Lawrence Berkeley Laboratory

UNIVERSITY OF CALIFORNIA

**Materials & Molecular
Research Division**

SECONDARY DEFECTS IN QUENCHED PLATINUM

María Isabel Pérez
(M.S. thesis)

November 1979

MASTER

Prepared for the U.S. Department of Energy under Contract W-7405-ENG-49

DISCLAIMER

This book was prepared as an exhibit of work submitted by an agency of the United States Government to the United States Government and any agency thereof nor any of their employees makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, disclosure, product or process disclosed or represented that it is about its software, product, service, process, system, or any specific information which is disclosed or derived by trade name, trade dress, manufacturer, or otherwise, does not constitute, and use of this is independent recommendation or backing by the United States Government or any agency thereof. The views and opinions of authors representing herein do not necessarily state or reflect those of the United States Government or any agency thereof.

a mis padres y a Francisco

SECONDARY DEFECTS IN QUENCHED PLATINUM

María Isabel Pérez

Materials and Molecular Research Division, Lawrence Berkeley Laboratory
and Department of Materials Science and Mineral Engineering
University of California, Berkeley, Ca. 94720

ABSTRACT

The structure of secondary defects in two different platinum purities, quenched under ultra high vacuum, has been studied by transmission electron microscopy.

Faulted loops on {100} planes have been observed in both materials. In the less pure platinum, Pt B (but purer in terms of carbon content), the defects were observed after quenching, whereas in the purest one, Pt A (but less pure in terms of carbon content), the defects were formed after a long annealing. The Burgers vector of the loops formed after the quenching was $a/3\langle 100 \rangle$ and for loops formed during long annealings, was $a/2\langle 100 \rangle$, apparently.

It was found that in Pt E the loop density increased and the loop size decreased as the quenching temperature was lowered. The same effect was produced by increasing the carbon content by a small amount. The loop formation was totally inhibited by adding a larger amount of carbon and also when the quenching was performed in poor vacuum (~ 0.01 torr) or in air.

The defects can be interpreted as pure vacancy loops heterogeneously nucleated at carbon atom clusters or, as plate like precipitates of carbon atoms and vacancies formed by a co-precipitation mechanism. The discussion of the experimental results obtained is based upon these two models.

TABLE OF CONTENTS

1. INTRODUCTION	1
2. EXPERIMENTAL WORK	5
2.1. Material Preparation, 5.	
2.2. Quenching and Annealing, 5.	
2.3. Thin Foil Preparation, 6.	
2.4. Carburizing Treatment, 7.	
3. RESULTS	8
3.1. Secondary Defect Structure in Platinum B, 8.	
3.1.1. Loops with $b=a/3 < 100 \rangle$, 8.	
3.1.2. Effect of Quenching Temperature, 9.	
3.1.3. Effect of Annealing in the Secondary Defect Structure, 10.	
3.1.4. Nature of the Loops, 12.	
3.1.5. Annealing at Higher Temperatures, 12.	
3.2. Secondary Defect Structure in Platinum A, 13.	
3.3. Effect of Carbon Atoms and Quenching Atmosphere in the Defect Formation, 14.	
4. DISCUSSION	15
4.1. Precipitation of Vacancies, 15.	
4.2. Analysis of the Impurities Present in the Two Materials, 16.	
4.3. Model I for Carbon-Vacancy Interaction, 17.	
4.3.1. Model Formulation, 17.	
4.3.2. Experimental Evidence for the Model, 18.	
4.3.3. Stability of the Loops, 19.	
4.3.4. Burgers Vector of the Annealed Defects, 20.	
4.4. Model II for Carbon-Vacancy Interaction, 21.	
5. ACKNOWLEDGMENTS	25
6. REFERENCES	26
7. FIGURE CAPTIONS	28
8. TABLES	30

9. FIGURES	32
------------------	----

1. INTRODUCTION

How vacancies cluster and precipitate during quenching and annealing experiments in metals is a matter of considerable complexity. A great number of experimental and theoretical studies on pure and impure FCC metals have been carried out during the last twenty years and, the formation of many types of clustered vacancy defects have been reported. In spite of these many studies, there is not yet a theory capable of explaining all the observations. Many questions are still unanswered. Three important questions among others are: (1) How and when a growing vacancy cluster collapses to form a dislocation loop? (2) Which of the physical parameters, involved in the process, is the responsible parameter for determining the type of secondary defect formed? (3) What conditions are necessary for a certain parameter to determine the type of secondary defect?

Although it is true that one of these parameters, the stacking fault energy, is an important factor in determining which type of secondary defect will be formed (i.e., whether or not a dislocation loop will contain a stacking fault), it is also true that this is only one factor and in many cases not the most important one. For example, it has been found that impurities, even in very small amounts, play an important role in the formation of clustered vacancy defects in many metals¹⁻⁵.

The secondary defects which were first observed in aluminum, were prismatic loops⁶. It was believed (at that time) that the difference in energy between perfect and Frank loops, due to the high stacking fault energy in Al, was determinant in the type of loop formed. Later, Frank sessile loops were also observed in pure Al^{5,7,8}. These defects were interpreted as metastable loops which could be unfaulted by increasing the temperature of the specimen^{5,9,10}. Cotterill¹⁰ suggested that there is a critical temperature, T_w , at which the loops become unfaulted. He suggested that if, during the quenching, a cluster reaches the size at which it would collapse

to form the dislocation loop before the temperature T_{uf} , the resultant loop should be unfaulted. If on the other hand, the cluster reaches the critical size at a temperature below T_{uf} , it will be faulted. But the instant at which a vacancy cluster will become a stable nucleus during the quenching depends on the type of nucleation as well as on the rate of growth of the clusters; parameters which could be modify by other variables, as for example the amount of impurities.

It is known that vacancies may be trapped by impurity atoms¹¹. This trapping, which results in the formation of vacancy-impurity complexes¹², may modify the annealing rate of vacancies¹³. The complex may be less mobile than the vacancy itself resulting in a decreasing of the annealing rate¹⁴; or the complex mobility may be faster than the vacancy mobility resulting in an increasing of the annealing rate¹⁵. Thus, impurities can strongly modify the process of secondary defect formation. On the other hand, impurities can also act as heterogeneous points of nucleation for vacancies⁵.

Cotterill and Segal⁵ suggested that impurities having the largest binding energies with vacancies should have the strongest effect on the vacancy clustering process.

Some interstitial impurities produce a large distortion in the lattice, due to the difference between their radius and the interstitial hole radius of the host metal. Table I shows the atomic misfits, for octahedral sites-hard sphere model, of interstitial impurities in FCC metals. The lattice strain associated with these impurities may result in a strong interaction between the vacancies and interstitial impurities. Large binding energies have been suggested for these interstitial atom/vacancy complexes, as for example carbon-vacancy in Ni¹⁶ oxygen-vacancy in Ag¹⁷ and carbon-vacancy in Pt^{18,19}. Therefore, a critical effect of these impurities on the vacancy cluster defect formation is expected.

