FIELD ION MICROSCOPE STUDIES OF SOLUTE ATOMS IN DILUTE
PT ALLOYS. I. CONTRAST DUE TO AU, NI AND W ATOMS

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ABSTRACT

Platinum based alloys containing dilute concentrations of either gold, nickel or tungsten atoms were examined in the field ion microscope during pulsed field evaporation. The contrast patterns produced by the solute atoms were established by comparing the images of the alloy specimens, which contained known concentrations of solute atoms, and the images of pure platinum control specimens. The gold and nickel atoms appeared as α -dark spots, i.e., single dark atomic sites lying within net planes which were visible during all stages of the pulsed field evaporation. In many cases platinum atoms near solute atoms were preferentially field evaporated producing β -dark spots, i.e., single dark atomic sites which appeared suddenly within a well resolved net plane during observation and remained there until all the atoms of that plane evaporated. The tungsten atom contrast was more complex and generally consisted of a cluster of contrast effects which often contained multiple α -dark spots, β -dark spots and also bright spots.

1. INTRODUCTION

In the present work (Part I) we establish the type of field ion microscope (FIM) image contrast produced by individual Au, Ni and W atoms in dilute solution in a Pt matrix. With the aid of this information we then proceed in the following Part II⁽¹⁾ to determine directly the radial distribution functions for Au and Ni atoms by direct observation of their lattice positions in the bulk during pulsed field evaporation.

DuBroff and Machlin⁽²⁾ have carried out a previous FIM study of these same dilute alloy systems with this same objective in mind. They restricted their attention to contrast effects appearing on {102} net planes and found dark atomic sites in the Au-Pt and Ni-Pt alloys and both dark sites and bright spots in the W-Pt system. The concentrations of dark sites in the Au-Pt and Ni-Pt alloys and bright spots in the W-Pt alloy corresponded to the nominal solute atom concentrations in the alloys within a factor of about 2. On the basis of these results they concluded that the dark sites represented the positions of Au and Ni atoms and that the bright spots corresponded to W atoms. They concluded that the "dark site" contrast could have been due to either preferential field evaporation of the solute atoms leaving surface vacancies (dark sites) behind, or to local electron transfer at the solute atom sites. On the other hand, the bright spot contrast may have been due to atoms standing "proud" on the surface or again to local electron transfer. In any case DuBroff and Machlin were careful to emphasize that the determination of the exact mechanism for contrast was unimportant within the context of their work. Of

primary importance was the establishment of a definite type of contrast which could be associated with the lattice position of a given type of solute atom.

The conclusions of DuBroff and Machlin regarding the identification of the types of contrast produced by different solute atoms are questionable, however, for at least several reasons. Firstly, the agreement between the concentration of image effects and nominal solute atom concentration was only approximate. Secondly, the nominal solute atom concentrations themselves were apparently known only roughly. In one case chemical analysis of the solute concentration in the actual specimen used yielded a result which differed from the expected concentration by almost a factor of 2. Thirdly, it has been clearly established elsewhere^(3,4,5) that {102} planes of well annealed pure Pt specimens have artifact vacancy concentrations of the order of $1\frac{1}{2}$ at. % even while operating with a clean FIM having a residual background gas pressure of the order of 1×10^{-9} torr and imaging with high purity helium gas at low temperature (25°K).

In the present work considerable care was taken to avoid these difficulties. Homogeneous alloy specimens were prepared in the form of wires, and their chemical compositions were determined by direct chemical analysis. Also, solute atom contrast effects were observed only on a number of selected {hkl} net planes (excluding {102} planes) which were shown to be free of artifact vacancy effects by means of extensive control tests using pure Pt specimens. In addition, large numbers of sites in the bulk were examined under carefully controlled field evaporation conditions where every site on the selected planes was imaged and recorded on film at least several times during the evaporation.

