NEON GAS IMAGING OF GOLD IN THE FIELD ION MICROSCOPE

by

Robert S. Averback and David N. Seidman

15 March 1973

Cornell University

Ithaca, New York

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Atomic Energy Commission, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

Report #1973

Issued by

The Materials Science Center

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

leg

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor 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, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

NEON GAS IMAGING OF GOLD IN THE FIELD ION MICROSCOPE*

by

Robert S. Averback and David N. Seidmant

Cornell University
Department of Materials Science and Engineering
Ithaca, New York 14850
U.S.A.

ABSTRACT

An improved field ion microscope (FIM) technique has been developed for the neon gas imaging of gold specimens. The technique produces images which are stable at best image voltage at a tip temperature (T_p) of 30 K or less. The first stage of the technique consisted of the development of an end form at 55 K in the presence of a partial pressure of air ($\sim 2 \times 10^{-8}$ Torr gauge pressure) and neon gas ($\sim 3 \times 10^{-5}$ Torr gauge pressure) followed by further field evaporation at 28 K. The second stage involved neon gas imaging of the previously developed end form in a baked FIM in a background pressure of $(0.5 \text{ to } 3) \times 10^{-9}$ Torr. The FIM images obtained in conjunction with the field ionization characteristic curves showed that there is a working range (in the sense defined by Southon and Brandon). A detailed study was made of artifact vacancies detected on the {203}, {321}, {315}, {421}, {671}, and {731} planes, and it was found that at 28 K their concentration was $< 2.5 \times 10^{-3}$ at. fr. Approximately 191,000 atomic sites were examined for artifact vacancies. The artifact vacancy concentrations measured in the present study were a factor of 13 to 60 lower than those measured earlier by Schmid and Balluffi who employed a background pressure of $\sim 5 \times 10^{-8}$ Torr in their FIM. Hence, the artifact vacancy concentrations detected in gold are dependent upon the background partial pressure employed in the FIM. This latter result plus the result that the images are only stable in ultra-high vacuum (UHV) conditions indicates the need for UHV conditions for the successful imaging of gold surfaces.

* This research was supported by the United States Atomic Energy Commission. Additional support was received from the Advanced Research Projects Agency and the National Science Foundation through the use of the technical facilities of the Materials Science Center at Cornell University.

† John Simon Guggenheim Memorial Foundation Fellow 1972-73.

1. Introduction

An extensive effort has been made in our laboratory over the last few years⁽¹⁻⁵⁾ to obtain stable field ion microscope (FIM) images of well-annealed gold specimens. The first motivation for this effort has been a desire to apply the FIM technique to the question of whether or not a self-interstitial atom (SIA) can undergo uncorrelated long range migration in the Stage I portion⁽⁶⁾ of the isochronal recovery spectrum of irradiated gold. This question concerning gold has been a controversial topic for a number of years [e.g., see Schilling et al.⁽⁶⁾, Venables⁽⁷⁾, Venables and Thomas⁽⁸⁾ and Seeger^(9,10)] and it would be desirable at this point to apply the experimental techniques developed earlier in this laboratory for studying SIA's in tungsten^(11,12,13) and platinum^(14,15) to gold. The second motivation has been a wish to measure directly the relative populations of monovacancies and divacancies in quenched gold⁽¹⁶⁾ specimens. We have already applied the FIM technique to a study of vacancies in quenched platinum^(17,18), and therefore our ability to examine stable FIM images of gold is an important first step towards the problem of examining quenched gold specimens in the FIM. For a recent review of the Cornell FIM work on point defects see Seidman⁽¹⁹⁾.

Our main experimental achievements in the imaging of gold, prior to the present investigation, have been to obtain quasi-stable* images employing a 25% neon-helium gas mixture at specimen temperatures (T_T 's) as low as approximately 16 K, and also quasi-stable pure neon gas images at values of $T_T \gtrsim 27$ K. These quasi-stable neon images exhibited artifact vacancies on all crystallographic planes in the range 1 to 14 at. %^(3,4).