In studying annealing of point defects in FCC metals, in the specific case of Pt many works have been done using resistivity measurements; however, because of the difficulty of preparing thin foils of Pt, relatively few have used electron microscopy. In this regard Pt has

been comparatively neglected.

Ruedl *et al.*²⁰, using TEM observed only small voids in Pt specimens (using beaten foils) quenched from a temperature close to the melting point and annealed under vacuum. The voids were preferentially located on dislocations and were difficult to eliminate, since they appeared to be stable up to $\sim 800^{\circ}\text{C}$. Cizek *et al.*²¹ studied Pt of two purities; the lower-purity sample (99.9%) showed only black dots (in bright field TEM analysis) after a quenching from 1300°C and annealing in air, while in the most pure one (99.99%), voids similar to those of Ruedl *et al.*²⁰ were observed. The authors²¹ also found that the black dot density in the less pure material decreases with the number of quenchings. They concluded that the black dots were formed by vacancy clustering in the presence of impurities.

Jackson²², using resistivity measurements, studied vacancy annealing in quenched pure platinum. He found the annealing in this case was markedly different from the annealing of quenched gold, silver and aluminum. Also, the author²² found the residual excess resistivity, due to the existence of vacancy clusters, was much lower than for the other metals. He concluded that sinks for vacancies in Pt were much different than those in Au, Ag and Al. He finally proposes that the high surface energy of Pt³⁸, $\gamma_s = 2100 \pm 700 \text{ erg-cm}^{-2}$, is the strong barrier to nucleation of loops, and could explain why these defects had not been observed in this metal; an argument which was in agreement with other researchers^{21,22}.

More recently, Westmacott^{23,24} reported the formation of faulted loops on {100} planes in different purities of Pt, after quenching from a high temperature under high vacuum (10^{-8} torr). When the quenching was done in air, only voids were observed in the less pure material. The conclusion was that carbon and oxygen impurities play a critical role in the formation of these secondary defects.

The occurrence of faulted (100) loops, which have not been observed in any of the other FCC metals, was very surprising since the removal of an atom layer on a (100) plane

leads to the formation of a very high energy stacking fault, higher than in a similar defect formed on a (111) plane.

The aim of the present work is to try to explain in detail the mechanism under which these faulted loops on {100} planes are formed in Pt. In order to do this, (1) A complete characterization of the defects has been done; (2) The influence on the defect formation and stability of the following parameters has been determined: (a) the amount of impurities, (b) the quenching temperature, and (c) the annealing temperature and annealing time. Two different mechanisms for the formation of the loops are proposed. Since it has been found that the quenching atmosphere can strongly influence the vacancy clustering^{23,24}, the experiments were conducted under an ultra high vacuum system. Experiments under well controlled conditions for two different purities of platinum were performed. An extensive transmission electron microscopy analysis of the defects was done in all cases.

2. EXPERIMENTAL WORK

2.1. Material Preparation.

Two different purities of platinum were used: Pt A of 99.99wt% pure, supplied by The National Bureau of Standards, and Pt B of 99.76wt% pure. Details of the chemical analysis for both materials are given in Table II. The analysis of carbon, for both materials, was made in a Leco low carbon analyzer in which the limit content detectable is 0.001wt% in samples of 0.5g.

The carbon content of the two platinum samples was also determined by proton activation analysis¹⁹. Using this method the carbon concentrations found, 3040appm for Pt A and 1000appm for Pt B, were approximately one order of magnitude higher than those given by the combustion method (see Table II). However the concentration ratio between the two materials was the same in both methods. The proton activation analysis is a new method and it has been successfully used to determine nitrogen in atmospheric aerosols⁴⁰, the detection limit in this case was 200ppm and the average percent difference in comparison with traditional combustion analysis was 14%. For the case of carbon in platinum neither the accuracy nor the limit of detection has been determined.

The samples were rolled at a thickness between 0.0025 and 0.0020 inches. In order to avoid contamination, the rolling apparatus was carefully cleaned, and the foils were etched in aqua regia before and after the rolling process. Then specimens of 1 by 0.16 inches were cut.

2.2. Quenching and Annealing.

The apparatus used for quenching has been described by Westmacott and Peck²⁵, and

consisted of a Varian feedthrough fixed to a vacuum pumped system, in which pressures in the range of 10^{-4} torr were obtained. The samples were Joule-heated by clamping them to the feedthrough electrodes. Most of the quenching treatments were done by heating the sample until the center portion of the sample melted, interrupting the heating current and producing a rapid cooling, mainly by conduction. When the quenching was done from lower temperatures, a small thermocouple of Pt-Pt 5% Rh was spot-welded to the foils to measure the temperature. In this case the quenching was done by switching off the heating current. Using this method, according to Westmacott and Peck²⁵, the quenching rate in the center of the sample is approximately $2500^{\circ}\text{C}/\text{sec}$. In order to study the effect of the quenching atmosphere, some of the quenches were done in a deliberately poor vacuum ($\sim 10^{-2}$ torr) or in air.

When necessary, the samples were annealed (in a salt bath) immediately after the quenching.

2.3. Thin Foil Preparation.

The specimens for the electron microscope were always obtained from the center portion of the foils. After a pre-thinning in boiling aqua regia for about 5 to 10 minutes, the specimens were prepared using an ac electropolishing technique²¹. The electrolyte used was a solution of equal volumes of phosphoric, sulfuric and nitric acids at room temperature. The current density was between 0.2 to $0.5\text{A}\cdot\text{cm}^{-2}$ (ac) and the voltage, 3 volts. After approximately 5 hours of electropolishing, the foils were carefully washed in water and methanol and then dried. Small samples were cut from the end of the foils and observed in a Phillips Electron Microscope EM-301 operated at 100KV.

2.4. Carburizing Treatment.

To study the effect of carbon content on the clustering of vacancies, more carbon was added to both materials (Pt A and Pt B), by two different methods. The first method has been described in detail by Dahmen²⁶. With this method carbon layers of about 350Å thick were deposited on both sides of the foil, diffused into the sample by annealing in a vacuum of 10^{-7} torr at 1500°C for one hour, and then quenched from near the melting point. After the treatment, the carbon content should theoretically be ~400appm, but it is estimated that less than half of the deposited carbon is diffused into the foil, by this method. As it has been reported by Dahmen²⁶, an appreciable decarburization takes place during vacuum annealing. However no analysis was performed because the difficulties of making this in small samples (a minimum of 0.5g is required).

The second method consists of putting the samples in a graphite crucible with graphite powder inside. High purity material was used for both purposes, the crucible and the powder. The crucible was then put into a silica capsule and heated at 1500°C for two hours to achieve the solid solution equilibrium. After the carburizing treatment the samples were carefully cleaned and quenched as usual.

