Influence of Plastic Deformation and of Substitutional Gold on Defect Production and Recovery in Platinum*

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Both the production of defects in Pt by deuteron irradiation at 6°K and the subsequent thermally activated recovery of these defects are perturbed when Pt is deformed or alloyed before irradiation. The atomic rearrangements that occur in the various recovery substages are known with perhaps less ambiguity in Pt than in any other metal. This knowledge is used to analyze in detail the interactions between the defects injected before the irradiation and the atoms displaced during the irradiation that produce these perturbations.

I. INTRODUCTION

OVER the past 15 years many investigations have been carried out in which pure, well-annealed specimens have been irradiated at low temperature and the subsequent thermally assisted recovery of the defects injected by the irradiation carefully studied.[1,2] These investigations have shown that defect recovery is not a simple process. As many as 22 annealing substages have been resolved in some systems.[3] To better understand these processes many investigations have been performed in which specimens have been doped before the irradiation with impurity atoms or with defects injected by plastic deformation. Typical results of such investigations have been given.[4–7] It was hoped that the nature of the radiation-injected defects whose thermally assisted motion produces recovery could be determined from their interaction with the dopants. Because of the complexity of these interactions, an unambiguous picture has not yet emerged from these investigations. In addition, these investigations revealed that defect production is also not a simple process. The initial production rate and the change in production rate with defect concentration depend strongly upon the treatment given to the specimen before irradiation.[8]

As we pointed out in a previous paper,[9] the simplest dopant to use in the study of low-temperature recovery after irradiation is the lattice vacancy. That paper described the effects of different quenched-in vacancy concentrations on defect production by 20-MeV deuterons in Pt and on thermal recovery of these defects. Based on that work we identified the particular defect configurations that recover in the substages seen during anneal of irradiated Pt. Our investigations have been aided by the use of a technique not available to most previous workers. We are able to simultaneously irradiate an anneal as many as six different specimens. This allows us to make detailed comparisons of the effect of various preirradiation treatments free from uncertainties arising from differences in annealing treatments.

Platinum is a particularly valuable metal to use in the study of the effects of deformation on defect production during irradiation and on the recovery of these defects. After room-temperature deformation, not only is the dislocation density increased but also supersaturations of point defects remain in the lattice. In the present work, the concentration of these defects was as high as 10^{-4}. The point defects can be removed by selective annealing treatments with but small changes in the dislocation density. This allows us to separate effects primarily due to point defects from those primarily due to dislocations. The addition of a small concentration of Au to Pt before irradiation produces changes in production and recovery of Frenkel pairs that are qualitatively similar to changes produced by some substitutional impurities in Al and Cu. Since the specific recovery mechanisms in irradiated Pt have been well identified by the aid of quenched-in vacancies[9] the specific atomic processes affected by substitutional Au atoms can be determined.

In Sec. II we describe the significant experimental details of this investigation; the results obtained are presented in Sec. III, and these findings are discussed in Sec. IV.

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II. EXPERIMENTAL

The unalloyed specimens were prepared from wires chemically purified to 99.999% purity and supplied hard drawn to 0.002 and 0.004 in. diam.[10] Before deformation the wires were refined thermally in our laboratory, as described in an earlier publication, to give a total impurity concentration less than 1 ppm.[11] This treatment produced a bamboo-type grain structrue with the grain size approximating the wire diameter. All deformations were at room temperature. Some specimens were pulled to 9% extension, others were rolled between Pt foil to $\frac{1}{2}$ or $\frac{1}{3}$ the original diameter, and the remainder were drawn through diamond dies to 60% reduction in area. Some deformed specimens were annealed for 30 min at 310°C. This ageing treatment removes most of the point defects but retains most of the dislocations produced by the deformation. It does not reduce the yield strength of deformed Pt.

The annealed samples irradiated in each run were prepared from the most heavily deformed wire used in that run by giving it an anneal identical to the original anneal. Following this second anneal, the resistivity at 4.2°K was never greater than 4.5×10^{-9} Ω cm as compared to resistivities of $(3.7\pm0.25)\times10^{-9}$ Ω cm in specimens before deformation. The data of Huebener show that the reduction of thickness in the most heavily deformed specimens should increase the resistivity at 4.2°K by about 0.5×10^{-9} Ω cm.[12] We conclude that the impurity concentration in the deformed and annealed specimens is at most only slightly greater than it was before deformation.

Care was taken to avoid contaminating the wires during deformation. The wires themselves and the foils and dies were washed in nitric acid before deformation and the wires were again washed after deformation to remove surface contamination that could be injected into the lattice by deformation or irradiation. A wire drawn to 60% reduction in area and heated for 1 h at 700°C had a resistivity at 4.2°K of 4.4×10^{-9} Ω cm as compared to 5.9×10^{-9} Ω cm for as-received 99.999% pure wire given the same anneal. This indicates that our specimens, as deformed, probably had impurity concentrations smaller than 10 ppm after deformation.

The Pt–0.1%-Au alloy was supplied hard-drawn as 0.010-in.-diam wire.[13] This was annealed for 30 min at 1000°C and drawn without further annealing to 80% reduction in area. Some of this wire was then annealed at 1400°C for 20 min, cooled to 500°C over a period of 1 h, and quenched to room temperature. This treatment should retain the Au in solid solution without quenching in many lattice vacancies.[14,15] Specimens made from wire given the anneal after drawing are considered the annealed alloy; specimens not given this anneal are the deformed alloy.

The irradiations were carried out at the Argonne National Laboratory 60-in. cyclotron, using the crysotat and auxiliary equipment previously described.[16] A deuteron beam current of 35×10^{-10} d/cm² sec was used for the first irradiation in this investigation, run 5. With such a current there was no recovery below 11.5°K for the unaged specimen drawn to 60% reduction in area. In subsequent irradiations the deuteron current was held below 16×10^{10} d/cm² sec and recovery measurements show that all specimens remained below 7°K during irradiation with the exception of the alloys. The temperature of these during irradiation was between 8 and 14°K.

The deuteron beam was spread so that it filled the beam tube and covered a much larger area than the entrance window to our cryostat. In this way we generally had a nearly uniform flux through our specimens; run 8 was probably the least successful in that respect. The effect of beam inhomogeneities and fluctuations is least for comparisons of pair of specimens located at positions n and $n+3$ ($n=1$, 2, or 3) since the member of these pairs have almost identical locations relative to the beam.[17] Not all of the specimen positions were used for the work reported in this paper, but all except one of the deformed or alloyed specimens were paired (n and $n+3$) with pure, annealed specimens or with other deformed specimens.

The damage was measured by the change in electrical resistivity of the specimens. Relative changes in the resistivity of any one specimen could be measured to better than 5×10^{-12} Ω cm but the absolute resistivity increment from a given deuteron flux is uncertain by about 10%. These uncertainties arise from beam inhomogeneities and an imperfect knowledge of the length of specimen that subtends the beam. The largest and smallest resistivities added by irradiation in this work were 33×10^{-9} and 3.4×10^{-9} Ω cm.

The annealing procedure was the same as in our earlier work.[18] The specimens were pulsed to a monotonically increasing series of annealing temperatures, held at temperature for 10 min, and then cooled to below 5°K for resistance measurements. The time to heat from the previous annealing temperature to the next annealing temperature was less than 30 sec and to cool to the previous temperature was less than 1 min. Heating and cooling times were this long only above 150°K; below 30°K cooling times were less than 20 sec.

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[11] J. J. Jackson, in *Reinstoffprobleme*, edited by E. Rexer (Akademie-Verlag, Berlin, 1966).