In the present paper we report on an improved experimental technique for obtaining stable† neon images of gold at a T_T as high as 30 K. In addition

* By quasi-stable we mean that the FIM specimen was always field-evaporating very slowly at best image field (BIF).

† By stable we mean that the evaporation rate as measured on a specific (hkl) plane at 21 K was less than 5×10^{-3} atom sec⁻¹ (hkl)⁻¹. The planes employed for these measurements were the {203}, {321}, {315}, {421}, {671} and {731}. The stability of an image is a strong function of the background pressure attained in the FIM prior to admitting the imaging gas. Our experience indicates that the lower the background pressure in the FIM, the greater is the stability of an image.

we provide information concerning the temperature dependence of the field ionization characteristics of gold emitters and also show that our new technique reduces the artifact vacancy concentration to less than 6×10^{-3} at. fr. for all crystallographic planes studied. The results obtained in the present investigation on the stability of gold FIM surfaces and the observed artifact vacancy concentrations demonstrate the need for the use of ultra-high vacuum conditions in the imaging of this relatively soft non-refractory metal.

2. Experimental Techniques

2.1. SPECIMEN PREPARATION

The FIM specimens were prepared from both COMINCO 99.999 and 99.9999 wt. % pure gold wire (0.127 mm diam). The wires were cut into approximately 2.5 cm long segments and air-annealed between 800 and 850°C for approximately 20 min. The end of each wire segment was then sharpened by electropolishing in a solution consisting of equal parts of 15 wt. % HCl and glycerol at 3.0 to 3.8 Vac. The polishing procedure consisted of immersing the segment to a depth of 3 mm in the electrolyte at 3.8 Vac until the diam was reduced to about 0.025 mm. The specimen was then withdrawn 1.5 mm and the tip polished at 3.0 Vac until the tip diam was approximately 0.01 mm. The specimen was then immersed to the original 3 mm depth for the final polishing at 3.0 Vac^(20,21). As a result of observing the final moments of polishing with a 15X binocular it was noted that the tip edge receded up the wire in a series of "mini-dropoffs". A sharp tip with an average radius of curvature ($\bar{r}_T$) of less than 200Å was obtained by breaking the electrical polishing circuit at the instant of a dropoff. To obtain blunter tips a few voltage pulses were applied after the dropoff.

2.2. DEVELOPMENT OF AN END FORM

The development of an end form was performed in a bakeable stainless steel FIM which was equipped with a vacsorb pump, vacion pumps, a titanium

sublimation pump, and an oil diffusion pump (Dow Corning DC705 oil) which was baffled with a Granville-Phillips liquid nitrogen cold trap. Further details regarding the FIM, the liquid helium cryostat, and temperature measurement have been reported previously^(11,22,23).

The development of an end form was carried out in two stages. In the first stage the FIM was baked* to 100°C. When the gauge pressure was less than 5×10^{-9} Torr the specimen was cooled to approximately 55 K. Gauge pressures of 2×10^{-8} Torr air and 3×10^{-5} Torr neon were subsequently admitted dynamically into the FIM via Varian leak valves. The neon was Linde research grade gas (~99.999 vol. % pure). Next the tip voltage (V_T) was increased slowly until both the symmetry of the lattice could be recognized and a few net planes resolved. The value of T_T was then decreased to approximately 28 K in approximately 10 K intervals (V_T was increased at each step). Finally, at 28 K the tip was developed to the point where all but the low field regions (e.g. (111)) were resolved. The low field regions usually displayed considerable pitting due to field etching. The value of V_T was then decreased to zero and the FIM was rebaked. This first stage of the development of an end form usually required about 1 hour. The images obtained in this first stage were remarkably symmetric and they exhibited no streaks or distorted ring structure.

The second stage of the development of an end form began after the FIM had been rebaked and after the pressure was typically 5×10^{-10} Torr, although pressures as high as 3×10^{-9} Torr were used occasionally. The tip was then recooled to 30 K and pure neon was admitted dynamically to a gauge pressure of 3×10^{-5} Torr. The image usually reappeared with a value of $\bar{V}_T$ which was 20 to 50% greater than the value at the end of the first stage of development. This procedure for obtaining totally developed and stable FIM images of gold had a success to failure (i.e., the specimen underwent catastrophic failure prior to being fully imaged) ratio of approximately 40%. It is noted that

* We have also obtained satisfactory specimens employing an unbaked FIM in this first stage and admitting only neon gas into the FIM.

without the first stage of development in a neon-air mixture (or equivalently in an unbaked FIM) we had virtually no success.

