing of the electrical resistivity ($\Delta\rho$) between 125 and 425 °C for specimen Q2(Pt + 0.1 at.% Au) following electron irradiation near 80 °K.

nealed by passing current through them for approximately 10 min at 1300 °C and cooled slowly. The ratio between the room-temperature resistance and that at 4.2 °K was typically 2000 (without a size correction).

B. Irradiation Procedure

Irradiation was carried out using the facilities of the University of Illinois Materials Research Laboratory. These include the liquid-nitrogen cryostat described by Lwin, Doyama, and Koehler[12] and the Van de Graaff accelerator of that laboratory. The temperature of the sample was monitored during irradiation and later during the annealing by a copper-constantan thermocouple (2 mil diam) attached to a dummy specimen. The temperature was recorded with a microvolt amplifier and chart recorder throughout the run. Temperature variations

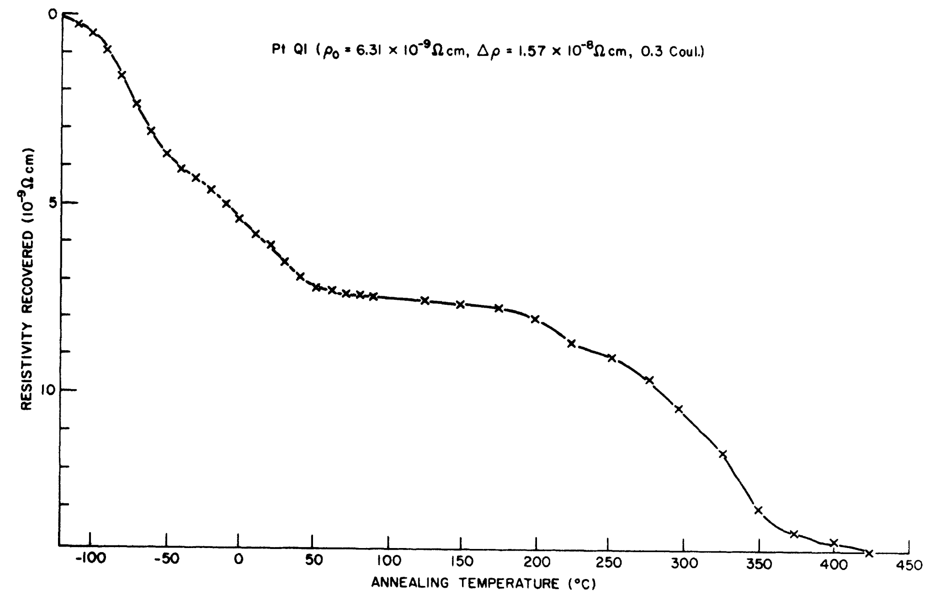

FIG. 7. Isochronal recovery of the electrical resistivity ($\Delta\rho$) for platinum specimen Q1 following 3-MeV electron irradiation near 80 °K, and annealed for 30 min every 10 °C below 100 °C and for 30 min every 25 °C above 100 °C.