2. EXPERIMENTAL

2.1 Specimen Preparation

Alloys of 4.0 at. % Au-Pt, 0.62 at. % Au-Pt, 0.65 at. % Ni-Pt and 0.15 at. % W-Pt were prepared. Each alloy was annealed at 1300°C for 100 h in a 5×10^{-6} torr vacuum in order to achieve homogeneity and was subsequently drawn into 5 mil diam. wire. Several portions of the wire were immediately removed for chemical analysis. High purity research grade Pt wire (purity 99.999%), also of 5 mil diam. was obtained from the Sigmund Cohn Corp., Mt. Vernon, N. Y. for use as control specimens.

Before preparing FIM specimens each wire was mounted on a holder similar to that described by Berger⁽⁴⁾, and was heat treated by resistive heating in order to characterize its thermal history. The heat treatments for the different materials were as follows: The Au-Pt wires were first annealed overnight at a high temperature (about 1300°K) and then quenched into a saturated brine solution maintained at -5°C at an approximate quenching rate of 3×10^4 °K sec⁻¹. The Ni-Pt and W-Pt alloys were also annealed at about the same temperature and then air quenched to room temperature by simply cutting off the heating current abruptly. The quenching rate was estimated to be about 3×10^3 °K sec⁻¹(6). The pure Pt wires were also annealed at about 1300°K for several hours and then slowly cooled to room temperature to attain a well-annealed state.

Sharply tipped FIM specimens were obtained from the wires by means of an electro-polishing technique employing a molten salt solution of NaNO₃ and 15% by wt. NaCl at 325°C. The polishing

procedure consisted of periodically dipping the specimen into the solution about 5 mm in depth and quickly removing it after a period of about 0.2-0.3 sec. The total immersion time was about 10-20 sec.

2.2 FIM and Data Recording System

The specimen was then dissected atom by atom in a FIM by pulsed field evaporation and the image was recorded after each pulse on ciné film⁽⁷⁻⁹⁾. Relatively small voltage pulses were applied so that only a few atoms were removed from each net plane per pulse. Each plane evaporates preferentially from its edges and therefore gradually shrinks in size. Every atomic site in each plane was therefore imaged and recorded at least several times during some stage of the overall dissection process. In this technique a cylindrical volume in the specimen tip associated with each resolvable $\{hkl\}$ net plane is sampled as indicated in Fig. 1. Each volume consists of a stack of successive $\{hkl\}$ planes which are revealed and photographed sequentially during the field evaporation.

The FIM and photographic recording system used for this purpose was very similar to that described by Chen and Seidman⁽⁷⁾ and by Scanlan, et al.⁽⁸⁾. The only difference was that the microscope was pumped dynamically by a CVC oil diffusion pump baffled with a Granville-Phillips liquid nitrogen cold trap. A vacuum of 2×10^{-9} torr could easily be obtained after about 12 h of pumping.

The actual operating procedure was quite straightforward. After the specimen was loaded into the FIM, the system was baked at $\sim 150^\circ\text{C}$ for 2 h and pumped until a pressure of 2×10^{-9} torr was reached. The specimen was then cooled down to about 30°K by means

of a continuous-transfer liquid helium cryostat⁽¹⁰⁾ attached to the specimen mounting block. Research grade He was leaked in through a Varian leak valve continuously at a gauge reading about 3×10^{-5} torr and the whole system was operated in a dynamical mode. The tip was then slowly pulse field evaporated at a rate of about 50 pulses per atomic layer and the corresponding image was recorded on 1000 ft rolls of ciné film until sufficient data were accumulated. The film was then developed and analyzed.

2.3 Analysis of FIM Film

A Vanguard Motion Analyzer was used to scan the rolls of film. This machine could be operated forwards or backwards with a continuously variable speed up to $24 \text{ frames sec}^{-1}$. Single frame advance (or reverse) could also be achieved by pushing a button. In addition, movable X and Y crosshairs were available on the viewing screen, and the coordinates of these crosshairs were displayed continuously on digital readout meters with a sensitivity of ± 0.0005 in. This feature is very important because, in Part II, it was used to measure the coordinates of individual solute atoms.