2.3. SPECIAL TECHNIQUES

To search for point defects in the bulk of an FIM tip it is necessary to dissect a specimen atom-by-atom and record each stage of the dissection on film. The dissection was accomplished by either dc field evaporation or pulse field evaporation; both techniques were found to be suitable for gold. The pulse field evaporation method is preferred since the image can be photographed at Best Image Voltage (BIV), while the atoms are pulse desorbed between photographs. However, the evaporation voltage (V_E) for gold is only slightly greater than the BIV at 30 K, and thus the dc field evaporation method is also a practical method for gold at this T_T but not at values <25 K.

The FIM images were intensified internally using a Bendix micro-channel plate^(24,25) and a phosphor screen and then recorded on Kodak-4X 35 mm film using an automated⁽²⁶⁾ Automax ciné camera. The exposure time with this arrangement was 0.4 sec and the filming rate was variable continuously down to 1.6 frame sec^{-1} . The analysis of the film was performed on a Vanguard Analyzer which allowed the film to be scanned⁽²⁶⁾ frame by frame or at a speed which was variable continuously from 1 to 24 frame sec^{-1} .

The total ion current (i_t) from the tip was measured indirectly by measuring the total light intensity (I_t) output of the phosphor screen. At constant values of the voltage drops across the micro-channel plate and between the micro-channel plate and the phosphor screen, the whole intensification assembly is a linear amplifier. Hence I_t is directly proportional to i_t . The value of I_t was measured with a Photovolt Model 520M photometer.

3. Experimental Results

3.1. FIELD IONIZATION CHARACTERISTICS

The field ionization characteristics of gold were determined by measuring the characteristic curves (I_t versus V_T) at a series of different T_T 's. The experiments were performed by first field evaporating a gold specimen to a final end form at 21 K. Then for a series of T_T 's the characteristic curves were measured employing an increment of 200 Vdc in V_T at each value of T_T . At each T_T the value of V_T was never allowed to exceed V_E , hence $\bar{r}_T$ remained a constant throughout the experiment. Thus the values of V_T are directly proportional to the average electric field ($\bar{E}_T$) at the tip. At the values of V_E indicated in fig. 1 almost the entire surface was field evaporating uniformly at a very slow rate.

The characteristic curves of gold recorded at 21, 25, 31, 35 and 49K are shown in fig. 1. These curves consist of two distinct regimes.* The low field regime (Regime I) is approximately linear on a log I_t versus V_T plot and the slope of Regime I is temperature dependent. Regime II (the high field regime) of the characteristic curve exhibits maxima and minima on the log I_t versus V_T plot. The effect of increasing V_T at 21 K on the field ionization characteristics of the individual crystallographic planes can be seen from an examination of the eight micrographs shown in fig. 2. These micrographs indicate clearly that the ionization characteristics as a function of V_T (or $\bar{E}_T$) differ greatly among the various {hkl} planes. The characteristic curve recorded at 21 K shown in fig. 1 corresponds to the micrographs of fig. 2. From fig. 1 it can be seen that the working range at 21 K extends from 5.0kV to the V_E of 10.8kV; the BIV is approximately 10.2kV. The working range was taken to extend from the cut-off voltage (V_C) to V_E . The quantity V_C was defined as the value of V_T at which the linear portion of the characteristic curve deviated from a tangent line. See fig. 9

* It is noted that in fig. 1 the total normalized image intensity is plotted on the ordinate, hence the units are dimensionless.