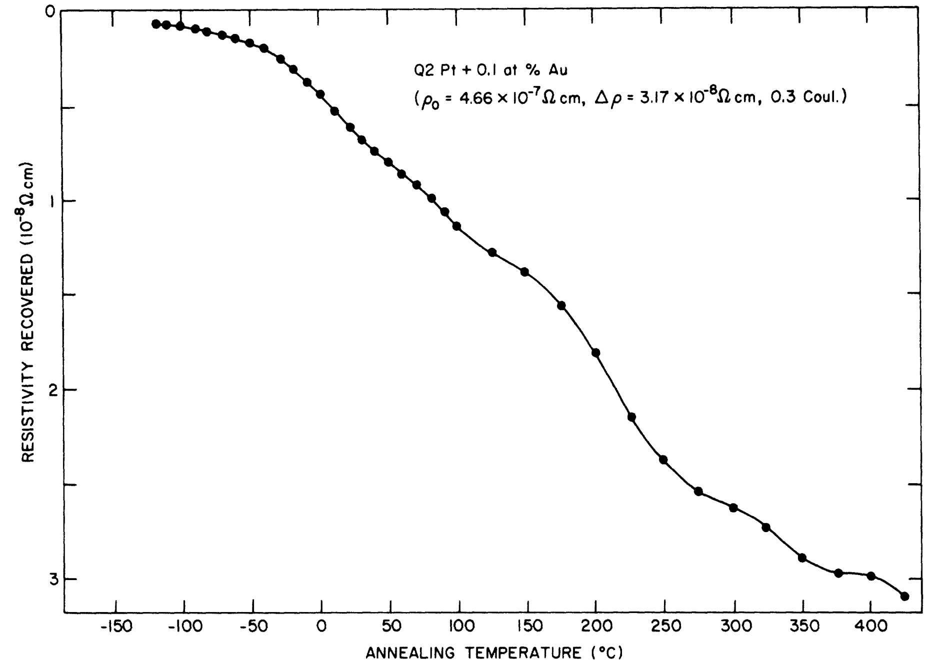

FIG. 8. Isochronal recovery of the electrical resistivity ($\Delta\rho$) for platinum-gold alloy specimen Q2 following 3-MeV electron irradiation near 80 °K, and annealed for 30 min every 10 °C below 100 °C and for 30 min every 25 °C above 100 °C.

followed exactly the variations in the current from the Faraday cage. This current was also monitored during the irradiation. In these irradiations, a (3 ± 0.025)-MeV regulated beam energy and a current of 7 μ A/cm^2 were used. The beam uniformity was checked by irradiating a glass slide mounted on a dummy block with a short pulse (a few seconds) of electrons. The uniformity in coloring due to the formation of color centers in the glass was checked by a photodensitometer. The fluctuation in the density of the light was less than 1%.

The temperature of the samples was maintained below 90 °K both during the irradiation and prior to subsequent annealing. For production data, samples were removed from the electron beam, measured at liquid-helium temperature, and reinserted into the beam. In this way damage production on the same sample can be monitored.

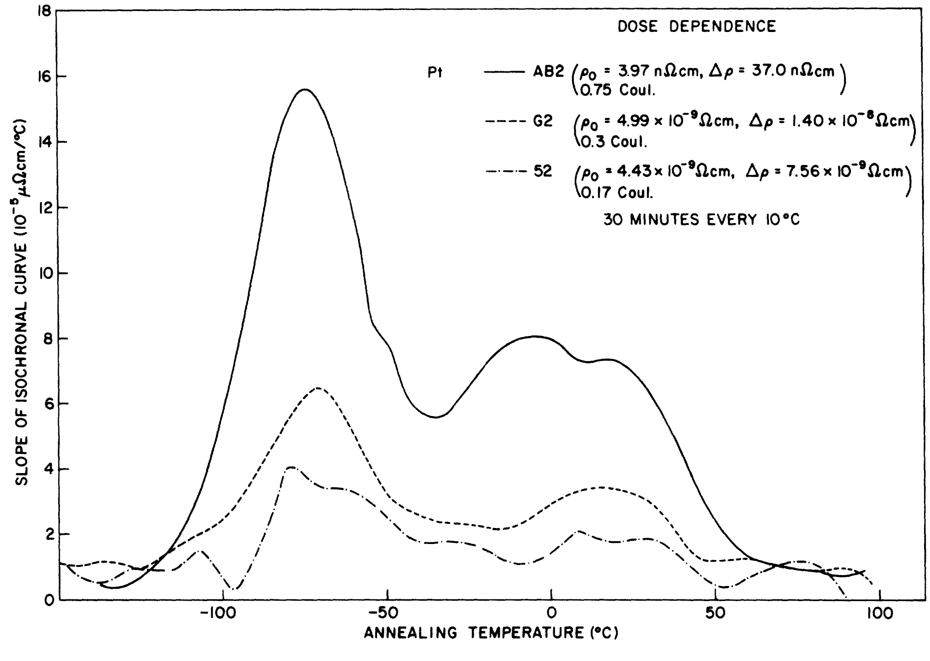

FIG. 9. Slope of isochronal curves for platinum specimens *AB*2, *G*2, and 52. This figure shows the dose dependence.