Each film roll was run through the analyzer in a forward direction, and one particular crystallographic plane (Fig. 1) was watched during each scan. With the aids of the backward operational mode and the single frame advance and reverse, each plane was examined in great detail. The numbers of atoms and defects observed in each crystallographic plane during the field evaporation sequence were recorded layer by layer.

The criteria which were employed for counting both atoms and defects are as follows:

- (1) For smaller net planes (≤ 20 atoms), we counted both atoms and defects within the first ring (see Fig. 2) immediately after the last atom of the preceding plane was evaporated.
- (2) For larger planes (≥ 20 atoms), we counted both atoms and defects within the first ring when the first two outer rings of atoms became visible even though several atoms of the preceding plane still remained near the center.

The first ring of atoms was disregarded in order to avoid any ambiguity which might arise if a defect, say a vacancy, happened to occupy a site belonging to the first ring. In such a case it is impossible to decide whether such a vacancy is actually associated with a bulk defect or whether it is simply due to irregular field evaporation from the edge of the plane. If one or two atoms were missing along the first ring, a smooth ring was assumed. Such a case is shown in Fig. 2 where the first ring was taken to be the solid line, and only sites within the ring were counted. By doing this we avoided any possible bias due to the presence of defects. Usually, the shape of the outer ring of atoms for a particular plane remained roughly the same throughout the entire field evaporation sequence.

3. RESULTS

3.1 Chemical Analysis

Two samples from each alloy were sent to both the Analytical Chemistry Laboratory of the Materials Science Center at Cornell

University and the U. S. Testing Company for chemical analysis to determine the actual solute contents. The results are shown in Table 1.

3.2 Pure Platinum Control Specimens

A FIM image of a well annealed pure Pt specimen is shown in Fig. 3. One octant of the (001) oriented Pt specimen is indexed. The net planes which we examined were {315}, {203}, {517}, {214} and {213}.

Single artifact vacancies were the only defects ever observed on the surfaces of the control specimens. The final total atom and artifact vacancy counts obtained from these planes in all four control specimens are listed in Table 2. In most cases the artifact vacancy concentrations were of order 10^{-5} atom fraction. In no cases did they ever exceed 1.5×10^{-4} atomic fraction. Since the solute contents of the alloys were about 15×10^{-4} atom fraction, or higher, we concluded that these five planes were suitable for the present alloy studies.

3.3 Alloy Specimens

All the alloy specimens were run under the same experimental conditions as those of the control specimens. Several distinct point defect contrast patterns were observed in the alloys at concentrations much larger than any possible artifact vacancy concentrations. We may classify them into the following three elementary categories:

- (i) α -dark spot: a single dark atomic site lying within a net plane. It is visible during all stages of the imaging of the plane during field evaporation (e.g., frames 5-8 in Fig. 4).
- (ii) β -dark spot: a single dark atomic site which suddenly appears within a well-resolved net plane during observation and remains there until all the atoms of that plane are evaporated (e.g., frame 7 in Fig. 4 and frame 2 in Fig. 5a). Once formed, β -dark spots are identical to α -dark spots.
- (iii) Bright spot: a single spot of atomic dimensions which appears much brighter than the nearby atoms (e.g., Fig. 6). In the great majority of cases the bright spots occurred at the positions of normal lattice sites. However, in some cases they were observed at apparent interstitial positions.

Various combinations of the above three elementary contrast patterns were observed in the different alloys as discussed below.

Au-Pt alloys

Both α - and β -dark spots were observed in the Au-Pt alloys. No bright spots were found. Figure 4 shows the field dissection of four successive $\{315\}$ planes of a 4.0 at. % Au-Pt specimen. Three α -dark spots and one β -dark spot were found between frames 5 and 18. Figure 5a shows the field dissection of six successive $\{203\}$ planes of a 0.62 at. % Au-Pt specimen. One α -dark spot and one β -dark spot were found between frames 2 and 17.