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[16] K. Herschbach, Rev. Sci. Instr. **37**, 171 (1966).

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[18] K. Herschbach and J. J. Jackson, Phys. Rev. **153**, 694 (1967).

III. RESULTS

A. Defect Production

The various treatments given before irradiation to the specimens studied in this investigation are shown in Table I. This table also gives the resistivity increment in each specimen due to the irradiation and (when measured) the resistivity increment in the first 10–20% of irradiation. These last two sets of entires show that radiation annealing (the decrease in damage rate with increase in defect concentration) was altered by some of the treatments. To bring out these differences we have plotted in Fig. 1 the ratios of the production rates of several pairs of specimens. The pairs are chosen so that beam fluctuations affect both members nearly equally. The absolute values of the ratios are affected by the errors in our measurements of absolute resistivity increments but the relative changes in the ratios are independent of these errors.

The data show that, within the limit of error, plastic deformation at room temperature does not change the initial damage rate from that in pure, annealed platinum. The rate is significantly increased by deformation followed by ageing at 310°C. The initial rate is higher in fully annealed Pt–0.1%-Au alloy compared with that in pure Pt, but is lower in the deformed alloy.

Earlier carefully controlled studies of radiation annealing showed that the instantaneous damage rate in annealed platinum at a dose of 22×10^{14} d/cm^2 is about $3\frac{1}{2}$% less than the rate at 4.4×10^{14} d/cm^2.[17] Quenching rapidly from high temperature greatly increases radiation annealing, but the increase is negligible for the vacancy concentration quenched into specimen 13-6.[9] Figure 1 shows that after heavy plastic deformation the damage rate decreases very slightly more than in pure annealed Pt, that deformation followed by ageing at 310°C gives an 11% decrease in production rate over the range of measurements, and that alloying with 0.1% Au results in a decrease in rate intermediate between those from the other treatments. Deformation decreased the magnitude of radiation annealing in the alloy.

B. Recovery in Stage I

Isochronal recovery between 10 and 35°K of a pure Pt specimen well annealed before irradiation is shown as the solid line in Fig. 2. Annealing data for a deformed specimen and for an alloyed specimen are also shown. Differential recovery curves for the three specimens of Fig. 2 and for specimens given treatments P and R2+A are shown in Fig. 3. From the latter figure it is evident that much of the structure resolvable in stage-I recovery of undeformed Pt is lost after deformation. In particular, for the heavily deformed specimens, R3 and D60, the recovery peaks at 10 and 15°K are lower and broader than in undeformed Pt, and more than half of the total recovery takes place between 18 and 28°K in a broad peak with no resolved structure. The recovery of much less heavily deformed specimen P lies between that of undeformed specimens and that of specimens receiving treatments R2, R3, and D60.

Because of the loss of coolant, we have no annealing data for alloyed specimens between 8 and 14°K. From that temperature to 22°K Pt–0.1%-Au has qualitatively the same recovery features as does pure undeformed Pt; similar substructure is resolved although fractional recovery is reduced. The ratio of the maximum heights of the three recovery substages observed in the alloy to the heights of the corresponding substages in pure Pt are 1.0, 0.77, and 0.57 in order of increasing temperature.

Details of the effects of different treatments on recovery can be displayed free from errors due to

TABLE I. Specimen preparation and resistivity increments. Symbols: D60, drawn to 60% reduction in area; D80, drawn to 80% reduction in area; R3, rolled to $\frac{1}{3}$ thickness; R2, rolled to $\frac{1}{2}$ thickness; P, pulled to 9% extension; A, aged 30 min at 310°C; Q 3.5, quenched to give a resistivity increase of 3.5×10^{-9} Ω cm.

Run	Specimen position	Treatment before irradiation	Total dose 10^{14} d/cm^2	Resistivity increment 10^{-9} Ω cm: Initial 4.4×10^{14} d/cm^2	Resistivity increment 10^{-9} Ω cm: Total $\Delta\rho_I$
5	2	D60			31.1
	5	D60+A	44		33.3
	6	Annealed			31.5
8	1	R3	4.4	3.40	3.4
	4	Annealed		3.50	3.5
	2	R2		3.03	24.8
	3	R3	37.4	3.11	27.9
	6	Annealed		3.12	25.2
11	1	R2+A	22	3.43	16.3
	4	Annealed		2.98	14.9
13	1	Alloy annealed		4.19	21.0
	2	P		3.15	15.3
	3	R2+A		3.22	14.9
	4	Annealed	22	2.93	15.0
	5	Alloy D80		2.64	12.4
	6	Q 3.5		2.87	14.4

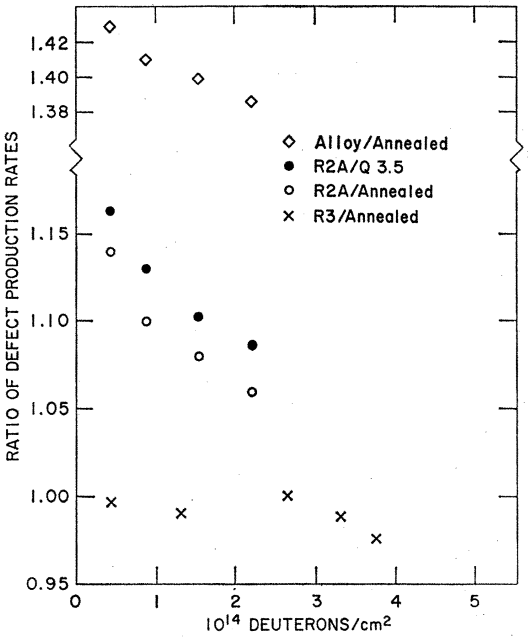

FIG. 1. The effect of various treatments on the initial defect production rates in Pt irradiated near 6°K with 20-MeV deuterons.

irreproducibility in annealing times and temperatures in a plot of the difference in recovery between two specimens given different preirradiation treatments but irradiated and annealed together. We have described this technique more fully in an earlier paper.[19] The differences in percent of damage remaining in several pairs of specimens irradiated and annealed simultaneously are plotted in Fig. 4. One member of each pair was pure, undeformed Pt, the other member was deformed or alloyed before irradiation. The treatments chosen for Fig. 4 show the effect of alloying, of large and of small deformations, and of deformation followed by ageing. The particular specimens plotted are those for which we have the most extensive data. The other

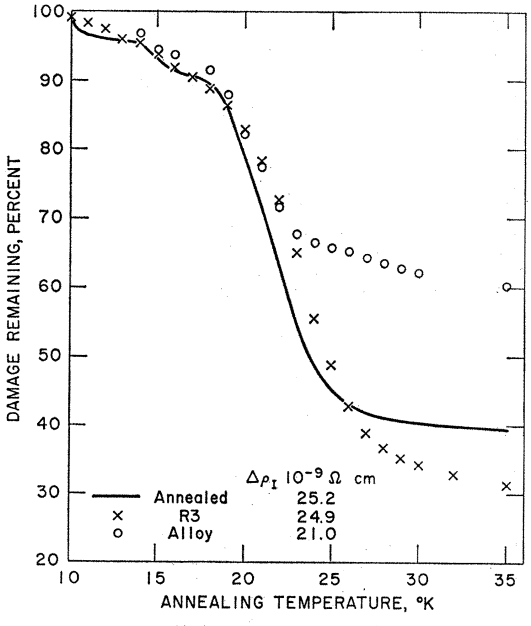

FIG. 2. The effect of deformation and of alloying with Au on stage-I recovery of dueteron-irradiated Pt.