in Chen and Seidman⁽²⁷⁾ for an illustration of the method used to determine V_C . The quantity V_C was found to be a linear function of T_T , and the slope of the straight line was $63V(K)^{-1}$. The values of V_C as function of T_T are given in Table 1 for a specimen with an $\bar{r}_T$ of $\sim 400\text{\AA}$. This value of the slope for gold is a factor of almost 2.4 greater than the value of $26.7V(K)^{-1}$ found for the (111) plane of tungsten⁽²⁷⁾ imaged with helium. Table 2 exhibits the temperature dependence of V_E for a specimen with an $\bar{r}_T$ of $\sim 400\text{\AA}$. The fractional decrease in V_E over this 28 K temperature range is approximately $5 \times 10^{-3}(K)^{-1}$ with respect to the V_E at 21 K. It should be noted that at approximately 30 K the BIV is the same as V_E . Thus these results explain why the neon images recorded by Ast and Seidman⁽³⁾ at a T_T of greater than 30 K were quasi-stable. The fractional increase in V_C over this temperature range was approximately $1.3 \times 10^{-2}(K)^{-1}$ with respect to the V_C at 21 K. The fractional increase in V_C is a factor of approximately 2.6 times greater than the fractional decrease in V_E . Hence the temperature dependence of the width of the working range is controlled by the dependence of V_C on T_T . It is noted that the above definition of a working range is not particularly useful for point defect research, as the atomic contrast and resolution required for this type of investigation is attained usually only between the BIV and V_E . The useful range for point defect research is therefore controlled mainly by the decrease of V_E with T_T rather than the increase of V_C with T_T .

3.2. ARTIFACT VACANCIES

An "artifact vacancy" is defined⁽³⁾ as a dark spot in the FIM image of a net plane which appears in a position normally occupied by a bright spot corresponding to a normal lattice atom in the surface. Hence, in well-annealed specimens of gold the observation of a dark spot within a net plane, as defined above was taken to be an artifact vacancy.

Two different types of artifact vacancies were detected. The first type was one which appeared in the interior of an $\{hkl\}$ plane as it was field

evaporated inwards from its edges. In the process of continuous and uniform field evaporation every atomic site within a net $\{hkl\}$ plane was revealed. Hence, the presence of an artifact vacancy could be detected during this incremental field evaporation process. The second type of artifact vacancy appeared suddenly between photographs and involved the replacement of a normal imaged atom by an artifact vacancy (this is called a created vacancy).

Table 3 lists the results of our experiments, performed at T_T equal to 28 K, on two different well-annealed gold specimens. The identification of the quantities employed in this table is as follows:

- P = number of $\{hkl\}$ planar layers removed
- A = number of atomic sites examined on a specific $\{hkl\}$ plane
- N_v = total number of artifact vacancies observed
- N_v^i = number of created vacancies

The sites examined for artifact vacancies on a given net $\{hkl\}$ plane were those which were enclosed by one outer ring of atoms when the plane was at its maximum size. This counting method avoided including as artifact vacancies missing sites in the outermost ring of atoms which resulted from the geometric order in which atoms were field evaporated.

The values of $\bar{r}_T$ for specimens 1 and 2, as determined by the ring counting method⁽²⁸⁾, were 300Å and 250Å respectively. Specimen 1 was dissected by the dc field evaporation method and specimen 2 by the pulse field evaporation technique. The average concentration of artifact vacancies, for the $\{hkl\}$ planes examined, was 2.3×10^{-3} at.fr. and 2.5×10^{-3} at.fr. for specimens 1 and 2 respectively. The concentrations measured in the present experiments were a factor of approximately 13 to 60 lower than those measured by Schmid and Balluffi⁽⁴⁾. In our study 0.59 and 0.78 of the artifact vacancies were "created vacancies" (see N_v^i/N_v in Table 3) for specimens 1 and 2 respectively, whereas Schmid and Balluffi⁽⁴⁾ found that 0.50 of their artifact vacancies were "created vacancies".

A separate study was made to examine the effects of varying T_T between 28 and 42 K, and the method of field evaporation on the concentration of artifact vacancies. The results of this study are shown in Table 4. It is seen from this table that varying the method of field evaporation and T_T changes the concentration of artifact vacancies by a factor of approximately 2.9 and that all the concentrations were less than 10^{-2} at.fr. Varying T_T from 28 to 42 K increased the artifact vacancy concentration by a factor of 2.6. In Table 4 the only quantity which differs from those employed in Table 3 is A_s and its definition is:

A_s = the total number of sites examined for artifact vacancies.