The α - and β -dark spots were counted in a number of specimens of each alloy, and the results are summarized in Tables 3 and 4. Comparing Tables 3 and 4 with Table 2, we can see that, due to the presence of Au atoms, a large number of α -dark spots and β -dark spots were produced. The concentration of α -dark spots is plotted in Fig. 7 as a function of $\{hkl\}$ for both alloys, and is seen to be independent of $\{hkl\}$ and in good agreement with the concentration of Au atoms obtained by chemical analysis. We therefore conclude that the α -dark spots correspond to the positions of Au atoms. Either each α -dark spot corresponds to the surface vacancy left behind due to the preferential field evaporation of a Au atom or else the Au atom is still present but appears dark due to a redistribution of charge⁽¹¹⁾.

The β -dark spots were not randomly distributed but were found to form near α -dark spots. An excellent example is shown in Fig. 5a. The β -dark spot in frame 2 was found to be almost exactly on top of the α -dark spot in frames 15, 16 and 17. By studying the atomic geometry of the $\{203\}$ planes and the geometrical relations between successive layers, we found that this β -dark spot was one of the first nearest-neighbors of the α -dark spot. A ball model of the geometry is shown in Fig. 5b. The β -dark spot in the uppermost $\{203\}$ plane (plane 0) is represented by a missing black ball. The underlying α -dark spot occurred at the nearest-neighbor site in layer 5 marked by the arrow in Fig. 5b. This pattern of association between α - and β -dark spots was observed repeatedly.

A more complete study of the geometrical relationship between α - and β -dark spots was performed using the computer technique described in Part II⁽¹⁾. In this method the lattice coordinates of all the α - and β -dark spots were first obtained. The distances between any given β -dark spot and all α -dark spots appearing in successive underlying planar layers were then calculated. By analyzing 665 β -dark spots in this fashion it was found that 85% of the β -dark spots had at least one underlying α -dark spot in a first nearest-neighbor position. Also, careful examination of any β -dark spots which appeared singly showed that they were very close to the edge of the planar layer under observation. It therefore is quite possible that these β -dark spots had one, or more, first nearest-neighbor solute atoms (α -dark spots) lying just outside the planar region under observation. It is also noted that among the six nearest-neighbor Pt atoms* in layers above an underlying Au atom, the one located closest to the axis passing through the Au atom and perpendicular to the surface was usually preferentially evaporated. Thus, we conclude that the β -dark spots were surface vacancies produced by the sudden preferential field evaporation of Pt atoms in close proximity to underlying Au atoms.

It was also noted that the fraction of β -dark spots relative to α -dark spots for the 4.0 at. % Au-Pt alloy was about $\frac{1}{4}$, and that this fraction increased to about $\frac{1}{2}$ for the 0.62 at. % Au-Pt alloy. This effect might be expected on the basis of the above conclusion,

* For the {hkl} planes investigated, every surface atom had six first nearest-neighbors in underlying layers.

since overlap effects between the Au atoms in the more concentrated alloy would be expected to reduce their efficiency in producing β -dark spots.

Ni-Pt alloy

The solute atom contrast effects in this alloy were qualitatively similar to those found in the Au-Pt alloys. Both α - and β -dark spots were found, and no bright spots were observed. The complete results are given in Table 5. The concentration of α -dark spots agreed well with the concentration of Ni atoms, and we again conclude that the α -dark spots correspond to the positions of the solute atoms, i.e., the Ni atoms. Furthermore, the positions of the β -dark spots were highly correlated with the positions of the α -dark spots as in the case of the Au-Pt alloys. We may therefore conclude that the probability of the field evaporation of a Pt atom is increased when it is near a Ni atom. The origin of the β -dark spots is therefore basically similar to that in the Au-Pt alloys. However, the relative concentration of β -dark spots was lower than that in the corresponding Au-Pt system.