[19] K. Herschbach and J. J. Jackson, Phys. Rev. 158, 611 (1967).

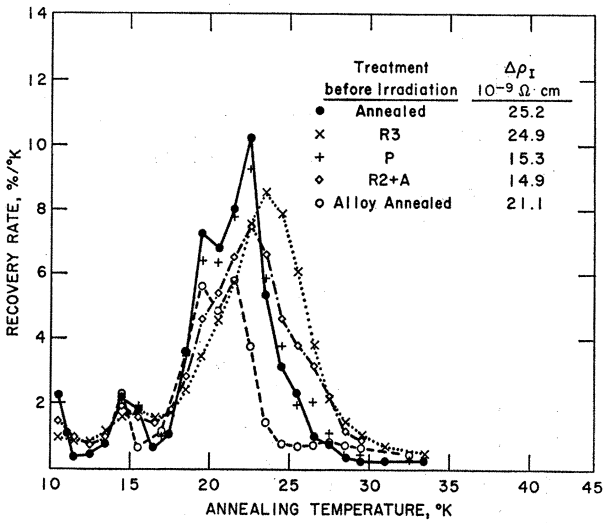

FIG. 3. The effect of various treatments on the stage-I recovery spectrum of Pt.

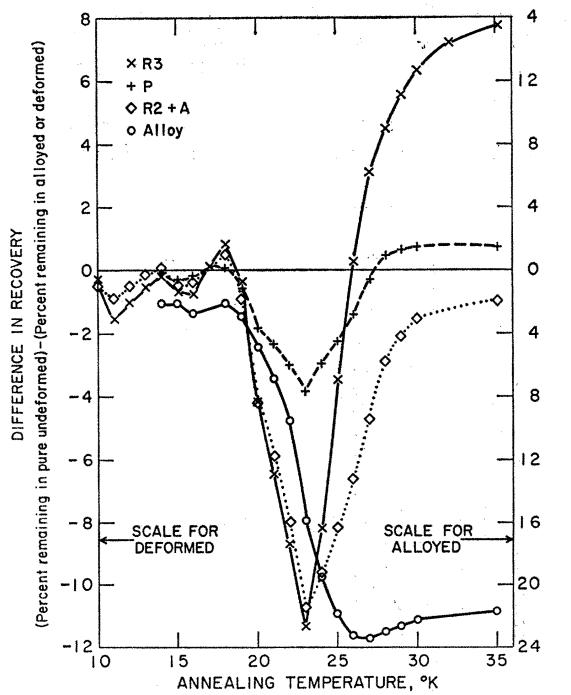

FIG. 4. The differences in recovery (the percentage of damage remaining in an untreated specimen minus the percentage remaining in specimens given various treatments before irradiation) versus the annealing temperature in stage I.

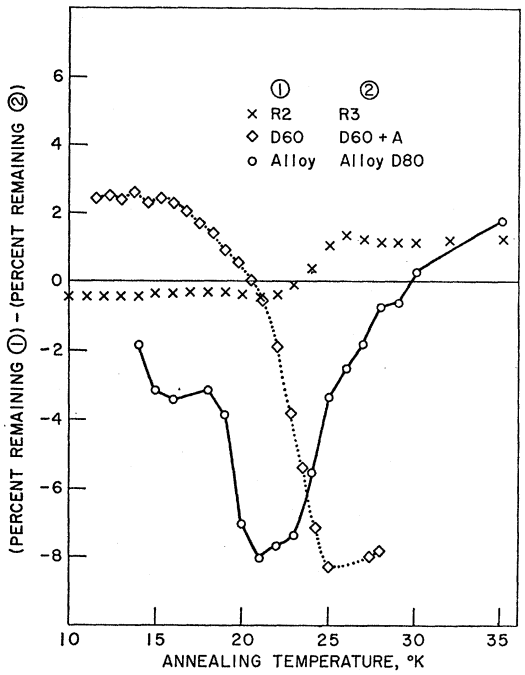

FIG. 5. The differences in recovery between specimens given various treatments before irradiation as a function of annealing temperature in stage I.

types of large deformations, D60 and R2, gave curves quite similar in shape to those found after R3. Several additional pairs are plotted in Fig. 5. These show explicitly the effect of deformation on recovery of the alloy, the effect of ageing at 310°C after treatment D60, and the effect of increasing the deformation from R2 to R3.

At 19°K the annealing rate in all deformed specimens decreases markedly relative to the rate in undeformed

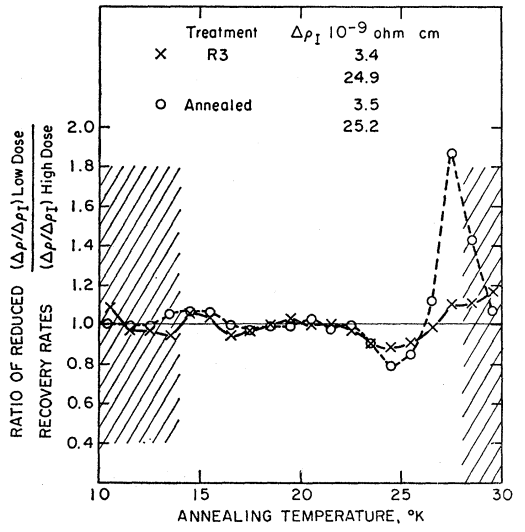

FIG. 6. The ratio of the normalized recovery rates $[(\Delta R/\Delta T)/\Delta\rho_I]$ for two pairs of specimens given different doses.

Pt. All deformed specimens except the alloy are farthest behind at 23°K. The specimen given the smallest deformation, P, is retarded much less than the more heavily deformed specimens. Differences in magnitude of retardation are not so great among any of the more heavily deformed specimens. Above 23°K the annealing rate is greater in deformed than in undeformed Pt. The annealing rate above 23°K in deformed Pt is reduced by ageing. Recovery in the alloy is much smaller than in pure Pt between 19 and 26°K.

At the conclusion of stage I, 35°K, the annealing rate in pure Pt has fallen to but a few percent of the maximum rate, deformed unaged specimens have all recovered more than specimens that were not deformed, deformed and aged specimens have recovered less, and the alloys are 20% behind pure, undeformed Pt in percent of total damage recovered.

The dependence of recovery on dose has been studied in only one pair of deformed specimens. These were given treatment R3 and irradiated in run 8 to resistivity increments of 3.4 and 24.9×10^{-9} Ω cm. The recovery of these two specimens is compared in Fig. 6 by means of the ratio plot devised by Sosin.[20] A ratio plot is also given in Fig. 6 for two annealed specimens irradiated in run 8 to 3.5 and 25.2×10^{-9} Ω cm resistivity increments. Below 23°K both ratios are very close to unity. Above that temperature both ratios fall below unity and then rise above unity, but the changes for the undeformed pair are greater than for the deformed ones.

C. Recovery above Stage I

The specimens from runs 8, 11, and 13 were annealed at temperatures up to 300°K, the highest practicable annealing temperature for our cryostat. Run 5 was ended at 28°K by a failure in the cooling coil. Unfortunately, Pt must be heated to near 700°K before all defects injected by deuteron irradiation recover; about 15% of the added resistivity remains at 300°K. Isochronal recovery curves between 30 and 300°K are shown in Fig. 7 for pure, for deformed, and for alloyed Pt specimens. The differences in percent recovery of several pairs of specimens are shown in Fig. 8.