3. RESULTS

3.1. Secondary Defect Structure in Platinum B.

3.1.1. Loops with $\underline{b} = \underline{a}/3 \langle 100 \rangle$.

The relatively impure Pt, Pt B, was quenched from near the melting point ($\sim 1760^\circ\text{C}$) under a vacuum of $\sim 10^{-4}$ torr. This treatment resulted in the formation of stable planar defects lying on $\{100\}$ planes which show fringe contrast. The contrast analysis showed that they are faulted loops having a Burgers vector, $\underline{b}$, and a displacement vector, $\underline{R}$, equal to $\underline{a}/3[100]$. A set of micrographs showing these defects are given in Fig.1.

Assuming the contrast theory (diffraction contrast in two beam conditions,¹⁷ is valid for $\{100\}$ loops, the predicted behavior of loops with $\underline{b} = \underline{R} = \underline{a}/3[100]$ under various diffraction conditions is given in Table III, which is in complete agreement with the experimental results shown in Fig.1.

For example, with the diffraction vector $\underline{g} = \bar{1}\bar{1}1$ and the beam direction $\underline{B} = [121]$, the three sets of loops on $\{100\}$ planes are in contrast. The faults in each of the three sets are visible since $\alpha = \pm 2\pi/3$, but the dislocations are not since $\underline{g} \cdot \underline{b} = \pm 1/3$. With this beam direction and $\underline{g}$ vector ($\underline{B} = [121]$ and $\underline{g} = \bar{1}\bar{1}1$), the geometry shown by the defects is almost the same as for loops lying on $\{111\}$ planes and therefore difficult to distinguish from them. However, for $\{111\}$ defects one should have four sets in contrast with one of them edge on, while for $\{100\}$ defects there is not a set edge on (see Fig.1a). The geometry of the $\{100\}$ traces in a $[112]$ orientation can be observed in the stereographic projection of Fig.4b.

When the beam direction $\underline{B} = [011]$ and $\underline{g} = 200$ (Fig.1b) only the set of $\{100\}$ loops edge on with $\underline{b} = \underline{R} = \underline{a}/3[100]$ is seen, the other two sets are invisible since both $\underline{g} \cdot \underline{b}$ and α are zero.

This shows definitively that the loops are lying on {100} planes and the Burgers vectors are in $\langle 100 \rangle$ directions. The stereographic projection of the [011] orientation is shown in Fig.4a, where the traces of {100} planes have been drawn.

With $\underline{B} = [011]$ and $\underline{g} = \overline{1}33$ (Fig.1c) one set of loops is again edge on while the other two sets make equal inclination to the beam; for these sets $\alpha = \pm 2\pi$ and no fault contrast is observed ($d_{\text{loop}} \approx 700\text{\AA}$, $\xi_{133} = 385\text{\AA}$), whereas $\underline{g} \cdot \underline{b} = \pm 1$ and the dislocations are in strong contrast.

Finally, with $\underline{B} = [111]$ and $\underline{g} = \overline{2}02$ (Fig.1d), two sets of loops are seen for which $\alpha = \pm 4\pi/3$, and the fault contrast is observed in both sets. However, since $\underline{g} \cdot \underline{b} = \pm 2/3$ for one set and $\underline{g} \cdot \underline{b} = -(\pm 2/3)$ for the other, only one set shows the dislocation in contrast. Again the geometry shown by the loops was consistent with the trace analysis, see Fig.4c.

3.1.2. Effect of Quenching Temperature.

The effect of decreasing the quenching temperature, but maintaining all the other conditions constant, resulted in a decrease in the loop size. The loop density was more or less the same at 1500°C , but clearly larger at 1300°C . When the quenching was done from 1000°C , few defects were observed, and these were only at dislocations. Fig.2a,b,c show areas of different samples quenched from 1760, 1500 and 1300°C , respectively, imaged under the same contrast conditions. Fig.2d shows a representative area of a specimen quenched from 1000°C .

In order to estimate the density of loops, it was necessary to determine the thickness of the thin foils. This was done by counting the number of thickness fringes from the edge of the foil under the appropriate contrast conditions, as described by Hirsch *et al.*²⁷. This method gives only an approximate estimation of the true value, since important errors can be introduced by the inaccuracy in the calculation of the real extinction distance. Since there is no general method of measuring the deviation from the exact Bragg angle and the contribution of the

high order reflections cannot be completely avoided, the thickness of the foil can be over estimated by a range of 5 to 50%. In order to minimize the error special care was taken using this technique.

in some samples, when the foil surface was approximately parallel to a specific crystallographic plane and large defects cutting both surfaces of the foil were found, the trace method to estimate the foil thickness was used²⁷. If the electron beam is normally incident to the foil surface, the foil thickness t is given by $t = W \times \tan \phi$; where W is the projected width of the defect and ϕ is the angle between the plane of the defect and the foil surface. There is also a possible error using this determination which comes from the fact that the foil surface may not be exactly normal to the electron beam. This error can be avoided by determining the deviation of the foil surface from the ideal position, normal to the beam²⁷. The estimated thickness of the transparent regions of the foils using one of these methods or both, varied between 500 to 1500 Å.

Because of the inaccuracy in the estimation of the foil thickness and other errors which will be discussed later, only an approximate determination of the loop density per unit volume was possible. The equilibrium vacancy concentration in Pt at the quenching temperature (with data obtained elsewhere³⁹), the estimated loop density and the vacancy concentration stored in the defects, at each of the quenching temperatures are given in Table IV.

3.1.3. Effect of Annealing in the Secondary Defect Structure.

When a post-quench ageing treatment was given to the samples, a different structure was observed. A post-quench annealing of 24 hours at 400°C to the foils previously quenched from 1760°C resulted in the formation of faulted loops lying again on {100} planes but with different Burgers vectors. The shape of the loops were almost square, with the sides lying on <110> directions in contrast to the loops described earlier which had <100> edges²³. A

detailed contrast analysis was made, and a series of micrographs showing the results are given in Fig.3.

With the diffraction vector $g=\bar{1}1\bar{1}$ and beam direction $B=[\bar{1}12]$, Fig.3a three sets of loops are in contrast. Looking carefully at the micrograph it is possible to distinguish two kinds of defects: the smaller ones, type α , have circular shape and the larger ones, type β , have a square projected shape. While the α loops show only the fault in contrast, β loops show the fault as well as the dislocation in contrast for the three sets.

Keeping the same diffraction vector $g=\bar{1}1\bar{1}$, but tilting now to $B=[011]$, one set of loops become edge on, another increases the apparent size, and the last one shows the opposite of the former one (Fig.3b), which is consistent with the fact that they are now making equal inclination to the beam. With the same beam direction $B=[011]$ and using now the $[\bar{1}\bar{1}1]$ reflection we see again the three sets of loops in contrast, Fig.3c.