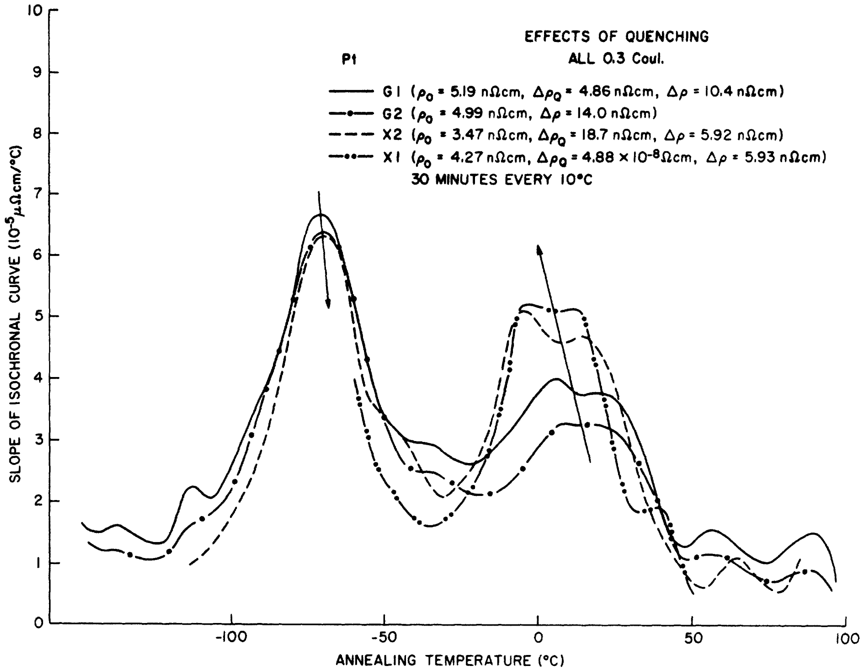

FIG. 10. Slope of isochronal curves for platinum specimens $G1$, $G2$, $X2$, and $X1$. This figure shows the effects of quenching.

The resistance of all samples was measured at liquid-helium temperature by immersing the sample block in this liquid. A standard potentiometric method was used. The current was supplied by a constant-current supply (Fluke model 318). The current supplied by this source was monitored by placing a 1-Ω standard resistor in series with it and measuring the voltage across this with a Leeds and Northrup K-3 potentiometer and electronic null detector. The voltage across the specimen was measured by a Rubicon thermofree six-dial microvolt potentiometer with a Rubicon photoelectric galvanometer. By reversing the current direction and taking the average of the readings for the two current directions, the effect of stray thermal emf's in the circuit was eliminated.