W-Pt Alloy

All three kinds of contrast patterns, i.e., α - and β -dark spots and bright spots, were found in this alloy. The results are summarized in Table 6. For this alloy neither the concentration of α -dark spots, β -dark spots or bright spots agreed with the solute atom concentration. However, careful examination of the film showed that most of the defects appeared in the form of rather

discrete clusters. A typical cluster consisted of a sequence of several α - or β -dark spots, then a bright spot, and then α -dark spots again. Figure 6 shows a field dissection of six successive {315} planes revealing such a cluster. One α -dark spot was found on the second layer and two more were found on the third layer. The fourth layer contained a bright spot which became visible in frame 15. Three more α -dark spots were then found below this bright spot on the fifth and sixth layers. Clearly all these defects were closely associated, and, in fact, a more detailed analysis proved that all of the α -dark spots were actually nearest neighbors of the bright spot. Figure 8 shows the lattice geometry of these seven defects. Clusters containing only α -dark spots were also observed. These clusters consisted of a series of dark spots similar to the example which we have described above except that no bright spot was detected. Occasionally, one or two β -dark spots were also found within a cluster.

The concentration of α -dark spots was almost one order of magnitude higher than the corresponding solute concentration as seen in Table 6. Furthermore, the concentration of bright spots was only about half of the W atom concentration. However, the concentration of clusters was found to be in good agreement with the W atom concentration obtained by chemical analysis. We therefore believe that to a good approximation each cluster was produced by a single W atom. Each W atom apparently acted as a center of perturbation in the lattice, and several dark or bright spots were associated with it.

In order to delineate more clearly the overall geometry of the clusters a computer technique⁽¹²⁾ was used which was capable of displaying the 3-dimensional spatial distribution of the defects on a cathode ray tube. In this method the coordinates of all the defects are specified, and their positions are displayed yielding a 2-dimensional view of the entire structure. A valuable feature of this technique is that it can also provide real-time rotation of the entire structure around any axis, thus creating an impression of 3-dimensionality. This feature enables one to examine the structure from all angles. An example is shown in Fig. 9 where each sharp bend represents the position of a defect. (The rectangular coordinate box is included merely to create an illusion of 3-dimensionality.) The volume of the alloy containing the defects in Fig. 9 was a cylinder with its axis approximately along $\langle 315 \rangle$ which contained 6414 lattice sites and 70 defects. The four views from different directions in Fig. 9 indicate that the defects were associated in eight clusters of various sizes as denoted by the arrows in Frame 3.

4. DISCUSSION

The present result that the positions of Au and Ni solute atoms appear as α -dark spots was established by:

- (i) working with selected $\{hk\ell\}$ net planes which were shown to be relatively free of artifact vacancies.
- (ii) using a fine-scaled field evaporation technique in which all sites associated with each selected net plane were imaged clearly.

(iii) carefully distinguishing between the α -dark spots and the β -dark spots which were observed and counted for the first time.

As mentioned previously, the α -dark spots in the Au-Pt and Ni-Pt alloys could have been due to either the preferential field evaporation of the solute atoms or else to local charge transfer. The present experiments did not allow us to decide between these possibilities. This question could probably be resolved by the use of an Atom Probe-FIM⁽¹³⁾ where the charge-to-mass ratio of field evaporated atoms can be analyzed by time-of-flight mass spectroscopy.

The conclusion of DuBroff and Machlin⁽²⁾ that Au and Ni solute atoms appear as dark spots is similar to ours. However, these authors did not distinguish between α - and β -dark spots, and their conclusion was not firmly established since it was based on data which were only semi-quantitative at best.