Tilting the specimen to the $[\bar{1}11]$ orientation and using the diffraction vectors $g=202$ and $g=0\bar{2}2$, Figs.3d and 3e, it can be seen that one set of loops is always in contrast while one of the other two is alternatively out of contrast in each case. Under these conditions, the visible β loops do not show fringe contrast, while the dislocations are in strong contrast. However, α loops do show fringe contrast. It can be noticed that loops out of contrast show a typical residual contrast denoted by arrows on the micrographs.

After this analysis, it is concluded that the habit plane of the loops must be $\{100\}$.

The corresponding diffraction patterns for the micrographs of Fig.3a-g are shown in Fig.3h-n.

To confirm the results, a trace analysis of the loops was done. The stereographic projection of $[011]$, $[\bar{1}12]$ and $[\bar{1}\bar{1}1]$ orientations are shown in Fig.4. The traces of $\{100\}$ planes and of $\langle 110 \rangle$ directions are indicated in each case. The three sets of loops are denoted by A,

B and C in Fig.3b and loop edge directions are indicated by lines TA1, TB1, TB2, TC1 and TC2 in Fig.3a,b,d,e. After the analysis, it was found that the loop edge directions were coincident with the $\langle 111 \rangle$ directions and that the habit plane for loops A was $(\bar{1}00)$, for loops B was (010) and for loops C was the (001) plane.

Type α loops are seen to be similar to the loops observed in the as quenched samples, i.e., with a Burgers vector $\underline{b}$ and a displacement vector $\underline{R} = a/3 \langle 100 \rangle$. The contrast behavior of β loops suggests that they have a Burgers vector and a displacement vector perpendicular to the habit plane, but with a magnitude larger than $a/3$. The possible magnitude of these vectors will be discussed later.

3.1.4. Nature of the Loops.

At this point it is important to analyze the nature of the loops, whether they are intrinsic or extrinsic. Using the simple method described by Foll and Wilkens²⁸, the loop character can be determined. Let 'a' and 'b' denote the loops marked on Figs.3e and 3g, respectively. In Fig.3g, in which $\underline{g} = 02\bar{2}$, loops 'a' appeared to be smaller than in Fig.3e. The opposite is true for loops 'b'. According to Foll and Wilkens' rules, this means that $(\underline{g} \cdot \underline{b})s > 0$ for loops 'a' (inside contrast), and $(\underline{g} \cdot \underline{b})s < 0$ for loops 'b' (outside contrast). Since s is positive we will consider only the $(\underline{g} \cdot \underline{b})$ value. According with the analysis, the habit plane of loops 'a' is (010) and of loops 'b' is (001), with normals pointing toward the beam source. So, for loops 'a' $\underline{g} \cdot \underline{b} > 0$ and then since $\underline{g} = 02\bar{2}$, $\underline{b} \cdot \underline{n} > 0$ ($\underline{n}$ is the normal to the plane). This result indicates that loops 'a' are vacancy in character. The same conclusion was obtained for the other set of loops for which $\underline{g} \cdot \underline{b} < 0$, then since $\underline{g} = 02\bar{2}$, $\underline{b} \cdot \underline{n} < 0$.

3.1.5. Annealing at Higher Temperatures.

When samples were annealed at higher temperatures, 450 to 550°C for one hour, larger

loops were observed. The loops were still faulted with the same Burgers vector as loops formed after 24 hours of annealing at 400°C, nevertheless the loop shape remained roughly circular.

The loop density was much less compared with the density after annealing at 400°C. From this it is concluded that larger loops grow at the expense of smaller ones. Fig.5 shows an example of these large loops after an annealing at 550°C, where it can be noticed that there are still some small loops present, noted by (*) on the micrograph. All the defects observed were truncated by the foil surfaces, thus, it was easier to do the analysis of the fault nature. Using the method described Gevers *et al.*²⁹, it was determined that the faults were intrinsic in nature, the micrographs of Fig.6 are showing an example of the analysis. All the characteristics of the fringes show that they correspond to $2\pi/3$ type of fringes. These type of fringes can correspond either to a stacking fault or to a planar precipitate.

Finally when the annealing temperature was increased at 600°C for one hour, all the defects were annealed out.

3.2. Secondary Defect Structure in Platinum A.

When the purest material (in terms of the total impurities), Pt A, was quenched from a temperature near the melting point in the same conditions as for Pt B, no defects were observed in the material as quenched. It was only after some annealing that black dots (in bright field) were observed, as shown in Fig.7. The micrograph of Fig.7 shows a representative example of the structure observed in Pt A after a post quench annealing at 400°C for one hour.

When the annealing time was increased to 24 hours, a structure observed was similar to that in Pt B after the same treatment, see Fig.8.

3.3. Effect of Carbon Atoms and Quenching Atmosphere in the Defect Formation.

After the quenching treatment given to samples of Pt B with a low amount of carbon added, the secondary defect structure observed was similar to that for Pt B after the quenching from 1300°C, as it is shown in Fig.9. The estimated loop density in these samples was $5.5 \times 10^{14} \text{cm}^{-3}$ and the estimated average loop size was 300Å.

When more carbon was added, using the second method described in Sec.2.4, no defects were observed in the samples after the usual quenching treatment; no loops were observed after an annealing at 400°C for 24 hours either. However, precipitates of a second phase were developed after this treatment, which give rise to extra spots in the diffraction pattern. Since this could be a matter of another study, no analysis has been done from these precipitates. In Fig.10 it is shown a micrograph and the corresponding diffraction pattern of the configuration showed by the samples in this latter case.

4. DISCUSSION

4.1 Precipitation of Vacancies.

Consider first the situation following the rapid quench from a temperature close to the melting point, i.e. $\sim 1760^\circ\text{C}$. Due to the quenching technique used, the cooling rate is appreciably high only until $\sim 400^\circ\text{C}$. From that temperature the rate starts to decrease more rapidly. As it was reported by Westmacott and Peck²⁵, using this apparatus, the resultant quenching curves have a long tail which would produce a considerable annealing in certain samples. Thus, we will consider the final temperature of cooling as 400°C , even when all the samples were cooled to near ambient temperatures before opening the furnace. The vacancy concentrations at 1760°C and 400°C are $C_v = 8 \times 10^{-4}$ and $C_v = 2.5 \times 10^{-11}$, respectively, ($E_f = 1.49\text{eV}$ ²⁹, see Fig.11). If no vacancies are lost during the cooling, there is a supersaturation of the order of 2×10^7 after the quenching.