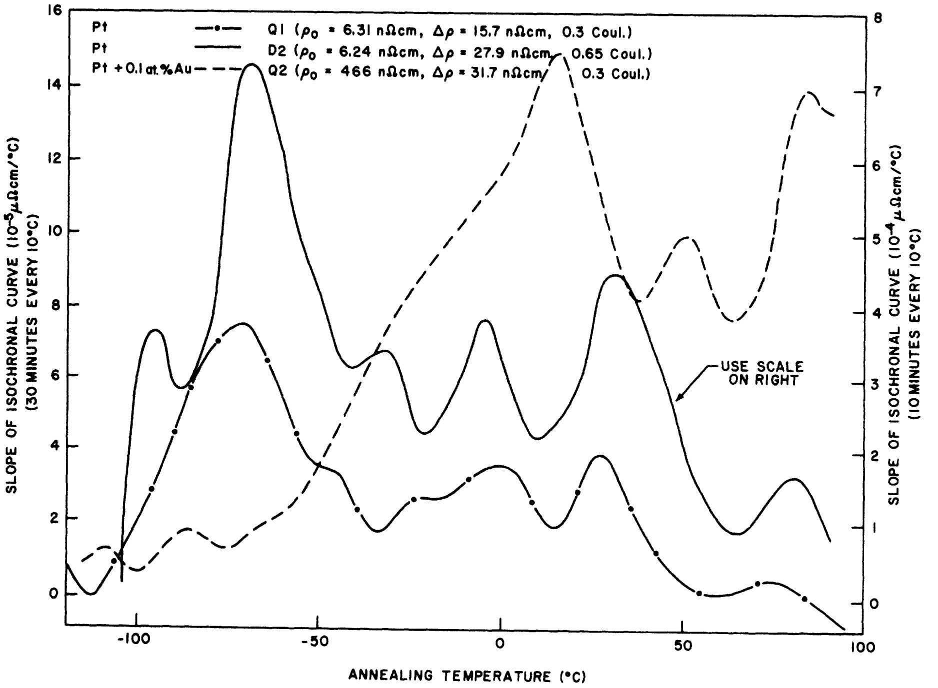

FIG. 11. Slope of isochronal curves for platinum and platinum-gold specimens $Q1$, $D2$, and $Q2$.

Sample blocks were annealed in an immersion bath. This consisted of a double Dewar system with liquid nitrogen as a cooling source and an electric heater for heating. The cooling was controlled by changing the vacuum in the inner Dewar. The heater was controlled by a deviation amplifier, set point, and silicon-controlled-rectifier power supply. Annealing bath liquids were changed in order to facilitate annealing over the wider range required by platinum samples. For temperatures from −155 to −40 °C, liquid Freon No. 12 (dichlorodifluoromethane) was used. Various mixtures of alcohol and water were used in the −40 to 90 °C range. For anneals between 100 and 200 °C, an oil bath was used. Above 200 °C salt baths were used. Annealing times and temperature intervals were selected on the basis of maximum resolution consistent with adequate determination of activation energies. The procedure followed, 10-C ° intervals for 30 min with measurements after 4, 10, 18, and 30 min, gave considerable detail in the annealing structure of stage II. Above 100 °C, samples were transferred to a specially designed sample container for higher-temperature anneals. They were carried out with 25-C ° intervals for 30 min. Since the thermocouple was mounted on a dummy sample within each block, corrections for heating and cooling times could be made by using the analysis of Grenning and Koehler.[13] These corrections were calculated on the CDC 3600 computer at Argonne National Laboratory.

III. EXPERIMENTAL RESULTS

A. Procedure

Table I gives the integrated flux used to irradiate each sample, the preirradiation resistivity ρ_0, the resistivity after quenching ρ_Q, the resistivity after irradiation ρ_r, and the treatment after irradiation. Figure 1 displays the resistivity increase as a function of dose for eight samples. Damage production in the alloy sample is very large compared to pure material. Samples containing vacancies in various concentrations suppress production. It is noted that after the quenched-in resistivity $\Delta\rho_Q$ reaches a certain value between 10.2 and 20.0 nΩ cm, no further suppression of production occurs. The points on the production curve (Fig. 1) for samples having the quenched resistivity greater than the above value ($X1$, $\Delta\rho_Q = 49.8$ nΩ cm; $X2$, $\Delta\rho_Q = 18.7$ nΩ cm; P2, $\Delta\rho_Q = 105$ nΩ cm) lie on a straight line. For all other samples this straight-line behavior is not followed.

We have attempted to fit the production data using the equation

$$\Delta\rho = A\varphi^N , \qquad (1)$$

where $\Delta\rho$ is the change in resistivity, A is a constant, and φ is the integrated flux. We conclude that Eq. (1) is a poor representation of the production processes for our irradiation. N was not a constant but changed with φ.

B. Annealing

In Table I the annealing treatment given to the samples is listed. Some samples were annealed at −150, −85, −55, and −15 °C for long times to study annealing kinetics. Most of the samples were annealed according to the scheme detailed earlier. Some of the typical resulting isothermal anneals are shown in Figs. 2–6 and typical derived isochronal anneals in Figs. 7 and 8. To obtain the isochronal curves, the raw data points are fitted with segments of seventh-order polynomials and these segments are joined together. For the slopes of these curves these seventh-order-polynomial segments were differentiated. The slopes of the isochronal anneals are shown in Figs. 9–11. The CDC 3600 digital computer at Argonne National Laboratory was used for all of the calculations and curve plotting.

Three main annealing peaks are observed; one at −75 °C, a double peak near 0 °C, and one near 325 °C.