For pure, undeformed Pt the annealing rate decreases fairly steadily in the interval 35–150°K except for a small substage near 110°K. There is very little recovery between 150 and 240°K. Above that temperature the annealing rate once again increases. In the terminology normally used in speaking of recovery of fcc metals[1] the region between approximately 35 and 240°K is stage II and the interval 240–300°K lies in stage III.

In stage II, up to perhaps 110°K, all deformed specimens recover faster than do the corresponding undeformed ones. The total amount of recovery increases with the amount of strain through treatments

[20] A. Sosin and K. R. Garr, Phys. Rev. 161, 664 (1967).

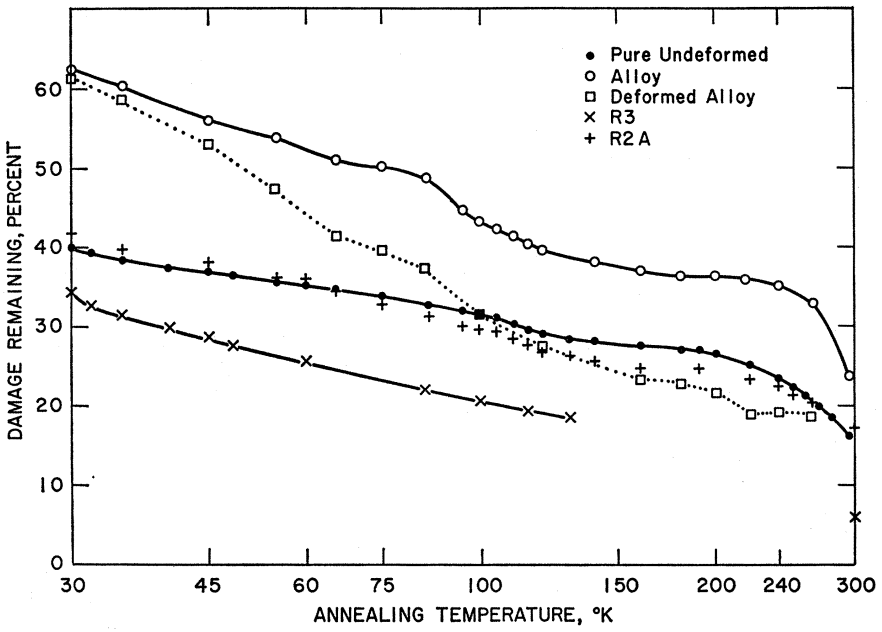

FIG. 7. Recovery in stages II and III of Pt given various treatments before irradiation with 20-MeV deuterons.

P, R2, and R3. The difference in fractional recovery between deformed and undeformed specimens reaches a maximum in stage II as is also observed in a comparison of quenched with unquenched specimens. The greater the magnitude of this maximum the lower is the temperature at which it occurs. The annealing substage at 110°K is broadened by deformation. In the lightly deformed and the deformed-then-aged specimens a smaller percentage of the total damage recovered between 240 and 300°K than recovered in undeformed Pt. From 35°K up to at least 130°K the effect of dose is similar in deformed (R3) and undeformed specimens.

Between 30 and 240°K about 26% of the resistivity added by irradiation recovers in the undeformed alloy, as compared to 14% in undeformed pure Pt. The enhancement in recovery occurs mainly in two substages, one near 40°K and the other near 95°K. Since damage production was significantly greater in the alloy, the amount of resistivity recovered in stage II is double that in pure Pt. The recovery substage seen near 110°K

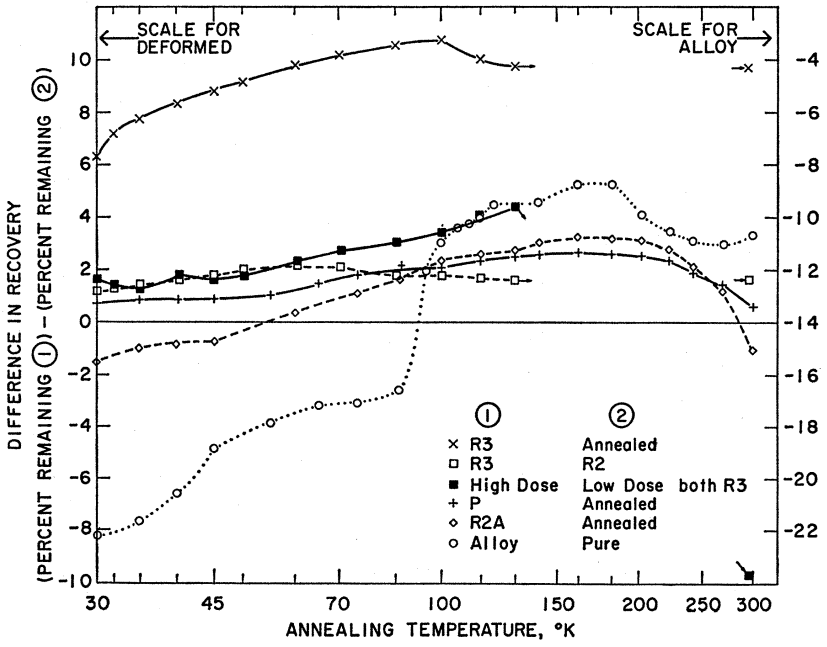

FIG. 8. The differences in percent recovery between pairs of specimens as a function of annealing temperature in stages II and III.

in pure Pt is also present in the alloy. The deformed alloy has much the same recovery spectrum as the annealed alloy but the fractional recovery is increased by the deformation. The actual resistivity decrease in stage II, however, is the same in both alloyed specimens within the precision of our measurements. Above 240°K there was more recovery in the alloyed specimens than in pure Pt.

IV. DISCUSSION

For the purposes of this paper, dislocations are well described as cores of large lattice distortion set into elastically strained regions. The dislocations are split into two partials connected by a region of stacking fault. Both the core regions and the stacking faults disrupt the lattice regularity needed for the propagation of focusing collision sequences. In the elastic region the interaction between a dislocation and a point defect separated by a distance r derives from a potential varying as

$$V=(A\sin\theta+B\cos\theta)/r\,. \tag{1}$$

The magnitudes of A and B are functions of the type of dislocations and point defects under consideration.[21] These interactions reduce the degeneracy of sites for interstitial atoms so that the energy of an interstitial is greater in some sites and lower by the same amount in an equal number of other sites.

When interstitials and vacancies are close together the energy barrier for a jump of the interstitial toward the vacancy is significantly less than for a jump away. There are several distances of separation of these close Frenkel pairs that are stable at 4°K. The energy needed for recombination of some of these Frenkel pairs is changed by dislocation-interstitial interactions. During an isochronal anneal these perturbations spread the recombination of Frenkel pairs nearly symmetrically over a wider range of temperatures than would be the case in a lattice free from dislocations.

When interstitials are sufficiently far from vacancies so that the first jump is not significantly biased toward a sink, then the interstitial migrates freely and may sample a representative portion of the lattice before reaching a sink. Recovery due to the migration of such interstitials is sensitively detected by observing changes in annealing resulting from changes in the general lattice.

Interactions between dislocations and interstitials reduce the number of interstitials that diffuse to vacancies in a given temperature-time pulse. Although as many barriers to migration are lowered as are raised, the Boltzmann function that governs thermally activated migration increases the time of dwell behind raised barriers more than it reduces the time behind lowered barriers. The migrating defects are trapped in certain regions of the lattice. The ratio of the number of distinct sites visited during an annealing pulse at temperature T in a lattice containing sets of sites at which the energy of interstitials are changed by $\pm b_i$ to the number in a lattice without such perturbations is of the form

$$\frac{R_N}{R_0}=\left[1+\sum_{i=1}^{N} f_i\left(\cosh\frac{b_i}{kT}-1\right)\right]^{-1}. \tag{2}$$

The summation is over all levels b_i and the f_i are functions of the number and distribution of the sites perturbed by an energy b_i. The f_i are increased by plastic deformation.