The vacancies in excess may disappear by migration to free surfaces or grain boundaries, by joining dislocations or by condensing on particular lattice planes to form plates or loops. As proposed by Frank³⁰, the condensation of vacancies in FCC metal can occur on $\{111\}$ planes, giving rise to a stacking fault bounded by a dislocation with a Burgers vector $\underline{b} = a/3[111]$, or to a prismatic loop with Burgers vector $\underline{b} = a/2[110]$ or to other defects². The clustering on $\{111\}$ planes has been observed in most of the studied FCC metals such as Al, Cu, Ag, Au, Ni and alloys, especially Al alloys.

The fact that in Pt the vacancy condensation occurs on $\{100\}$ planes, as already shown in Sec.3.1.1, was very surprising because the resultant configuration, after the removal of an atom layer on a $\{100\}$ plane, followed by the collapsing of the adjacent planes (see Fig.12), is a very high energy stacking fault analogous to an A-A type of stacking on $\{111\}$ planes, therefore

very unstable. However, the present and earlier results show that $\{100\}$ loops are very stable, since they remain faulted after cutting the foil surfaces in deformed regions of the foil, and also, after long annealing treatments.

Why then does Pt prefer to form loops on $\{100\}$ planes? and, why are they so stable? These questions will be answered later, after considering the interaction of vacancies with the material impurities.

4.2. Analysis of the Impurities Present in the Two Materials.

Before discussing the quenching experiments, it will be convenient to analyze the impurities present in the samples and their possible interaction with vacancies.

There is a large difference in the concentration of substitutional impurities between the two materials, mainly in Rh, Pd and In content, which have atomic radii slightly smaller than the platinum radius. Since it is believed that only those oversized impurities have a strong attraction with vacancies¹³, the effect of these impurities should not be very critical and can be neglected for the moment. On the other hand, carbon which is the most important interstitial impurity in platinum, is in high concentration in both materials, compared with its solubility limits (see Fig.11).

Interstitial impurities normally occupy octahedral interstices; but only when the impurity/metal atom size ratio R_i/R_m is <0.41 , these impurities can occupy the interstitial position without producing lattice strain. In the specific case of carbon in platinum, $R_i/R_m=0.56$ (see Table I), therefore it is $\sim 37\%$ oversize. Consequently the elastic strain associated with the presence of carbon is large and a strong attraction between carbon and vacancies is expected.

There are three possible interaction mechanisms between carbon and vacancies: (1) the carbon could be dispersed through the lattice as single atoms and become bound to vacancies to form single vacancy/carbon complexes; (2) the carbon could be clustered, and act as strong point of nucleations for vacancies; or (3) the carbon/vacancy complexes could start clustering in some defined crystal planes to form platelike precipitates. The first two possible mechanisms could occur at once, which will be discussed in Sec.4.3. The last possible mechanism will be discussed in Sec.4.4.

4.3. Model I for Carbon-Vacancy Interaction.

4.3.1. Model Formulation

Let us assume that carbon atoms are dispersed throughout the lattice as single atoms, and that even when some carbon clusters may exist at high temperature (before the carbon is in supersaturation), a significant number of clusters start to form when the point of saturation is reached. It is also assumed that heterogeneous nucleation of vacancies in platinum is much more important than homogeneous nucleation. Indeed, by the experimental observations, it seems that homogeneous nucleation is practically inhibited in Pt. The energy required to form the first vacancy nucleus is proportional to the surface energy of the metal and, Pt has a much higher surface energy than any other FCC metal studied ($\gamma_s = 2100 \pm 700 \text{ erg-cm}^{-2}$). This energy barrier could be a reason that homogeneous nucleation is extremely difficult in Pt.

Carbon atoms can start clustering on {100} planes and the strain field associated with these clusters can lead to the formation of loops on {100} planes much easier than on {111} planes. It is assumed the first stable cluster to form these loops is already a plate like cluster on a {100} plane and not a spherical cluster (because of the heterogeneous nucleation)

4.3.2. Experimental Evidence for the Model.

The facts that in Pt B the loops are already present after the quenching and, that in Pt A a long annealing treatment is needed for these defects to form, are very consistent with the present model. In Pt A the carbon concentration is three times larger than in Pt B ($C_c/C_v=0.6$), therefore more vacancies can be trapped by single carbon atoms and also, a larger number of carbon atom clusters may pre-exist as heterogeneous nucleation points when the vacancy supersaturation enough to start the clustering, is reached. Thus, it is more difficult to form the first stable cluster for the vacancy loops to start growing. It is only after long annealing that certain number of vacancy clusters can reach a size large enough to form the stable configurations on {100} planes.

Since the solubility of impurities increases with temperature, for a given carbon concentration the number of carbon atom clusters should increase as the temperature is lowered close to the point of saturation (i.e., close to 670°C for Pt B and 950°C for Pt A). Therefore, when Pt B is quenched from a high temperature, the number of carbon atom clusters, which are already present before the supersaturation of vacancies become enough to start condensing, is smaller than when the quenching is performed from a lower temperature. This is consistent with the observation that larger loops, but in lower density, are observed after the quenching from ~1700°C (see Fig.2). The opposite has been observed in very pure aluminum, in which homogeneous nucleation is believed to be dominant⁵.

There is other evidence which supports the assumptions described in the present model. When little more amount of carbon is added to Pt B (Sec.2.4), the effect is almost similar to the effect of decreasing the quenching temperature. That is, the number of pre-existing nucleation points is increased in such a way that a large density of loops, smaller in size, are observed. It could also happen that a larger number of vacancies are trapped by single carbon atoms, since the vacancy concentration at the loops is lower than that in the quenched

Pt B in the original conditions. However, this difference could be due only to the inaccuracy in the determination of the volumetric loop density. Since the inaccuracy in the estimation of the foil thickness (Sec.3.1.2) and since the transparent regions of the thin foils were always very small and not uniform, the estimation of the loop density was only an approximate value.

When much more carbon was added to the foils by heating the samples with graphite powder at 1500°C, Sec.2.4, the number of possible heterogeneous nuclei and the number of vacancies trapped by single carbon atoms are increased, to the point where the formation of the loops is totally inhibited.

Finally, a consistent explanation holds for the observation that, no defects are formed when the quenching is performed in a poor vacuum or in air. It could be that either, the carbon content is reduced to a very low level, where no clusters can be formed (no nucleation points), or that vacancies can be trapped by carbon and oxygen atoms together and no condensation is possible.

4.3.3. Stability of the Loops.

Assuming that the (100) loops are pure vacancy loops, the high energy of these defects can be reduced by an unfauling mechanism which leads to the formation of perfect loops. For a sessile Frank loop, this process occurs generally by the nucleation of a Shockley partial, which then sweeps across the stacking fault transforming the faulted defect into a perfect glissile loop^{5,36}. Such a mechanism seems to be much more difficult to occur for a fault lying on a (100) plane, since there is not a simple partial dislocation able to move by shearing across this kind of plane to sweep away the stacking fault.