Previously, within the author's knowledge, little or no evidence has been presented to indicate the presence of the annealing peak near −75 °C. In the present work this peak is reproducible and removes approximately a quarter of the resistivity increment produced by irradiation (Figs. 2, 3, 7). This peak shows almost no dependence upon vacancy concentration introduced prior to irradiation (Fig. 10) and, other than the expected increase in size, no dependence upon dose (Fig. 9). This peak is, however, strongly dependent upon impurity content and was completely missing in the platinum–0.1-at.%-gold alloy sample $Q2$ (Fig. 11).

The annealing stage near −75 °C is probably due to the breakup of interstitials from some residual impurity, because the temperature associated with this stage is not dependent on the dose nor the amount of quenched-in vacancies. Once an interstitial is freed from an impurity, the interstitial migrates rapidly, annihilating with a vacancy. The time required for the interstitial to reach a sink is not large compared to the breakup time; therefore, the peak temperature does not depend much on the dose or the concentration of vacancies. The activation energy associated with the peak at −75 °C is 0.48 eV. If this is due to the breakup of interstitials from some impurity or defects, the binding energy associated with the process would be $0.48 - 0.07 = 0.41$ eV, where 0.07 eV is the interstitial migration energy in platinum.[1]

The peak near 0 °C is quite broad and consists of at least two peaks; these were resolved to some degree for samples $AB1$, 52, $X2$, and $G1$ (Figs. 9, 10). Only a small temperature dependence on the preirradiation vacancy concentration and dose was observed. Appreciable recovery was observed above 35 °C for the alloy sample (Fig. 11). Between 35 and 175 °C for pure or quenched samples the recovery rate is quite small (Figs. 2, 3, 7). The addition of gold to platinum suppressed the peak near −75 °C and enhanced the recovery peaks near −10 and 15 °C. It particularly increased the 15 °C peak. The activation energy associated with the doublet near 0 °C is 0.74 eV. If this is due to the breakup of interstitials from some impurity or defects, the binding energy associated with this stage is $0.74 - 0.07 = 0.67$ eV, again using 0.07 eV as the interstitial migration energy in platinum.

The recovery peak at 325 °C is labeled as occurring in stage III as described above. A number of features concerning this stage are evident. This stage grows and shifts to lower temperatures with increasing dose and/or increasing preirradiation concentration of vacancies. Some broadening of the low-temperature side of this peak is observed. The alloy sample shows a double peak near 325 °C and strong shifting to lower temperatures. Because of the annealing steps used, good resolution of these peaks was not possible with this sample.

Activation energies were calculated by the change of slope method[14] for each sample. The average value of these energies at the peak temperature of the annealing are 0.48 ± 0.04 eV at −75 °C, 0.74 ± 0.04 eV at 0 °C, and 1.45 ± 0.04 eV at 325 °C.

The order of kinetics was determined using samples isothermally annealed for long times (~ 800 min). The analysis follows that used by the authors in an earlier paper.[15] The annealing near −75 °C followed a first-order process and the annealing near 325 °C followed a second-order process.

C. Determination of the Activation-Energy Spectrum

Primak has analyzed the annealing of defects assuming a continuum of activation energies. Calculations assuming first-order kinetics have been carried out in a number of papers.[16–20] Second-order processes were treated by Dworschak, Herschback, and Koehler.[11] A spectra with kinetic processes distributed in activation energy can be treated using the rather general assumptions that the frequency factor is nearly constant and that different processes have different activation energies. Details of the analysis for first- and second-order processes can be found in Ref. 21. A mixture of first- and second-order processes was also tried. Based on results from Sec. III B for the order of kinetics for annealing below 100 °C, first-order kinetics were assumed. The results are shown in Figs. 12–14. For the annealing above 100 °C, second-order kinetics were assumed. The following results are shown in Figs. 15 and 16: (i) One peak near 0.5 eV corresponding to the recovery stage near −75 °C is found in the spectra. (ii) A double peak around 0.75 eV corresponding to the double annealing stage near 0 °C is also found in the spectra. (iii) For the platinum-gold alloys, the peak around 0.5 eV is suppressed and the peak near 0.75 eV is greatly enhanced (see Fig. 14). (iv) For specimen *G*1, one peak is near 1.48 eV and a very broad peak is centered at 1.6 eV. This broad peak could consist of two peaks. (v) For the platinum-gold alloys, a broad peak centered at 1.4 eV and a sharp peak near 1.7 eV were found.