The principal effect of adding substitutional impurities to a lattice is the creation of new states into which interstitials can be bound. Substitutional impurities also disrupt long-range propagation mechanisms.

Earlier investigations[9] on irradiated Pt have proven that recovery below 23°K is by close pair recombination. Above that temperature interstitial free migration to sinks becomes important. In stage II, annealing represents the release of interstitials from various traps and their migration to sinks. In stage III, a second type of interstitial bcomes mobile, and in stage IV, the remaining point defects are removed by the migration of vacancies to sinks; among these sinks are interstitial clusters formed in stage III. The findings of this investigation will be interpreted on the basis of the processes described above. We first consider the effects of the various treatments on defect production and later the effects on the thermally assisted recombination of Frenkel pairs.

A. Defect Production

1. Influence of Deformation and Partial Annealing

In deuteron irradiation studies of Al, Au, Cu, and Ag we have found that room-temperature plastic deformation, before the irradiation, increases both the initial rate of resistivity increase and the amount of radiation annealing and decreases the percentage of total damage that recovers in stage I.[22] The same results have been seen in electron[6] and neutron[8] irradiated Cu. When Pt is deformed at room temperature and irradiated with deuterons the damage rate and the amount of radiation annealing are the same as in undeformed Pt and the percentage of damage that recovers in stage I is increased; however, ageing the deformed Pt at 310°C before irradiation yields the results seen when the other four metals are deformed and irradiated.

After extensive deformation at room temperature only one recovery stage is seen in pure Al, Au, Ag, and Cu.[23] This is stage V, dislocation rearrangements and recrystallization. When Pt is so deformed in addition

[21] J. Friedel, *Dislocations* (Pergamon Press, Ltd., London, 1964).

[22] J. J. Jackson and K. Herschbach, Brookhaven National Laboratory Report, 1967 (unpublished).

[23] L. M. Clarebrough, M. E. Hargreaves, and G. W. West, Proc. Roy. Soc. (London) A232, 252 (1955).

to stage V, two other recovery stages, III and IV, are resolved. When heavily deformed Pt is aged for 30 min at 310°C, about $\frac{2}{3}$ of the resistivity added by the deformation is removed, but the hardness is unchanged indicating that a large decrease in point-defect concentration but no extensive rearrangement of dislocations has taken place. Both the remaining excess resistivity and the total increase in hardness recover in stage V. The characteristic effects observed upon irradiation of Cu deformed at room temperature are seen in Pt so deformed only when the point defects injected by the deformation are removed.

Our experiments in which deformed and undeformed specimens of Au,[17] of Al,[19] and of Ag[22] have been irradiated and annealed together show that although a smaller fraction of the total damage recovers in stage I in the deformed metal, the amount of resistivity decrease in stage I is the same in deformed as in undeformed specimens. This is also true for Pt deformed and aged at 310°C. The amount of recovery in stage II of pure fcc metals is not much changed by deformation before irradiation. The excess resistivity produced in deformed over that in undeformed noble metal or Al specimens by deuteron,[17,19] neutron,[8] or electron[6] irradiation is recovered in stage III. Thus, the sum of the numbers of close pairs and of crowdions and the number of trapped interstitials produced by irradiation are very little changed in deformed fcc metals lacking excess point defects from the number produced by an identical irradiation in undeformed specimens. Essentially all of the excess production of defects in the former consists of split interstitials and their associated vacancies.[24]

In an earlier paper[9] we showed that recovery in Pt after deuteron irradiation is best fitted by a model in which most of the interstitials that recover in stage III are produced during irradiation and not during low-temperature annealing. Since the extra resistivity produced by irradiation of deformed fcc metals recovers entirely in stages III and IV, we propose that this extra resistivity represents extra production during irradiation of split interstitials and their associated vacancies. Dislocations provide a set of special sites where this production takes place.

This enhancement cannot be explained on the basis of a change in the threshold for direct displacement. Lattice-binding energies are reduced appreciably only in the cores of the dislocations. Diffusion data gives a mean binding energy there about $\frac{1}{2}$ the value in the undisturbed lattice.[25] Even for an unrealistically high dislocation density of 5×10^{12} cm/cm^3, fewer than 0.1% of the atoms are in cores. The number of direct displacements in such regions when irradiated with 20-MeV deuterons is about 50% greater than in an equal volume of perfect lattice where the displacement threshold is about 40 eV.[26] Thus the production enhancement from this cause is less than 0.05%, as contrasted to our observed 12–15% enhancements.

It is more likely that the enhancement results from defocusing of collision chains at dislocations. In this way one event can sample a much larger section of the lattice and has a greater chance of interacting with a severely disturbed region. We earlier proposed a similar process to explain enhancements when the lattice contains quenched-in vacancies.[9] Since collision sequences are defocused at stacking faults the number of sites for enhancement is much greater than the number of core atoms.

The magnitude of the initial enhancement is given by

$$N_D-N_U=fl_0\rho S\sim N_U/7\,. \tag{3}$$

In this expression N_D and N_U are the initial defect production rates in deformed and undeformed Pt, f is the ratio of sequences that can produce extra defects upon interacting with dislocations to the defects produced in undeformed material, l_0 is the initial mean length of these sequences, and S is the cross section per unit dislocation length for the interaction. Equation (3) can be used to estimate the parameters involved in this model. If f is taken[7] as $\frac{1}{2}$, S as 10 lattice spaces, and ρ as 2×10^{11} cm/cm^3, then l_0 becomes 350 Å. On this model the extra radiation annealing in deformed metals results from blocking of these sequences by accumulated damage. The observed decrease in the enhanced production then gives an estimate of $l_0\sigma$, where σ is the mean cross section for blocking by the Frenkel pairs. If these are assumed to be randomly distributed and the sole blocking mechanism, then $(l_0\sigma)^{-1}=n$, the Frenkel pair density to reduce the enhancement $\frac{1}{2}$. In the present case $n\sim1.5\times10^{18}$ defects/cm.[3] Using the value of l_0 from above this gives $\sigma=4.8\times10^{-14}$ cm^2. Such calculations are good only to one order of magnitude since some quantities, particularly ρ, are not better known. Some workers claim that the values of l_0 and σ calculated on this model are impossible and propose that collision sequences are of no importance in radiation damage.[27,28] Other studies argue that the path length to dissipate the energy transferred during irradiation is great enough to support our model.[29–31]

Another model has been proposed which maintains that the enhancement of resistivity increment during irradiation by deformation before the irradiation reflects

[24] This assumes that the same defect whose migration is responsible for stage III in Pt is also responsible for stage III in other fcc metals. There is much evidence that this assumption is true [W. Bauer, A. Seeger, and A. Sosin, Phys. Letters **24A**, 193 (1967)] but it is not essential to our argument. It is sufficient that some defect mobile in stage III be produced during irradiation.

[25] D. Turnbull and R. E. Hoffman, Acta Met. **2**, 419 (1954).

[26] W. Bauer and W. F. Goeppinger. Phys. Rev. **154**, 584 (1967).

[27] M. T. Robinson, Phil. Mag. **12**, 741 (1965).

[28] J. R. Beeler, Jr., Phys. Rev. **150**, 470 (1966).