The β -dark spots in the Au-Pt and Ni-Pt alloys must have been due to the preferential field evaporation of Pt atoms which were either nearest-neighbors or near-neighbors of underlying solute atoms. In general, the solute atoms may have caused the enhanced field evaporation of nearby Pt atoms by reducing their binding energy and/or producing displacements at the surface by means of their dilatational fields which enhanced the local electric field causing field evaporation. Looking at the electronic structures of Pt and Au atoms, we note that Pt has an incomplete d-shell while Au has a complete d-shell. Since an incomplete d-shell makes a substantial contribution to the binding energy, we expect the Pt-Pt binding to be stronger than the Pt-Au binding. Thus, the energy to remove a Pt atom from the surface of the Pt lattice should be reduced

if a Au atom is in a nearest-neighbor site. On the other hand, the electronic structures of both Ni and Pt atoms are similar, since both have an incomplete d-shell. We therefore expect the Pt-Ni binding to be stronger than the Pt-Au binding. Thus, the energy required to remove a Pt atom in juxtaposition to a Au atom is lower compared to a similar situation in the Ni-Pt system. We also note that Au atoms act as positive centers of dilatation in Pt whereas Ni atoms act as negative centers⁽¹⁴⁾. Surface Pt atoms in close proximity to Au atoms would therefore tend to be displaced outwards where they would be subjected to an increased local electric field in contrast to the case for Ni. However, careful examination of the images of Pt atoms which were preferentially evaporated to produce β -dark spots indicated that they were not visibly brighter than the images of other nearby Pt atoms just before evaporation. Any outward displacements therefore could not have been very large.

The observation that the number of β -dark spots was only $\sim \frac{1}{4}$ to $\frac{1}{2}$ the number of α -dark spots in these systems suggests that thermal activation plays a role in the preferential field evaporation process. Other variables which could play a role are the geometry of the plane and the location of the atom within the plane.

The results for the Pt-W system are considerably more complicated. We have already concluded that each W atom produced a perturbation in its vicinity which generally produced a cluster of defects during field evaporation which often consisted of α - and

β -dark spots and also bright spots. The great majority of the α -dark spots must have been Pt atoms which were preferentially field evaporated from sites in the vicinity of the W atoms, since the ratio of α -dark spots to W atoms was about 5. Furthermore, the β -dark spots and bright spots must also have been associated with Pt atoms which were perturbed in some way. These defects were generally observed during field evaporation before the last defect in the cluster, which was always an α -dark spot, was observed. Clearly, once the W atom was removed no further defects should have been observed in a given cluster. Therefore, these defects could not have been W atoms. This result suggests that the last α -dark spot observed in each cluster might have been a W atom. However, it is clear that this result cannot be established firmly on the basis of the present results.

The β -dark spots were evidently due to the preferential field evaporation of Pt atoms near the solute atom as was the case in the Au-Pt and Ni-Pt alloys. The bright spots were evidently due to outward displacements of Pt atoms near the W atoms. This conclusion regarding the origin of the bright spots is contrary to that of DuBroff and Machlin⁽²⁾ who claimed that the bright spots were the W atoms themselves.

The present work does not establish unambiguously the contrast produced directly at a W atom. However, it seems well established that a W atom produces a relatively large perturbation in its vicinity which is capable of producing multiple dark spots and bright spots. The explanation of this effect is not clear at present.

Tungsten is highly soluble in Pt⁽¹⁵⁾, and both metals possess very similar atomic radii⁽¹⁶⁾. Therefore, we would not expect a strong dilatational field around a W atom. It seems therefore that the effect of a W atom on reducing local binding energies must be relatively large. If this were the case the bright spots could have been due to outward displacements of Pt atoms caused by the combined effects of the applied force due to the electric field and a reduced binding force.

In conclusion, we note that the present clusters resemble somewhat the defect contrast patterns observed by Seidman, et al.^(17,18) in their field ion microscope studies of the contrast effects produced by self-interstitial atoms in Pt and W crystals. Multiple bright and dark spot contrast effects were observed in clusters in the vicinity of single interstitial atoms. However, in this case the effects were attributed to displacements associated with the strong dilatational field which is known to exist around self-interstitial atoms.

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Table 1. Chemical Analysis Results

Alloy	Solute Concentration (at.%)		
	Result 1*	Result 2**	Average
Au-Pt (high)	4.19±0.12	3.9±0.2	4.0±0.3
Au-Pt (low)	0.62±0.02	0.61±0.02	0.62±0.03
Ni-Pt	0.67±0.02	0.63±0.02	0.65±0.04
W-Pt	0.15±0.01	---	0.15±0.01

* Reported by Analytical Chemistry Laboratory, Materials Science Center, Cornell University, Ithaca, New York 14853.