However, one could think that the energy of the configurations is high enough to make these defects very unstable and therefore, likely to be unfaulted by any mechanism no matter how difficult. But the loops remain faulted and they grow to a relative large size during the

annealing. This makes it seem doubtful that the loops are only metastable.

Therefore considering the defects as pure vacancy loops there is not a clear explanation for the stability shown by them. An alternative model is proposed in Sec.4.4.

4.3.4. Burgers Vector of the Annealed Defects.

By the contrast analysis of the loops formed in the material after the 24 hours of annealing at 400°C, or after 1 hour of annealing at temperatures higher than 450°C, it was determined that the Burgers and displacement vectors are normal to the habit plane of the loops. Because the fringes produced by the defects are not visible when the diffraction vector is of the type 220, it could be concluded that the magnitude of the Burgers and displacement vectors is $a/2$. However, a displacement vector with this magnitude should give rise to a π type of fringes; and it has been determined that the fringes correspond to a $2\pi/3$ type rather than π or δ fringes. The conclusion that the fringes are of $2\pi/3$ type comes from the facts that they are symmetrical in bright field, asymmetrical in dark field, the branching occurs at the center of the fringe pattern, and the fringe contrast was affected by small changes in the magnitude of the deviation from the Bragg position.

On the other hand, it was determined without doubt, that loops are of vacancy type and also that fringes correspond to defects intrinsic in nature.

Using the hard sphere model, it is impossible to explain the existence of a single vacancy type loop on a (100) plane, with a Burgers vector perpendicular to the plane and magnitude $a/2$. The discrepancy could be explained if defects are thought as two layers of faulted defects. It may be possible that, the interaction between the two layers of defects gives rise to contrast behavior different to the contrast produced by any of the other defects studied. The second defect can start growing on the parallel plane, next to the first defect adjacent plane, and the growth would continue until the the dislocations around the defects form a dipole, process

which would decrease the dislocation line energy.

The above idea is supported by the observation of some double defects, in which the second layer was still smaller in size with respect to the first layer, on specimens of Pt B which were annealed only 12 hours at 400°C. Examples are shown in Fig.14. Moreover, it can be observed in the micrograph of Fig.6 one of the loops does not show fringe contrast in the center part, but only some very faint residual contrast. This configuration can be explained as a three layer faulted defect, with a displacement vector $R = a/3 \langle 100 \rangle$ for each one. The overlapping of three layers should make the fringes disappear, as it is observed in the loop A of Fig.5.

There is other alternative explanation for the discrepancy in the contrast behavior of the secondary defects after the annealing, which will be discussed later in terms of the second model proposed to explain the (100) type of secondary defect formation in Pt.

4.4. Model II for Carbon-Vacancy Interaction.

Since both, carbon and vacancies, are in supersaturation at 400°C in both materials, see Fig.11, the precipitation of both should occur. Let assume then that this process occurs by a co-precipitation mechanism.

Using the hard sphere model and taking into account only the lattice strain due to the carbon atoms and vacancies in solution, the total energy of the system can be decreased if the configuration of the complexes is such that a carbon atom and a vacancy are next nearest neighbors. This configuration has been schematically represented in Fig.13b, where carbon atoms are located in the substitutional positions. Since interatomic potentials have not been considered in this simple model, the final stable position of carbon has not been calculated. The stable posi-

tion should be somewhere in between the substitutional and the normal interstitial position^{16,32}, but to determine the theoretical final stable configuration is a matter of more complicated calculations.

Now, once the complexes are formed, they can condense in particular lattice planes in the same way as vacancies do. Comparing the precipitation of these complexes with normal vacancy precipitation on {111} planes, if a complete collapse to form a stable defect with $b=a/3\langle 111 \rangle$ occurs, the carbon atoms will be forced to be again in interstitial positions with a consequent increase in the energy of the configuration. On the other hand, if precipitation occurs on {100} planes, the limited relaxation of the atoms in the $\langle 100 \rangle$ directions, due to the contact between adjacent atoms, still leaves holes sufficiently large for the accommodation of the carbon atoms in a stable position without producing extra strain in the lattice. See in Fig.13 the schematic sequence of these carbon/vacancy plate precipitate formation on {100} planes, which lead the final configuration of an $a/3\langle 100 \rangle$ loop.

The last configuration of Fig.13d is the same configuration for the case of pure vacancy condensation on {100} planes shown in Fig.12, i.e., an imperfect dislocation loop with $b=a/3[100]$. In this model, due to the presence of carbon atoms, an unfaulting by shear cannot occur since this process would have to be accompanied by a return of the carbon atoms to normal interstitial sites, making the process energetically unfavorable.

Once the plate like precipitates of vacancies and carbon are formed, more complexes and carbon atoms can migrate to the defects during the annealing. The final precipitate configuration after long annealings can be interpreted as two layers of vacancies with a layer of carbon atoms in between. Because the carbon plate like precipitates are very thin they do not contribute to the diffraction process, therefore they can not give rise to a δ fringe pattern. The fringes in this case will be only due to the displacement of the lattice, normal to the precipitate habit plane, therefore, they will be α fringes with symmetric bright field and asymmetric dark

field behavior.

Because of the configuration of the defects (described above) the fringes correspond to an intrinsic type of defect, which is consistent with the results.

The magnitude of the displacement vector is $a/2$. With this displacement vector the overlapping of two precipitate layers should make the fringes disappear, which would be the case of loop A in Fig.5.

Let us analyze now the influence of the quenching temperature and amount of carbon dissolved in Pt, in the formation of these precipitates.

If it is assumed that no vacancies are lost during the quenching, the ratio of carbon atoms to vacancies, C_v/C_v , is 0.2 when Pt B is quenched from $\sim 1760^\circ\text{C}$. Now, when the quenching temperature is lowered to 1300°C , C_v/C_v is 3.2. With this ratio equal to 3.2, loops are still formed after the quenching. Analyzing now the case of Pt A, when the foils are quenched from $\sim 1760^\circ\text{C}$, the ratio C_v/C_v is 0.6 and the loops are not formed in this case.

The later point is still not perfectly explained by the present model. One possible explanation is that substitutional impurity clusters can be points of nucleation for the carbon/vacancy complexes. Because in Pt A these clusters are more difficult to form (since substitutional impurities are in a lower level) an annealing is necessary to form the precipitates on {100} planes.

Substitutional impurity clusters acting as nucleation points for the complexes would explain also the formation of a larger density of loops in Pt B when the quenching temperature is lowered to 1300°C .

However, if the above explanation account for the differences in the precipitate formation between Pt A and Pt B, the same explanation can not account for the formation of a

higher precipitate density when more carbon is added to Pt B.

Although the present model can explain very well the stability of the loops and the discrepancies between the displacement vector of the defects and the fringe contrast showed by them, the same model can not explain consistently the differences in density and size of the precipitates in the two materials after the different treatments.