[29] D. O. Thompson, T. H. Blewitt, and D. K. Holmes, J. Appl. Phys. **28**, 742 (1957).

[30] J. L. Brimhall and B. Mastel, J. Appl. Phys. **38**, 3027 (1967).

[31] M. L. Swanson, Phys. Status Solidi **23**, 649 (1967).

deviations from Matthiessen's rule.[32] In particular it is proposed that the resistivity of Frenkel pairs in a lattice containing dislocations which give a larger contribution to the resistivity than do the point defects is much greater than the resistivity of the same concentration of Frenkel pairs in a lattice in which the dislocation contribution to resistivity is negligible.

This theory gives a satisfactory qualitative explanation of the enhancement of both the resistivity injected by irradiation and radiation annealing but its predictions disagree with observation during recovery. On this theory the resistivity added by the point defects in deformed material is always greater than that added by those in undeformed material given an identical irradiation. As the resistivity added by the point defects falls below that added by dislocations, the rate of decrease of resistivity gradually becomes greater in the deformed than in the undeformed material. This difference in rate increases monotonically until recovery is complete. On the contrary, we find in both Ag and Al[22] that the rate of decrease of resistivity in deformed specimens abruptly becomes greater than in undeformed ones at a temperature which is independent of the ratio of the contribution of point defects and of dislocations to the specimen resistance. This temperature corresponds to the onset of split interstitial migration. In both metals the kinetics of stage-III recovery in deformed specimens are appropriate to the diffusion of a greater density of interstitials to a greater density of sinks than in undeformed material. Data on other metals are similar.[6] Finally, in the annealing after deuteron irradiation of quenched Pt, all of the enhanced resistivity disappears before half of the resistivity injected into a nonquenched specimen has recovered.[9]

These observations indicate that most of the enhanced resistivity increment seen upon irradiation of quenched or deformed specimens represents an increased density of defects and that, while Matthiessen's rule is not strictly obeyed, deviations from it are not sufficient to explain the enhancements. While the matter is not yet settled, we believe that the long-range propagation model best fits the data.

2. *Influence of Deformation without Aging*

Platinum deformed at room temperature and not further annealed contains supersaturated point defects, both interstitials and vacancies. The distribution of these defects is not completely known, but theoretical considerations indicate that their locations are highly correlated, that sequences of vacancies and of interstitials are created by the nonconservative motion of dislocations.[21] These point defects disturb the regularity of the lattice needed for the propagation of collision sequences and so reduce the number of focusons that reach dislocations. In this way they act as do the point defects injected during irradiation which also decrease production by blocking long-range transport events, with the difference that the point defects from plastic deformation are all present before the irradiation. We have also observed in deformed Al[19] and Ag that an irradiation followed by an anneal to remove the defects that recover, in stages I and II reduces the amount of radiation annealing seen in a subsequent irradiation. The data indicate that the point defects remaining from the first irradiation also reduce the defect production rate in the second irradiation but the observed decreases are less than the uncertainty in measurements of absolute resistivity changes in two successive irradiations.

We believe that the damage production rates in undeformed Pt and in deformed (but not aged) Pt are approximately equal because the excess point defects in the latter reduce the principal process responsible for enhanced defect production in deformed and aged Pt, the defocusing of collision sequences at dislocations.

Although deformation without ageing has little effect on the total number of defects produced by deuteron irradiation, it does increase the ratio of the number of defects that anneal in stage I to the number that anneal in stage III. This is also a consequence of the blocking mechanism. Fewer focusons are converted to stage-III interstitials in deformed, unaged Pt than in undeformed Pt.

There are two other mechanisms by which the number of close Frenkel pairs are increased relative to the total number of defects. The first of these is purely statistical. The point defects produced by deformation [a concentration of $(1.5\pm0.5)\times10^{-4}$ is produced by a deformation such as R3] increase the probability that any given site occupied by a radiation produced vacancy or interstitial will be so close to an already existing interstitial or vacancy that the pair is more likely to combine than to separate during heating. Based on the nearest- and next-nearest-neighbor positions being unstable and the next four nearest positions being those of correlated recovery, there are 68 such sites around each vacancy. Since $\frac{1}{2}$ of the Frenkel pairs produced by deuteron irradiation of undeformed Pt are already close pairs, the enhancement of close-pair production by the above process comes to perhaps $\frac{1}{2}\%$ of the total number of defects produced.

The second mechanism for increased close-pair production considers the interaction of focusons with vacancies produced by the deformation. Our studies of deuteron irradiation of quenched Pt showed that the directed defocusing of collision sequences at small vacancy clusters, probably divacancies, results in the production of a greater number of point defects than are produced in Pt lacking these vacancy clusters and that these extra defects are all close Frenkel pairs.[9] Divacancies are seen in the recovery of deformed Pt and it is believed that a variety of vacancy clusters

[32] K. H. Fischer (private communication).

are directly produced by deformation.[21] When a specimen containing a quenched-in vacancy concentration equivalent to the vacancy concentration injected by deformation R3 is irradiated to the dose of these experiments, the close-pair concentration is increased by about 3% of total defect concentration above the concentration injected into an unquenched irradiated specimen. The increase in the deformed specimen is probably less than in the equivalent quenched specimen because the greater lattice disturbance in the former reduces the mean distance of collision chains, so that fewer focusons reach vacancy clusters.

The net change in the total defect production from these various cases is less than the accuracy of our measurements of absolute resistivity. For four pairs of deformed and undeformed specimens that were irradiated together, the ratio of damage in deformed to undeformed Pt ranges from 97 to 102%, with a mean value just over 99%. Our measurements of radiation annealing are nearly one order of magnitude more accurate than absolute-damage measurements. Over the dose range of these experiments, the defect production rate in deformed specimen R3 decreases more than does the rate in undeformed Pt by about 1% of the initial rate. This extra radiation annealing is mainly attributable to increasing damage reducing the number of focusons that are defocused with extra production at vacancy clusters.

3. *Influence of Alloying with Au*

The increase in defect production rate in the Pt–0.1%-Au alloy is another instance of the general rule that alloying increases the production of defects in annealed fcc metals by irradiation near 4°K. We have observed this in deuteron irradiations of Al[19] and Cu, and others have reported the same effect for electron and for neutron irradiations of various alloys.[33,34] It is particularly significant that the amount of resistivity remaining in the alloys at the end of stage II, the stage of release of interstitials from impurity traps, is greater both in percent of total damage and in absolute magnitude than in pure metals. This shows that the substitutional impurities not only trap defects but also increase the amount of conversion of crowdions to split interstitials. The extra production in all of these systems consists of split interstitials and an equal number of vacancies produced by the disruption of long-range events. The increased radiation annealing in all alloys is another consequence of this process. Fewer interactions between such events and impurities take place as the lattice regularity is reduced by radiation damage.

Deformed alloys remain difficult to interpret. We have observed for the present Pt-Au alloy and for Cu–0.03%-Au,[34] that room-temperature deformation reduces the production rate to below that for the pure metal. Since factors such as the amount of segregation of the Au after deformation and the point-defect impurity-binding energies are not known, speculation about defect formation in deformed alloys is not rewarding at this time.

[33] C. L. Snead, Jr. and P. E. Shearin, Phys. Rev. **140**, A1781 (1965).

[34] T. Klabunde (private communication).