IV. DISCUSSION AND CONCLUSIONS

In the annealing of most fcc metals four recovery stages occur. They are labeled I, II, III, and IV starting from low temperature. Stage I occurs in the range from 10 to 100 °K. Stage II is an ill-defined stage which is probably absent in very pure materials. Stage III for aluminum, copper, silver, and gold occurs at a temperature which is 0.22 ± 0.02 times the melting temperature. This predicts that stage III should occur at 170 °C (443 °K) in platinum. The present experiments show annealing stages at 0 and at 300 °C. Most previous research workers have assigned the 0 °C annealing to stage III. Actually its sensitivity to purity suggests that it is a stage-II peak and that the 300 °C peak is the intrinsic stage-III peak.

The activation energy associated with the stage near 325 °C was calculated to be 1.45 ± 0.06 eV which is the same as that found in quenching experiments.[22] This suggests that the stage-III annealing occurs by the migration of vacancies. Similar results have been observed in other metals. In pure Al, the activation energy associated with stage III was found to be 0.62 ± 0.02 eV which is close to that found by quenching experiments.[23,24] For pure gold the activation energy associated with the annealing of the quenched-in defect is 0.85 ± 0.02 eV and that associated with stage III in electron-irradiated specimens is 0.85 ± 0.03 eV.[25] The activation energy associated with stage III in quench-plus-irradiated specimens in 0.86 ± 0.02. These are the same within the experimental error. Since the activation energies associated with stage III has the same value as those from quenching experiments, the defects responsible for stage III in platinum are likely to be single vacancies as in aluminum and gold.

Shimomura[26] irradiated pure gold at 130 °K with 3-MeV electrons. He thinned the specimens at 2 °C (in some cases at −30 °C) and observed many interstitial clusters. The cluster size shrank as the annealing of stage III proceeds if he annealed the thick specimens, but the clusters did not shrink if he annealed a thin film. This was interpreted to

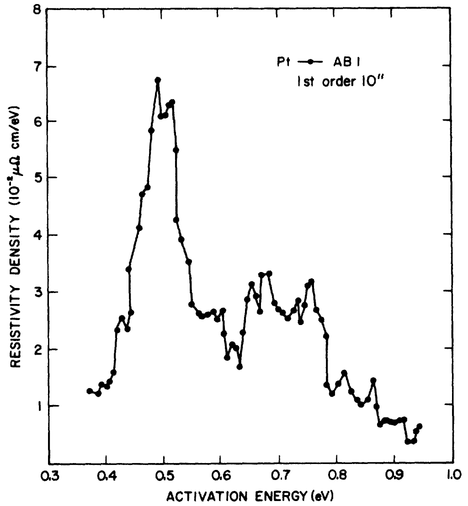

FIG. 12. Activation-energy spectrum for specimen *AB*1 (first order).

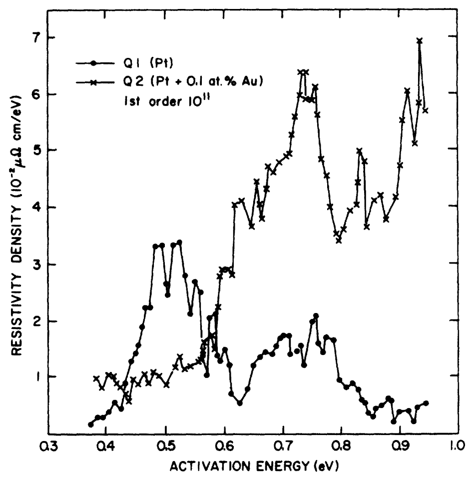

FIG. 14. Activation-energy spectra for specimens *Q*1 and *Q*2 (first order).

mean that the vacancies move to clusters during stage III in bulk material, but in thin films some vacancies escape to the surface. Hence there are not enough to eliminate interstitial clusters. In Shimomura's experiment the resistivity estimated from the size and the number of interstitial clusters is not enough to explain the resistivity observed in the experiment. About 80% of the resistivity may come from interstitial clusters which are too small to observe by an electron microscope.