This number represents a sum over all the $\{hkl\}$ planes examined.

4. Discussion

4.1. FIELD IONIZATION CHARACTERISTICS

The results presented in section 3.1 show, for the first time, that gold has a working range in the sense defined originally by Southon and Brandon⁽²⁹⁾. This finite working range in conjunction with the FIM images demonstrates conclusively that it is possible to obtain completely stable neon images of gold. The strong increase in V_C with increasing T_T in cooperation with a mild decrease in V_E with increasing T_T is responsible for the limited range of temperatures over which the gold FIM specimens are completely stable. The fact that V_C increased as T_T was increased indicates, once again for another metal, that the velocity of the imaging gas atom when it is ionized is controlled by T_T .

A qualitative explanation for the existence of maxima and minima in regime II has already been discussed^(27,30,31,32) in terms of the combined effects of the regional variation of E_T on the FIM tip, the lateral supply of imaging gas atoms on the surface of the tip, and the slowly increasing field dependent radial supply function. The reader is referred to the work of Holscher⁽³¹⁾ and Chen and Seidman⁽²⁷⁾ for a detailed discussion of this problem.

The stability of the FIM surfaces at $T_T \leq 30$ K, in the present study, is attributed to the use of ultra-high vacuum conditions $[(0.5 \text{ to } 3) \times 10^{-9} \text{ Torr}]$ in the second stage of tip development. In our earlier investigations the background pressure in the FIM was always greater than approximately 5×10^{-9} Torr. Presumably at this higher background pressure the residual partial pressures of gases in the FIM react with the surface atoms to form gold-gas molecular complexes that field evaporate at values of the electric field which are less than the evaporation field of gold. Thus it is clear that the attainment of stable images of a soft non-refractory metal like gold requires the use of an ultra-high vacuum FIM. We want to emphasize that the $V_E(\bar{E}_T)$ we specify is for the vacuum conditions present during this study. It may be that with improved vacuum conditions that $V_E(\bar{E}_T)$ will be increased; which will therefore increase both the working range and the T_T at which stable gold images can be obtained.

4.2. ARTIFACT VACANCIES

Ast and Seidman⁽³⁾ have discussed in detail the following four possible mechanisms* for the formation of artifact vacancies in gold surfaces:

- (1) A lowering of the effective free energies of formation and motion of a monovacancy in certain surface planes⁽¹⁶⁾ due to the negative hydrostatic stress associated with the evaporation field.
- (2) The incident imaging gas atom transfers a certain fraction of its polarization** energy $(\frac{1}{2}\alpha E^2)$ to a gold atom on the surface in a manner to promote field evaporation of atoms at an E lower than the normal evaporation field.
- (3) The imaging gas atom or residual impurity atoms in the vacuum system react with a gold atom to form a gold gas molecule which field desorbs at a lower value of E than does a gold atom.

* We exclude the possibility that the artifact vacancies are a result of selective field evaporation of impurity atoms(33,34), because of the low impurity concentration of the specimens employed (see section 2) in comparison with the high concentration of artifact vacancies observed.

** The quantity α is the polarizability of a gas atom.

- (4) In the presence of a certain partial pressure of neon almost every surface atom has adsorbed a neon atom, and the artifact vacancies (dark spots) are due to those atoms which have not adsorbed a neon atom.

It was concluded that either (or both) the second or the third mechanism was responsible for the observed concentrations of artifact vacancies in the neon images of gold. The third mechanism is of particular interest with respect to our new results on artifact vacancies, since this mechanism is based on the experimental fact that metal-gas molecular ions have been observed by Müller et al. (35,36) with the aid of an atom probe FIM. In particular, they found a large number of gas-metal compounds of gold which was field evaporated at 78 K in a background pressure of 5×10^{-8} Torr in the presence of the gases helium, neon and argon at a pressure of 10^{-4} Torr. With the aid of the atom probe FIM they observed the compounds AuHe, AuHe₂, AuHe₃, AuAr, AuAr₂, AuH and AuH₂.