B. Defect Recovery

1. *Effects in Stage I*

The data displayed in Fig. 4 show that below 19°K none of the treatments affects recovery by more than 1% of total damage. From Fig. 3 it is evident that the changes in difference of percent damage recovered between deformed and undeformed specimens below 19°K result from the broadening by deformation of the annealing peaks near 10, 15, and 20°K. We have observed similar oscillations of difference in recovery curves resulting from dislocation strain fields for deformed Cu and Ag.[22] In an earlier paper we showed that in Pt the number of pairs recovering below 18°K is little affected by the presence of vacancies.[9] The insensitivity of the recovery of very close pairs to local surroundings is again emphasized in the Pt–0.1%-Au specimens. Even impurity atoms at an average spacing of 10 lattice distances do not significantly effect the amount of recovery below 19°K.

Recovery in the remainder of stage I, correlated recombination of more distant pairs and free migration of crowdions to sinks, is much more sensitive to the dopants considered in this paper. In addition to broadening of the recovery substages with loss of the details of annealing, both the amount of recovery and the temperature of maximum recovery rate are changed by deformation and by alloying with Au. When the dopants are quenched-in vacancies the temperature of maximum recovery rate is shifted by an amount less than can be measured ($\Delta T < 0.1$°K), the amount of resistivity recovered by close pairs recombination (19–23°K) is increased, and the amount recovered by free migration is unchanged.

In all fcc metals studied, deformation before deuteron irradiation reduces the magnitude of the maximum recovery rate and causes a loss of detail in the annealing spectrum, but Pt is unique in that the temperature of maximum recovery rate is changed (increased) by deformation. In an earlier paper[9] we demonstrated that free migration of interstitials first becomes significant in Pt near 23°K. One piece of evidence that recovery is not due to pair recombination is the deviation of a ratio plot such as Fig. 6 from unity. This deviation first becomes significant for both deformed and undeformed specimens at 23°K, showing that deformation does not change the temperature at which free interstitials begin to sample a representative portion of the lattice.

We attribute the shift in temperature of maximum

annealing rate in Pt as contrasted to the zero-temperature shift in Cu and Al to the much smaller energy gap between the last close Frenkel-pair-recombination substage and the beginning of interstitial free migration in Pt. Coltman and co-workers,[2] using very small defect concentrations to increase the temperature interval between these substages, found in Cu a separation of 19°K between the peak of the highest close-pair-recombination substage and the peak of the substage due to free migration. With a similar defect concentration in Pt, the substages were too close to be resolved by the 2°K temperature increments in their experiment. The recovery spectra of Cu and Al are very much alike.[3] A consequence of this small energy gap in Pt is that only very small changes are needed to transform correlated close-pair recombination to uncorrelated recombinations of interstitials with vacancies. In particular, the interactions of crowdions with dislocations provide these small perturbations.

At distances from the dislocation such that elasticity is applicable, Fleischer[35] has derived the potential

$$V_s=\frac{Gb^4\Delta\epsilon}{4\pi r}(\sqrt{2}\cos\theta+\sin\theta)=\frac{b}{r}W_s f_s(\theta) \qquad (4)$$

for the interaction between a crowdion and a screw dislocation. In this equation, G is the shear modulus, b is the Burger's vector, and $\Delta\epsilon$ is the tetragonality of the crowdion. In a right-handed coordinate system, if the dislocation lies along the z axis, the angle θ is measured in the x-y plane from the positive x axis to the projection of the crowdion onto the plane. For an edge dislocation the interaction can similarly be written[21]:

$$V_E=W_E(b/r)f_E(\theta), \qquad (5)$$

where W_E is the maximum edge dislocation-crowdion binding energy. In Pt, W_s has a value of about $\frac{1}{4}$ eV and W_E is larger by a factor $1/(1-\nu)\sim\frac{3}{2}$.[36] Here ν is Poisson's ratio.

Let the difference between the activation energy of the highest close-pair recombination process and the energy of free interstitial migration be ΔE; then the locus of all points lying within dislocation strain fields at potential gradients greater than $\Delta E/b$ is given by

$$\frac{\Delta E}{b}<\left[\frac{W^2b^2}{r^4}f^2(\theta)+\frac{W^2b^2}{r^4}\left[\frac{\partial}{\partial\theta}f(\theta)\right]^2\right]^{1/2}. \qquad (6)$$

In a previous paper[9] we presented the activation energy versus temperature spectrum for stage-I recovery in Pt. Near 23°K a difference of 1°K in temperature of recovery processes corresponds to a difference of 0.003 eV in activation energies. The failure of Coltman and co-workers[2] to separate first-order correlated recombination process from dose-dependent recombination using doses two orders of magnitude smaller than in the present work and our failure to resolve these processes using temperature pulses with increments of $\frac{1}{4}$°K indicate that the peak of the highest close-pair recombination process lies less than $\frac{1}{2}$°K (0.0015 eV) below the lowest temperature of significant interstitial free migration. Taking ΔE in Eq. (6) as 0.0015, then for the screw-dislocation case, all such close Frenkel pairs within a distance R of the dislocation given by

$$R=\left(\frac{3W_s}{\Delta E}\right)^{1/2}b=\left(\frac{0.75}{0.0015}\right)^{1/2}b=22.3b \qquad (7)$$

are located in strain gradients of sufficient magnitude that the energy barrier for separation is smaller than that for interstitial-vacancy recombination in some orientations of the crowdion. The value of R for edge dislocations is somewhat larger. The fraction of Frenkel pairs oriented for separation varies from $\frac{1}{12}$ at $r\sim R$ to nearly $\frac{1}{2}$ at $r\ll R$.

The fraction of direct displacements significantly affected by dislocation strain fields even in very heavily deformed Pt is not greater than 10^{-3}, but the above calculation shows that the fraction of Frenkel pairs subjected to gradients greater than $1.\times10^{-3}$ (eV/lattice space) is increased above this by a factor of about 500. Thus, a significant fraction of pairs that in an unstrained lattice would have recombined with an activation energy less than that of free interstitial migration do not so recombine in the deformed lattice. Naturally, some interstitials that would have had to migrate freely an appreciable distance in an unstrained lattice to reach a sink will be aided by lattice strains to combine with a vacancy, so that these recover with an activation energy less than that of free migration; however, since the amount of recovery in undeformed Pt within $23\pm\frac{1}{2}$°K is more than twice as great by correlated close-pair recombination as by free interstitial migration, and since the ratio of close pairs to free crowdions produced by an irradiation is increased by deformation, the back process is less important. Finally, recovery in the range of free migration is delayed by the perturbations in lattice potential produced by the dislocations.

The result of these processes in Pt is that by deformation, an appreciable fraction of recovery is transformed from close-pair recombination to recovery involving the migration of crowdions over comparatively large distances. The former process takes place below 23°K, the latter in a range above 23°K, and this range is extended upward in deformed as compared to undeformed Pt.