In silver the activation energy observed in the irradiation experiment is lower than that for single-vacancy migration. Gordon and Koehler[27] irradiated pure silver with 3-MeV electrons and obtained the activation energy of 0.67 ± 0.0 eV. Doyama and Koehler[28] quenched pure silver and obtained the activation energy 0.83 ± 0.0 eV for the migration of single vacancies. Dworschak, Herschbach, and Koehler[11] irradiated pure copper with protons at liquid-nitrogen temperature and obtained $E_{\text{III}} = 0.70 \pm 0.04$ eV, which is lower than the values for the migration of single vacancies (0.88 ± 0.1 eV). At present the reason is not clear.

When the results presented here for platinum are compared with recovery stages in aluminum, gold, silver, and copper, the so-called state III in platinum appears to correspond to stage II in the metals above. Both the dose and impurity dependence support this conclusion. Furthermore, we believe the so-called stage IV in platinum to be associated with the migration of free vacancies to interstitial clusters (large and small). The dependence of this

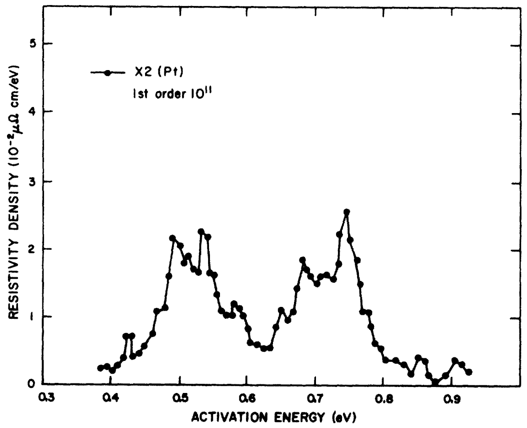

FIG. 13. Activation-energy spectrum for *X*2 (first order).

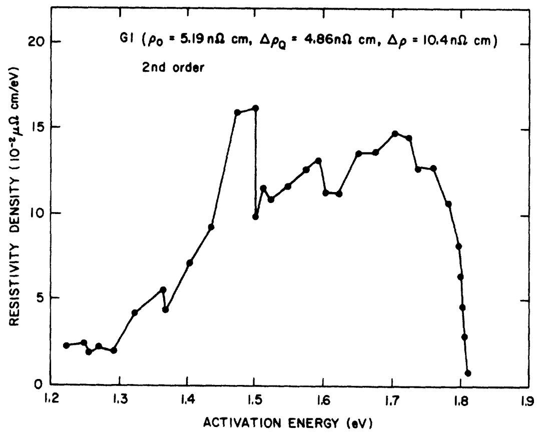

FIG. 15. Activation-energy spectrum for specimen *G*1 (second order).

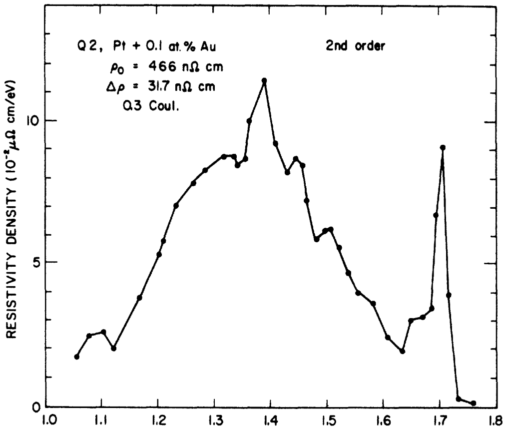

FIG. 16. Activation-energy spectrum for specimen *Q*2 (second order).

stage on vacancies induced by quenching prior to irradiation and the correlation between the activation energy from these irradiation experiments and the activation energy derived from independent quenching studies support our contention that this stage should be labeled stage III. In this way the physical phenomena associated with this stage will correspond to those in other metals such as aluminum and gold.

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