In the present experiment we found that the concentrations of artifact vacancies were a factor of 13 to 60 smaller than the concentrations measured by Schmid and Balluffi (4), at approximately the same T_T , in a background pressure of 5×10^{-8} Torr. Since our background pressures were a factor of approximately 17 to 100 lower than Schmid and Balluffi's pressures, the decrease we observed in the artifact vacancy concentration was approximately proportional to the background pressure in the FIM. This result indicates strongly that the artifact vacancies were formed as a result of a gas-metal interaction, and we are led to conclude that the third mechanism for artifact vacancy formation is probably the dominant one operating in the case of gold. This conclusion, that the third and not the second mechanism is more important in producing artifact vacancies, is further supported by the temperature dependence of the artifact vacancy concentration. For dc field evaporation the value of V_T is approximately equal to V_E . Since V_E ($\bar{E}_T$) decreases with increasing T_T , the

value of $\frac{1}{2}eE^2$ of each neon gas atom also decreases with increasing T_T . This effect, for the second mechanism, should lead to a lower artifact vacancy concentration as T_T is increased. This expectation is contrary to the experimental evidence obtained in both the present research and in the Schmid and Balluffi investigation. On the other hand, the decrease of V_E ($\bar{E}_T$) with increasing T_T decreases the potential barrier for impurity gas atoms to reach the specimen, hence increasing the flux of impurity gas atoms to the specimen. This fact may also explain the increased artifact vacancy concentration employing the pulse field evaporation technique, since in this case V_T is less than V_E except during the voltage pulse. Hence the use of ultra-high vacuum conditions is mandatory for obtaining gold images with a low artifact vacancy concentration.

The expected concentrations of vacancy type defects in quenched gold do not exceed approximately 7×10^{-4} at.fr. (37), hence the present imaging techniques are still not amenable to studying monovacancies in quenched gold. However, since the artifact vacancies are arranged randomly, the probability $[p(n)]$ of finding an artifact vacancy cluster of size n is approximately equal to $(2.5 \times 10^{-3})^n$ at T_T equal to 28 K (see Table 3). Therefore, vacancy defect clusters containing n vacancies (where $n > 1$) can be studied if they occur with a probability of greater than $\sim (2.5 \times 10^{-3})^n$. It is, however, certainly possible that the use of better vacuum conditions and purer neon gas, than employed in the present investigation, could lead to even lower values of the artifact vacancy concentrations.

Acknowledgements

We would like to thank Mr. R. Whitmarsh for technical assistance, Mrs. Karen Pratt for scanning film, and Dr. E. D. Boyes for information regarding his electropolishing solution. One of the authors (D.N.S.) wishes to thank the John Simon Guggenheim Memorial Foundation for support funds.