On the other hand, the model proposed in Sec.4.3 is consistent with the differences in the defect formation between the two materials and the differences in density and size of the loops after different quenching temperatures. However, it does not explain clearly the discrepancies between the fringe contrast showed by the defects and their displacement vector, and the stability of the loops.

•

To determine accurately the mechanism responsible for the formation of $\{100\}$ loops in platinum it is necessary: (a) to obtain more precisely the carbon solubility limit in Pt; (b) to make theoretical calculations of the contrast behavior of the defects on $\{100\}$ planes under different diffraction conditions; and (c) to obtain accurate values of the carbon concentration present in the samples used.

5. ACKNOWLEDGMENTS

I would like to express my gratitude to Dr.K.Westmacott, who guided this research with active interest and enthusiasm. Special appreciation is extended to professor G.Thomas and his group for the stimulating atmosphere and valuable friendship, and to professor J.Washburn for his critical review of the manuscript. Special thanks are due to Dr.R.Gronsky for his valuable cooperation and encouragement, and to Dr.U.Dahmen for many useful discussions and his interest in this work.

Acknowledgment is due to Carolyn Gosnell for her efficient assistance and patient cooperation in solving those many little problems with the laboratory equipments, and to the technical staff of the Lawrence Berkeley Laboratory who provided valuable support.

I specially would like to thank my dear family for the constant moral support. Words cannot express the love and gratitude I owe to my husband, Francisco, whose continuous help, encouragement and understanding throughout these years have been more than important to me.

This work was supported by the Materials and Molecular Research Division of the U.S. Department of Energy, under contract No.W-7405-ENG-48. I gratefully acknowledge the partial support of P.E.O. International Scholarship Fund, which made in part possible my stay at this University.

6. REFERENCES

1. Doyama, M., *Lattice Defects in Quenched Metals*, Cotterill, R.M.J. *et al.* eds., Academic Press (1965), p.185.
2. Thomas, G. and Washburn, J., *Rev. Modern Phys.*, 35(1963)992.
3. Westmacott, K.H., Barnes, R.S., Hull, D. and Smallman, R.E., *Phil. Mag.*, 6(1961)929.
4. Westmacott, K.H., *Phil. Mag.*, 24(1971)475.
5. Cotterill, R.M.J. and Segall, R.L., *Phil. Mag.*, 3(1963)1105.
6. Hirsch, P.B., Silcox, J., Smallman, R.E. and Westmacott, K.H., *Phil. Mag.*, 3(1958)897.
7. Yoshida, S., Kiritani, N. and Shimomura, J., *J. Phys. Soc. Japan*, 18 suppl. III (1963) 175.
8. Strudel, J.L., Vincelle, F. and Washburn, J., *Appl. Phys. Letter*, 3(1963)148.
9. Kiritani, N., Yoshida, S. and Shimomura, J., *J. Phys. Soc. Japan*, 18 suppl. III (1963) 47.
10. Cotterill, R.M.J., *Lattice Defects in Quenched Metals*, Cotterill, R.M.J. *et al.* eds., Academic Press (1965), p.97.
11. Damask, A.C. and Dienes, G.J., *Phys. Rev.*, 120(1960)99.
12. Johnson, R.P., *Phys. Rev.*, 56(1939)814.
13. Damask, A.C. and Dienes, G.J., *Acta. Met.*, 12(1964)797.
14. Cattaneo, F. and Germagnoli, E., *Phys. Rev.*, 124(1961)414.
15. Kino, J., Kabemoto, S. and Yoshida, S., *J. Phys. Soc. Japan*, 18 suppl. III (1963) 85.
16. Beeler, J.R. Jr., *Interatomic Potentials and Simulation of Lattice Defects*, Gehlen, P.C. *et al.* eds., Plenum Press (1972), p.19.
17. Quere, Y., *J. Phys. Soc. Japan*, 18 suppl. III (1963) 91.
18. Westmacott, K.H. and Perez, M.I., *J. Nuclear Mater.*, 83(1979)231.
19. Forouhi, R., Private communication.
20. Ruedl, E., Delavignette, P. and Amelinckx, S., *Radiation Damage in Solids*, Vienna: IAEA, 1(1970)363.
21. Cizek, A., Parizek, F., Orlova, A. and Tousek, J., *Czech. J. Phys.*, B20(1970)56.
22. Jackson, J.J., *Lattice Defects in Quenched Metals*, Cotterill, R.M.J. *et al.* eds., Academic Press (1965), p.467.

23. Westmacott, K.H., *J.Phys.F: Metal Phys.*, 3(1973) L217.
24. Westmacott, K.H., *Crystal Lattice Defects*, 6(1976) 203.
25. Westmacott, K.H. and Peck, R.L., *J.Phys.E: Scientific Instruments*, 9(1976) 521.
26. Dahmen, U., Ph.D.Thesis, Department of Materials Science and Mineral Engineering, Univ. of California, Berkeley (1979).
27. Hirsch, P.B., Howie, A., Nicholson, R.B., Pashley, D.W. and Whelan, M.J., *Electron Microscopy of Thin Crystals*, Butterworth (1965).
28. Foll, H. and Wilkens, M., *Phys.Stat.Sol.(a)*, 31(1975) 519.
29. Gevers, A., Art, A. and Amelinckx, S., *Phys.Stat.Sol.*, 3(1963) 1563.
30. Frank, F.C., *Proc.Phys.Soc.*, A62(1948) 202.
31. Swalin, R., *Acta Met.*, 5(1957) 443.
32. Blanter, M.S. and Khachatryan, A.G., *Met.Trans.*, 9A(1978) 753.
33. Selman, G.L., Ellison, P.J. and Darling, A.G., *Platinum Met.Rev.*, 14(1970) 14.
34. Siller, R.J., Oates, W.A. and McLellan, R.B., *J.Less Comm.Met.*, 16(1978) 71.
35. *Gase Und Kohlenstoff in Metallen*, Springer, Berlin (1976), p.650.
36. Saada, G.V., *J.Phys.Soc.Japan*, 18 suppl.III(1963) 41.
37. Edington, J.W., *Practical Electron Microscopy in Materials Science*, Philips, 2(1975) 37.
38. Blakely, J.M. and Mykura, H., *Acta Met.*, 10(1962) 565.
39. Schumacher, D., Seeger, A. and Harlin, D., *Phys.Stat.Sol.*, 25(1968) 539.
40. Clemenson, M., Novakov, T. and Markowitz, S., *Anal.Chem.*, 51(1979) 572.