This mechanism is unimportant in Cu and Al. Few sites are in stress fields large enough to span the much larger energy gap in these metals. It is of no consequence

[35] R. L. Fleischer, Acta Met. **10**, 835 (1962).
[36] A. W. Cochardt, G. Schoeck, and A. Wiedersich, Acta Met. **3**, 533 (1955).

in Au since free crowdion migration does not take place in this metal.[37]

In view of the interactions between dislocations and interstitials, the probability that mobile crowdions be strongly trapped at dislocations and prevented from reaching sinks in stage I should be considered. It is evident that in Pt dislocations themselves are not important sinks in stage I, since stage-I annealing is delayed when excess dislocations are present. Internal-friction measurements by Keefer and co-workers demonstrate that the number of pinning points on dislocations does increase at the end of stage I.[38] However, Keefer's analysis shows that because crowdions are constrained to move in only one dimension, only a very small fraction of the crowdions come closer than $10b$ to dislocations under the influence of the dislocation stress field during thermal migration. In particular, he proves that the stress field of screw dislocations repels all crowdions whose paths intersect the core of the dislocation and repels all crowdions located farther than $5.9p$ from the dislocation, where p is the minimum distance from the dislocation to the path of the crowdion. In the work of Keefer the number of additional dislocation pinning points added in stage I after an irradiation with 1-MeV electrons corresponds to an average concentration of 3.5×10^{-14} (crowdions/lattice atom). This should be compared with the concentration of Frenkel pairs injected by the irradiation 7×10^{-7} or to the decrease of 8×10^{-8} in the concentration of crowdions during the pinning process. Thus, in the recovery of undeformed Cu fewer than 1 in 10^6 of the freely migrating crowdions are bound tightly enough to dislocations to provide pinning points that persist to temperatures beyond those of stage-I migration. The most heavily deformed specimens considered in the present work, even after ageing, have a ratio of dislocation density to vacancy sinks only between two and three orders of magnitude greater than in Keefer's specimens. On this basis we expect that fewer than 0.1% of mobile crowdions (<0.01% of all injected defects) are trapped by dislocations at the end of stage-I recovery in Pt deformed as in this work

In the temperature interval spanning the two higher-temperature correlated recombination processes and the early part of free interstitial migration, approximately 18–26°K, the ratio of the annealing rate in the alloy to that in pure, undeformed Pt steadily decreases as the number of jumps for annihilation increases. In particular this ratio falls abruptly when mobile crowdions begin to sample a representative proportion of the lattice before recombining with vacancies. This shows that there is a large interaction between Au atoms and interstitials. Recovery of very close pairs is not measurably affected but pairs with larger separation, that in pure Pt would still recover by correlated recombination, are increasingly likely to be part of Frenkel-pair Au complexes in which the first jump of the interstitial is toward the Au atom rather than toward the vacancy. Even though the resistivity injected into the alloy was 40% greater than that into pure Pt by the same irradiation, the resistivity drop between 23 and 28°K in the alloy was only half that in pure Pt. If the resistivity of a bound Au interstitial complex is no greater than that of an isolated Au atom, then about half of the freely migrating crowdions are captured. Any increase in the resistivity of the complex over that of an isolated Au atom makes for an increase in the fraction of free crowdions that are trapped. The resistivity of an interstitial Au complex cannot be greater than that of a Au atom by more than half the resistivity of a free crowdion. This value corresponds to capture of all free crowdions by Au atoms. The above estimate assumes that the production of stage-I defects in the alloy is the same as in pure Pt. The data on the Pt-dislocation "alloy" indicate that this assumption is true to within a few percent.

Deformation affects stage-I recovery in the alloy qualitatively much the same as in pure Pt. The temperature of maximum recovery rate is moved higher by deformation, below this temperature annealing is retarded in the deformed specimen, and above that temperature the annealing rate is increased. Since the elastic strain fields around Au atoms in the Pt lattice are of much smaller magnitude and range than those associated with dislocations, the same explanation proposed above for these deformation effects in pure metals can be carried over the alloy with the consideration that in both alloyed specimens some interstitials are trapped at Au atoms.

2. *Effects in Stages II and III*

About 2½% more total damage recovers in stage II in heavily deformed than in undeformed Pt. This extra recovery occurs in the first half of stage II, below 150°K in aged specimens and below 100°K in unaged, deformed ones. The extra recovery in stage II implies that deformation increases the number of traps for interstitials. Since it is known that dislocations are pinned by interstitials injected during the irradiation and by those interstitials that do migrate to dislocations at the end of stage I,[38] this extra trapping is expected. No new resolvable substage is produced by deformation. The interstitial-dislocation traps have a nearly continuous range of pinning energies, as is to be expected from interactions varying as $r^{-1}\sin\theta$.

Although the amount of stage-II recovery in deformed specimens is not changed by ageing, recovery occurs at lower temperature in unaged, deformed specimens. The peak in the amount of enhancement of recovery in deformed relative to undeformed Pt, near 100°K for deformation R3, is similar to that observed

[37] W. Bauer and A. Sosin, Phys. Rev. **136**, A255 (1964).
[38] D. Keefer, J. C. Robinson, and A. Sosin, Acta Met. **13**, 1135 (1965).

in stage II for quenched Pt.[9] In the deformed case also the presence of excess lattice vacancies increases the probability that a vacancy is close enough to a trapped interstitial to reduce the energy barrier for escape and annihilation of the interstitial.

The fraction of total damage that recovers in the early part of stage III is reduced by alloying and by deformation followed by ageing from that in pure, undeformed Pt. These treatments increase the fraction of interstitials that form stable clusters in stage III by increasing the concentration of normal interstitials produced during the bombardment.[9] Deformation without ageing somewhat reduces the production during bombardment of normal interstitials, but such specimens contain more of both interstitals and vacancies than do undeformed specimens given the same irradiation. The relative concentrations of interstitials and vacancies produced by the deformation are not known. Recovery of resistivity after deformation is greater in stage IV than in stage III, but the defect distribution after deformation is such that clustering of interstitials may be especially significant in this case. Ageing deformed specimens at 100°C reduces the ratio of separate interstitial-type defects to vacancy-type defects. This treatment should increase the amount of recovery in stage III after a subsequent irradiation.

An anneal at room temperature reduced the resistivity of the deformed, low-dose specimen to below its preirradiation value. We have observed a similar result in the recovery of low-dose, quenched specimens.[9] In that case we explained the overshoot in recovery despite the conservation of total number of vacant lattice sites as resulting from the conversion of divacancies into two separate single vacancies. This conversion takes place because interstitials are preferentially produced near divacancies by the directed defocusing mechanism. The same process applies in the present case of deformed Pt with excess vacancies.

Most of the additional recovery during stage II in the alloy as compared with pure Pt results from the release of interstitials trapped during stage I from Au-interstitial complexes. This extra recovery is concentrated in two large substages, as has been reported for some other alloy systems.[39]

The magnitude of the resistivity decrease in stage II is the same in deformed as in undeformed alloy specimens. This implies that both specimens have nearly the same number of traps. Dislocations injected by the deformation are much less efficient traps than Au. The same recovery substages near 40, 95, and 110°K seen in the undeformed alloy are also present but broadened in the deformed alloy.

The resistivity remaining at the end of stage II in the deformed alloy was much smaller than in the undeformed one. On the model of this paper, these results imply that conversion to normal interstitials during irradiation is strongly reduced by deformation of the alloy. This correlates with the smaller defect production in the deformed alloy.

V. SUMMARY

The main conclusions from the present work are as follows:

(1) Defects are produced by the interaction of focusing sequences with dislocations and with substitutional Au atoms that would not have been produced in the absence of these interactions.

(2) The interactions of focusing sequences with dislocations are strongly quenched by the point defects produced during deformation and by defects injected during irradiation.

(3) The proportion of the total damage that recovers by correlated recombination of close Frenkel pairs is reduced by heavy deformation of Pt; correspondingly, the proportion by long-range migration of crowdions is increased.

(4) Au in 0.1% concentration produces two large sets of traps for mobile crowdions in Pt.

(5) A small increase in the density of traps for crowdions is produced in pure Pt by heavy deformation. In this case the traps have nearly a continuum of binding energies in contrast to traps produced by substitutional Au.

(6) The additional defects produced as in item 1 are normal interstitials (first mobile in stage III) and their accompanying vacancies.

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[39] K. R. Garr and A. Sosin, Phys. Rev. **162**, 669 (1967).