References

1. D. G. Ast and D. N. Seidman, Appl. Phys. Letters 13 (1968) 348.
2. D. G. Ast and D. N. Seidman, Cornell University Materials Science Center Report #1322 (1970).
3. D. G. Ast and D. N. Seidman, Surface Sci. 28 (1971) 19.
4. T. E. Schmid and R. W. Balluffi, Surface Sci. 28 (1971) 32.
5. D. G. Ast, Ph.D. Thesis, Cornell University (1972).
6. W. Schilling, G. Burger, D. Isebeck and H. Wenzl, in Vacancies and Interstitials in Metals, (North-Holland, Amsterdam, 1970) pp. 255-361.
7. J. A. Venables in Atomic Collision Phenomena in Solids (North-Holland, Amsterdam, 1970) pp. 132-61.
8. J. A. Venables and G. Thomas in Vacancies and Interstitials in Metals (North-Holland, Amsterdam, 1970) pp. 531-55.
9. A. Seeger in Vacancies and Interstitials in Metals (North-Holland, Amsterdam, Amsterdam, 1970) pp. 999-1014.
10. A. Seeger, Radiation Effects 2 (1970) 165.
11. R. M. Scanlan, D. L. Styris and D. N. Seidman, Phil. Mag. 23 (1971) 1439.
12. R. M. Scanlan, D. L. Styris and D. N. Seidman, Phil. Mag. 23 (1971) 1459.
13. L. A. Beavan, R. M. Scanlan and D. N. Seidman, Acta Met. 19 (1971) 1339.
14. P. Pétroff and D. N. Seidman, Appl. Phys. Letters 18 (1971) 518.
15. P. Pétroff and D. N. Seidman, Acta Met. 21 (1973) 323.
16. R. W. Balluffi, K. H. Lie, D. N. Seidman and R. W. Siegel in Vacancies and Interstitials in Metals (North-Holland, Amsterdam, 1970) pp. 125-167.
17. A. S. Berger, D. N. Seidman and R. W. Balluffi, Acta Met. 21 (1973) 123.
18. A. S. Berger, D. N. Seidman and R. W. Balluffi, Acta Met. 21 (1973) 137.
19. D. N. Seidman, J. Physics F: Metals Physics 3 (1973) 393.
20. E. D. Boyes, P. J. Turner and M. J. Southon, 18th Field Emission Symposium, Eindhoven, The Netherlands (1971).
21. E. D. Boyes, Ph.D. Thesis, University of Cambridge (1972).
22. D. N. Seidman, R. M. Scanlan, D. L. Styris and J. W. Bohlen, J. Sci. Instr. 2 (1969) 473.
23. D. N. Seidman and R. M. Scanlan, Phil. Mag. 23 (1971) 1429.
24. P. J. Turner, P. Cartwright, M. J. Southon, A. Van Oostron and B. W. Manley, J. Sci. Instr. 2 (1969) 731.

25. S. S. Brenner and J. T. McKinney, Surface Sci. 23 (1970) 93.
26. R. M. Scanlan, D. L. Styris, D. N. Seidman and D. G. Ast, Cornell University Materials Science Center Report #1159 (1969).
27. Y. C. Chen and D. N. Seidman, Surface Sci. 27 (1971) 231.
28. M. Drechsler and P. Wolff, in: Fourth International Conference on Electron Microscopy (Springer, Berlin, 1969) p. 835.
29. M. J. Southon and D. G. Brandon, Phil. Mag. 8 (1963) 579.
30. E. W. Müller, S. Nakamura, O. Nishikawa and S. B. McLane, J. Appl. Phys. 36 (1965) 2496.
31. A. A. Holscher, Ph.D. Thesis, University of Laiden, The Netherlands (1967).
32. H.A.M. van Bekelen, Surface Sci. 21 (1970) 21.
33. D. G. Brandon, in: Field Ion Microscopy, Eds. J. J. Hren and S. Ranganathan (Plenum Press, New York, 1968) pp. 53-56.
34. E. W. Müller, in: Field Ion Microscopy, Eds. J. J. Hren and S. Ranganathan (Plenum Press, New York, 1968) pp. 88-101.
35. E. W. Müller, S. B. McLane and J. A. Panitz, Surface Sci. 17 (1969) 430.
36. E. W. Müller, S. V. Krishnaswamy and S. B. McLane, Surface Sci. 23 (1970) 112.
37. R. O. Simmons and R. W. Balluffi, Phys. Rev. 125 (1962) 862.

TABLE 1

The Temperature Dependence^(a) of the Cut-off Voltage (V_c)

T_T (K)	21	25	31	36	49
V_c (kV)	5.0	5.3	5.8	6.0	6.8

- (a) For a specimen with an $\bar{r}_T$ of $\sim 400\text{\AA}$. The quantity $\bar{r}_T$ was determined by the ring counting method(28) between the (200) plane and the {204} planes.

TABLE 2

The Temperature Dependence^(a) of the Evaporation Voltage (V_E)

T_T (K)	21	25	31	36	49
V_E (kV)	10.8	10.6	10.3	10.1	9.3

- (a) For a specimen with an $\bar{r}_T$ of $\sim 400\text{\AA}$. The quantity $\bar{r}_T$ was determined by the ring counting method⁽²⁸⁾ between the (200) plane and the (204) planes.