7. FIGURE CAPTIONS

- Fig.1. Micrographs illustrating diffraction contrast behavior for loops on {100} planes with $b=a/3 < 100 \rangle$.
- Fig.2. Comparison of the defect structures in Pt B quenched from different temperatures. (a) 1760°C. (b) 1500°C. (c) 1300°C. (d) 1000°C.
- Fig.3. Loop analysis of Pt B quenched from ~1760°C and annealed at 400°C during 24 hours. All micrographs are two beam bright field images taken with different diffraction conditions, indicated in the micrograph. Loops α are similar to those in the as quench material. Loops β are developed after the annealing. 'a' and 'b' are loops used in the analysis of the defect nature. The diffraction patterns corresponding to micrographs 3a-g are shown in 3h-n, respectively.
- Fig.4. Stereographic projections of $\bar{1}12$, 011 and $\bar{1}11$ orientations used in the trace analysis of micrographs of Fig.3.
- Fig.5. Pt B quenched from ~1760°C and annealed at 550°C during one hour. (*) indicates small loops which are believed to be disappearing during the annealing. A indicates a multiple loop.
- Fig.6. Bright field and dark field images of a large defect cutting both surfaces of the foil. The analysis shows that this defect is intrinsic in nature.
- Fig.7. Pt A quenched from ~1760°C and annealed at 400°C during one hour.
- Fig.8. Micrographs showing the secondary defect structure in Pt A after quenching and annealing for 24 hours at 400°C.
- Fig.9. Micrograph showing the effect of adding a small amount of carbon to Pt B. The annealing conditions are similar than for samples of Fig.1 and Fig.2a. See text for details.
- Fig.10. Micrograph and corresponding selected area diffraction patterns of Pt B, after an annealing at 1500°C during two hours in contact with graphite powder, before the quenching at ~1760°C and a final annealing at 400°C during 24 hours. Note the weak extra spots in the diffraction pattern of 7c, taken from the same area in an approximately exact orientation.
- Fig.11. Graph of carbon and vacancy concentrations as a function of temperature. Two alternative curves for the carbon solubility (Ref.33,34) are plotted, but the present work is based on the data of Ref.33 as reported by Ref.35.
- Fig.12. Final configuration after the condensation of vacancies on a {100} plane and the relaxation of the lattice to form an $a/3[100]$ loop.
- Fig.13. Schematic representation of the events leading to the formation of an $a/3[100]$ loop by a co-precipitation of carbon and vacancies. (a) Isolated vacancies and carbon atoms. (b) Vacancy/carbon complexes formation. (c) Precipitation of the complexes on {100}

planes. (d) Final configuration of an $a/3[100]$ loop.

Fig.14. Pt B quenched from $\sim 1760^{\circ}\text{C}$ and annealed 12 hours at 400°C . Double loops are noted by arrows.

8. TABLES

TABLE 1.- Atomic Misfit Values Interstitial Atoms in FCC Metals.

Interstitial Solute.		Carbon, $R_C=0.77\text{\AA}$		Oxygen, $R_O=0.65\text{\AA}$		Nitrogen, $R_N=0.53\text{\AA}$		Hydrogen, $R_H=0.37\text{\AA}$	
Solvent Metal	$R_m (\text{\AA})$	R_C/R_m	Oversize (%)	R_O/R_m	Oversize (%)	R_N/R_m	Oversize (%)	R_H/R_m	Oversize (%)
Nickel	1.24	0.62	51	0.52	27	0.43	5	0.30	-27
Copper	1.27	0.61	49	0.51	24	0.42	2	0.29	-29
γ -Iron	1.29	0.60	46	0.50	22	0.41	0	0.29	-29
Platinum	1.38	0.56	37	0.47	15	0.38	-7	0.27	-34
Aluminum	1.43	0.54	32	0.45	10	0.37	-10	0.26	-37
Silver	1.44	0.53	29	0.45	10	0.37	-10	0.26	-37
Gold	1.44	0.53	29	0.45	10	0.37	-10	0.26	-37
Thorium	1.80	0.43	5	0.36	-12	0.29	-29	0.21	-49

TABLE II.- Chemical Analysis of the Materials.†

Material	Rh	Pd	Ir	Si	Ag	Fe	Ti	C‡	Nominal Purity
Pt A	40	-	-	70	5	52	20	480	99.99%
Pt B	3000	950	300	140	-	70	-	<160	99.76%

† Figures given in appm.

‡ Analysis made in a Leco low-carbon analyzer.

TABLE III.- Values of g and $\alpha(-2\pi g \cdot R)$ for $a/3(100)$ Loops.

g	$\underline{b} = \pm 1/3[100]$	$\underline{b} = \pm 1/3[010]$	$\underline{b} = \pm 1/3[001]$	$\underline{R} = \pm 1/3[100]$	$\underline{R} = \pm 1/3[010]$	$\underline{R} = \pm 1/3[001]$
$\bar{1}\bar{1}1$	$\pm 1/3$ (I)	$\pm 1/3$ (I)	$\pm 1/3$ (I)	$\pm 2\pi/3$ (V)	$\pm 2\pi/3$ (V)	$\pm 2\pi/3$ (V)
200	$\pm 2/3$ (V/I)	0 (I)	0 (I)	$\pm 4\pi/3$ (V)	0 (I)	0 (I)
$\bar{2}02$	$\pm 2/3$ (V/I)	0 (I)	$\pm 2/3$ (V/I)	$\pm 4\pi/3$ (V)	0 (I)	$\pm 4\pi/3$ (V)
$\bar{1}\bar{1}3$	$\pm 1/3$ (I)	± 1 (V)	± 1 (V)	$\pm 2\pi/3$ (V)	$\pm 2\pi$ (I)	$\pm 2\pi$ (I)

(I) Dislocation loop or stacking fault in weak contrast.

(V) Dislocation loop or stacking fault in strong contrast.

TABLE IV.- Loop and Vacancy Densities in Pt B for Different Quenching Temperatures.

Quenching Temperature ($^{\circ}\text{C}$)	Equilibrium Vacancy Concentration	Loop Density ($1/\text{cm}^3$)	Vacancy Concentration Stored at Loops
1760	8.0×10^{-4}	3.0×10^{14}	4.0×10^{-4}
1500	2.5×10^{-4}	3.6×10^{14}	2.0×10^{-4}
1300	5.0×10^{-5}	5.0×10^{14}	4.0×10^{-5}
1000	5.2×10^{-6}	-	-

9. FIGURES

XBB 780-13279

Fig. 1

XBB 790-15985

Fig. 2

XBB 790-15975

Fig 3

XBB 790-15976

Fig 3 cont

XBB 790-15977

Fig. 3 cont.

Fig 4

XBL 7911-14556

Fig 5

XBB 790-15982

Fig. 6

Fig. 7

XBB 790-15983

Fig. 8

Fig. 9

XBB 790-15984

Fig. 10

X BL 789-5815

Fig 11

XBL7911-7289

Fig 12

(a)

(b)

(c)

(d)

LOCATION OF CARBON ATOM

XBL 789-5816

Fig 13

XBB 790-15981

Fig. 14