** Reported by Chemical Services Division, United States Testing Company, Inc., 1415 Park Avenue, Hoboken, New Jersey 07030.

Table 2. Artifact Vacancy Counts Obtained with Pure Pt Control Specimens

Plane	Lattice Sites Counted	Artifact Vacancies Counted	Conc. of Artifact Vacancies (at.%)
315	213,999	33	1.5×10^{-2}
203	55,551	3	5.4×10^{-3}
517	95,910	9	9.4×10^{-3}
214	92,640	2	2.2×10^{-3}
213	129,969	3	2.3×10^{-3}
Total	588,069	50	8.5×10^{-3}

Table 3. Results for 4.0 at.% Au-Pt Specimens

Spec	Plane	Lattice Sites Counted	α -dark Spots Counted	β -dark Spots Counted	Conc. of α -dark Spots (at.%)
#1	315	20,018	797	207	3.98
	203	6,650	264	24	3.97
	517	4,449	172	26	3.83
	214	3,054	115	17	3.77
	213	1,960	79	21	4.03
	Total	36,131	1,427	295	3.96
#2	315	40,140	1,598	492	3.98
	203	8,008	329	62	4.10
	517	15,153	599	134	3.95
	214	17,764	703	169	3.96
	213	10,080	415	179	4.12
	Total	91,145	3,644	1,036	4.00
Grand Total		127,276	5,071	1,331	3.98

Table 4. Results for 0.62 at.% Au-Pt Specimens

Spec	Plane	Lattice Sites Counted	α -dark Spots Counted	β -dark Spots Counted	Conc. of α -dark Spots (at.%)
#1	315	38,234	249	109	0.65
	203	9,896	61	25	0.62
	517	17,994	102	51	0.57
	214	21,383	132	44	0.62
	213	21,098	141	109	0.67
	Total	108,611	685	338	0.63
#2	315	18,793	117	62	0.62
	203	5,056	39	18	0.77
	517	8,056	58	31	0.72
	214	8,995	56	54	0.62
	213	12,533	74	56	0.59
	Total	53,433	344	221	0.64
#3	315	23,666	140	71	0.59
	203	5,559	38	24	0.68
	517	10,412	58	50	0.56
	214	10,224	70	59	0.68
	213	13,747	90	42	0.65
	Total	63,608	396	246	0.62
#4	315	8,906	53	22	0.60
	203	2,292	15	8	0.66
	517	3,280	21	10	0.64
	214	4,748	31	8	0.65
	213	6,681	41	26	0.61
	Total	25,907	161	74	0.62
Grand Total		251,559	1,586	879	0.63

Table 5. Results for 0.65 at.% Ni-Pt Specimens

Spec	Plane	Lattice Sites Counted	α -dark Spots Counted	β -dark Spots Counted	Conc. of α -dark Spots (at.%)
#1	315	17,231	104	19	0.60
	203	5,345	37	4	0.69
	517	7,211	47	9	0.65
	214	6,557	40	14	0.61
	213	9,611	58	19	0.60
	Total	45,955	286	65	0.62
#2	315	39,340	240	36	0.61
	203	10,826	72	2	0.67
	517	21,676	139	19	0.64
	214	17,091	107	40	0.63
	213	24,763	147	25	0.59
	Total	113,696	705	122	0.62
#3	315	19,472	114	15	0.59
	203	6,710	42	0	0.72
	517	6,965	42	6	0.60
	214	5,887	36	16	0.61
	213	13,604	80	15	0.59
	Total	52,638	320	52	0.61
Grand Total		212,289	1,311	239	0.62