TABLE 3

Artifact Vacancy Concentrations for Various $\{hkl\}$ Planes of Well Annealed Gold Specimens Field Evaporated at 28 K

Plane (a) (hkl)	P (b)	A (c)	A/P (d)	N_V (e)	N'_V (f)	N'_V/N_V (g)	$\frac{N_V}{A} \times 10^3$ (h)
$\{203\}_1$	1,044	10,440	10	38	30	0.79	3.64
$\{203\}_2$	654	4,578	7	3	3	1.00	0.655
$\{321\}_1$	387	5,805	15	31	17	0.55	5.34
$\{321\}_2$	660	7,260	11	21	17	0.81	2.89
$\{315\}_1$	830	23,240	28	33	13	0.39	1.42
$\{315\}_2$	1,116	23,436	21	70	54	0.77	2.99
$\{421\}_1$	2,001	16,008	8	29	19	0.66	1.81
$\{421\}_2$	1,746	8,730	5	20	17	0.85	2.29
$\{671\}_1$	1,637	13,096	8	26	14	0.54	1.99
$\{671\}_2$	764	4,584	6	5	5	1.00	1.09
$\{731\}_1$	--	--	--	--	--	--	--
$\{731\}_2$	190	1,140	6	4	0	0	3.51
Total-1	5,899	68,589		157	93	0.59	2.29
Total-2	5,130	49,728		123	96	0.78	2.47

- (a) Subscripts 1 and 2 indicate Specimens 1 and 2 respectively.
 (b) Planar $\{hkl\}$ layers removed.
 (c) The number of sites examined for artifact vacancies.
 (d) The ratio of sites examined to the number of layers removed.
 (e) The total number of artifact vacancies observed.
 (f) The number of artifact vacancies observed to appear between photographs (created artifact vacancies).
 (g) The ratio of created artifact vacancies to the total number of artifact vacancies.
 (h) The atomic fraction of artifact vacancies.

TABLE 4

Artifact Vacancy Concentrations of Well Annealed Gold Specimens
Field Evaporated at 28, 34 and 42 K by Both the Pulse
Field Evaporation and dc Field Evaporation Techniques

Tip Temperature (K)	Technique of Field Evaporation(a)	A_s (b)	N_v (c)	N_v (d)	$\frac{N_v^*}{N_v}$ (e)	$\frac{N_v^*}{A_s} \times 10^3$ (f)
28.	dc	68,589	157	93	0.59	2.29
28	pulse	4,011	27	13	0.48	6.73
34	dc	45,483	69	40	0.58	1.52
42	dc	23,042	139	87	0.63	6.03

- (a) The term dc refers to the dc field evaporation method and the term pulse refers to pulse field evaporation method.
- (b) The total number of sites examined for artifact vacancies. This number represents a sum over all the {hkl} planes examined (see Table 3 for planes studied).
- (c) Total number of artifact vacancies observed.
- (d) The number of artifact vacancies observed to appear between photographs (created artifact vacancies).
- (e) The ratio of created vacancies to the total number of artifact vacancies.
- (f) The atomic fraction of artifact vacancies.

Figure Captions

Figure 1. The total normalized light intensity (I_t) as a function of the specimen voltage (V_T) at five different specimen temperatures (T_T) between 21 and 49 K. The average radius of curvature ($\bar{r}_T$) of the specimen was a constant value of 400Å. The voltage (V_E) at which uniform field evaporation commenced is indicated by an arrow on each curve.

Figure 2. A series of eight FIM micrographs recorded at 21 K showing the effect of increasing V_T on the image contrast. The value of V_E for this specimen is 10.8kV. The values of V_T for figs. 2a through 2h are as follows: fig. 2a, $V_T = V_E - 5.2\text{kV}$; fig. 2b, $V_T = V_E - 4.0\text{kV}$; fig. 2c, $V_T = V_E - 2.6\text{kV}$; fig. 2d, $V_T = V_E - 2.0\text{kV}$; fig. 2e, $V_T = V_E - 1.6\text{kV}$; fig. 2f, $V_T = V_E - 1.2\text{kV}$; fig. 2g, $V_T = V_E - 0.8\text{kV}$; fig. 2h, $V_T = V_E - 0.4\text{kV}$.

Figure 1.

Figure 2.