Table 6. Results for 0.15 at.% W-Pt Specimens

Spec	Plane	Lattice Sites Counted	α -dark Spots Counted	Conc. of α -dark Spots (at.%)	β -dark Spots Counted	Conc. of β -dark Spots (at.%)	Bright Spots Counted	Conc. of Bright Spots (at.%)	Clusters Counted	Conc. of Clusters (at.%)
#1	315	23,680	119	0.50	6	0.03	19	0.03	31	0.13
	203	5,658	20	0.35	3	0.05	2	0.04	6	0.11
	517	3,732	56	0.64	6	0.07	10	0.11	14	0.16
	214	7,659	36	0.47	0	0.00	1	0.01	8	0.10
	213	14,940	48	0.32	1	0.01	0	0.00	16	0.11
	Total	60,869	279	0.46	16	0.03	32	0.05	75	0.12
#2	315	92,437	715	0.77	69	0.07	89	0.10	147	0.16
	203	22,671	291	1.29	30	0.13	17	0.03	53	0.23
	517	27,238	242	0.69	18	0.07	24	0.09	46	0.17
	214	25,730	129	0.50	9	0.03	5	0.02	25	0.10
	213	25,110	89	0.35	3	0.01	12	0.05	26	0.10
	Total	193,186	1,466	0.76	129	0.07	147	0.08	297	0.15
#3	315	6,936	56	0.81	2	0.03	7	0.10	12	0.17
	203	924	4	0.43	0	0.00	0	0.00	1	0.11
	517	2,541	10	0.39	2	0.08	1	0.04	3	0.12
	214	2,470	16	0.65	1	0.04	1	0.04	4	0.16
	213	3,262	9	0.28	0	0.00	2	0.06	4	0.12
	Total	16,133	95	0.59	5	0.03	11	0.07	24	0.15
Grand Total		270,186	1,840	0.68	150	0.06	190	0.07	396	0.146

FIGURE CAPTIONS

- Fig. 1. Schematic diagram of the volume associated with a given $\{hkl\}$ net plane which is sampled during field evaporation.
- Fig. 2. Schematic diagram of an $\{hkl\}$ net plane. Only sites within the first ring (solid line) were counted.
- Fig. 3. A field ion image of a well annealed Pt specimen. One octant is indexed.
- Fig. 4. The dissection of four successive $\{315\}$ planes (as shown in frames 1 - 4, 5 - 12, 13 - 19, and 20 - 21) of 4.0 at. % Au-Pt specimen. Open circle represents an α -dark spot. Open circle with a cross represents a β -dark spot.
- Fig. 5. (a) The dissection of six successive $\{203\}$ planes (as shown in frames 1 - 4, 5 - 7, 8 - 9, 10 - 11, 12 - 14, and 15 - 17) of a 0.62 at. % Au-Pt specimen. (b) A ball model of the above $\{203\}$ planes. The center missing atom represents the β -dark spot in frame 2. The numbers indicate the evaporation sequence. The ball indicated by the arrow is the α -dark spot in frames 15 - 17.
- Fig. 6. The dissection of six successive $\{315\}$ planes (as shown in frames 1 - 4, 5 - 8, 9 - 14, 15 - 23, 24 - 26, 27 - 32) of a 0.15 at. % W-Pt specimen revealing a cluster consisting of six α -dark spots (open circles) and one bright spot (open circle with a cross).

Fig. 7. Concentration of α -dark spots in 4.0 and 0.62 at. % Au-Pt alloys as a function of $\{hk\ell\}$ net plane. Error bars represent the statistical uncertainties for each measurement.

Fig. 8. Geometry of the cluster observed in Fig. 6. The number indicates the planar layer in which the defect was found (see Fig. 6). ABC is a $\{315\}$ plane.

Fig. 9. Computer generated display of bulk distribution of defects observed on 381 successive $\{315\}$ planes in 0.15 at. % W-Pt alloy. Each bend represents the position of a defect. Each arrow in frame 3 locates a discrete defect cluster.
 $L = 58a_0$.

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Fig. 2.

(a)

(b)

● (α -dark spot)

⊗ (Bright spot)