Statistics of the atom-by-atom dissection of planes in an atom-probe field-ion microscope: The number of atoms detected per plane

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(Received 18 January 1983; accepted for publication 9 May 1983)

The statistics of the atom-by-atom dissection of planes, in the atom-probe field-ion microscope, have been investigated. Wusten specimens oriented in the [110] direction, with the probe hole over the center of the plane, were slowly pulsed field-evaporated on a plane-by-plane basis, and statistical analyses were made on the number of tungsten atoms detected per plane; 30 separate slow dissection experiments were performed. Observed fluctuations in the number of atoms per plane are used to infer a range of allowable values for the detection efficiency. We find that, in some cases, the number of atoms per plane can be described as following a binomial distribution. From these results detection efficiencies in the range 0.11 to 0.54 were inferred. This range of efficiencies can be understood with the aid of field-ion desorption images. In addition, a value for the detection efficiency of 0.2 was obtained under the assumption that all atoms in the area projected by the probe hole, along the specimen radius, were analyzed. Thus, we find that this geometrical procedure, a first-order approach to the problem, yields a reasonable result. Also, the results of a Monte Carlo simulation of atom-by-atom field evaporation of a large number of planes are presented. The Monte Carlo simulation shows that if a binomial distribution is obtained, the uncertainty in concentrations determined by the atom-probe technique will have only a small component owing to the uncertainty in the number of solvent specimen atoms—this is subject to the caveat that there are no special problems with the field-evaporation behavior of the solvent atoms. Although the statistical analyses were applied to a specific crystallographic plane and position, the methodology is reasonably general and can be applied to other situations.

PACS numbers: 07.80. + x, 61.60. + m

INTRODUCTION

By employing the field-ion microscope (FIM) it is possible to image the crystal structure of a large number of metals with atomic resolution. If, in addition, the time of flight (TOF) of each field-evaporated ion is measured, then it is also possible—employing the pulsed field-evaporation technique—to determine the chemical composition of individual atomic planes. Such studies have been extensively carried out at Cornell using an atom-probe FIM. 1 For example, at Cornell we have studied low-energy (50 to 1500 eV)-implanted 3He and 4He range profiles in tungsten employing this technique. 2-4 Tsong and co-workers have also used this approach to study the segregation of different solute atoms to the surface of FIM specimens. 5-7 Brenner and his colleagues have applied this technique to the study of precipitation. 8-11 Smith and associates 12-14 have put it to use in the study of phase transformations and segregation in steels. And Nordén and Andrén and company have utilized it in the study of segregation and precipitation in steels. 15-17

The Cornell atom probe measures the TOF of an ion using a straight flight tube and in this respect it is similar to the original atom probe invented by Müller et al. 18 The spatial resolution in depth for a composition profile is equal to the interatomic spacing of the crystallographic plane positioned under the probe hole. The atom probe is interfaced to a minicomputer and tens of thousands of events can be recorded in a single afternoon. Since the mass-to-charge (m/n) ratio of every event is obtained, one can determine concentration versus depth profiles for any observed species using this method.

Of concern in this paper are the statistics of the atomic collection process and the specimen surface area probed in such experiments. This area appears to be accessible from the geometry of the atom probe, in which case one can compute a detection efficiency by projecting the probe hole onto the specimen's surface. (The question of detection efficiency was first addressed by Brenner and McKinney 19 and more recently by Krishnaswamy et al. 20 and Nishikawa et al. 21) But, in so doing one must assume that the acceptance angle of the probe hole is determined by geometrical factors associated with the atom probe. However, a second means of obtaining detection efficiencies—which is independent of assumptions about the area probed—is available via experimentally obtained distributions of the number of atoms detected per plane.

We have collected a large body of data for the number of tungsten field evaporation events detected, that is, the number of tungsten ions detected per [110] plane, and we have used these data to infer values of the detection efficiency.
The results do not disagree with the detection efficiency obtained via a geometrical projection. We find that, in some cases, the number of atoms per plane can be described as following a binomial distribution. Finally, we have also simulated the detection of ions, employing the Monte Carlo technique, and we conclude that the uncertainty in values of the number of solvent atoms—that should be used for concentration determinations—is quite small if a binomial distribution for the number of atoms detected per plane is obtained. This is subject to the caveat that there are no special problems with the field-evaporation behavior of the solvent atoms.

I. APPARATUS AND TECHNIQUE

The basic layout of the atom probe is illustrated in Fig. 1. Tungsten specimens with a [110] orientation were inserted into the atom probe, after electrochemical polishing, and were developed via field evaporation to endforms having radii of $\sim 250 \text{ Å}$; the endforms exhibited the usual regular field-ionization image. The center of the [110] pole was subsequently positioned directly under the middle of the probe hole with the aid of the field-ionization image. After the helium-imaging gas was pumped away—and ultrahigh vacuum was obtained ($\sim 5 \times 10^{-10} \text{ Torr}$)—the field-evaporation process was continued by manually increasing the dc potential on the specimen and the proportional pulse and focusing lens voltages. All of the atom-probe data presented here were obtained using a pulse voltage equal to 10% of the steady-state dc voltage. The voltage on the focusing lens was held at a fixed and optimized fraction of the specimen voltage. All pulse field-evaporated ions with trajectories which passed through the grounded metal probe tube, metal focusing lens, and grounded mirror sleeve—and registered by the Chevron ion detector—were then recorded by the minicomputer. The pulse frequency was 60 Hz and a typical field-evaporation rate was 40 pulses per event. Results were obtained for specimen temperatures in the range 20–94 K (see Table I).

After a specimen was dissected, plots of all ions with values of $m/n$ between 40 and 70 amu (where $m$ is the mass of the ion and $n$ its charge state) were plotted versus the total number of pulses applied from the start of a field-evaporation sequence in ultrahigh vacuum. The range 40 to 70 amu encompasses all five isotopes of the $3^+ \text{ and } 4^+ \text{ charge states.}$

<table>
<thead>
<tr>
<th>Number of planes</th>
<th>Temperature of the specimen (K)</th>
<th>Average number of atoms per plane, $(\langle N \rangle)$</th>
<th>Experimental standard deviation $S$</th>
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<td>34</td>
<td>41</td>
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<td>32</td>
<td>31</td>
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<td>25</td>
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<tr>
<td>Y-13</td>
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</tr>
</tbody>
</table>

*The names of the runs are indicated to the left of each value for the runs discussed in this paper.*
states in which tungsten field evaporates. A portion (1) of such a plot is exhibited in Fig. 2(a). The steplike behavior exhibited in Fig. 2(a) can be understood on the basis of the gradual collapse of (110) planes, during pulsed field-evaporation, as depicted in Fig. 2(b). The passing of such a step thus indicates the complete evaporation of a single plane.

It is not difficult employing an audio ratemeter and the steplike field-evaporation behavior exhibited in Fig. 2(a) to optimally align a specimen with respect to both the probe hole and the Chevron ion detector. In particular, the steplike field-evaporation behavior is very sensitive to the accuracy of the alignment procedure. All the results presented in this paper are for the situation where the specimen, probe hole, and Chevron ion detector were optimally aligned.

II. ANALYSES

A. Detection efficiency obtained by geometrically projecting the probe hole

A first-order approach to the problem is to calculate the area radially projected by the probe hole onto the surface of a spherical specimen and then to divide the number of atoms in this area by the average number of atoms detected to obtain an efficiency. Such a projection is shown in Fig. 3. The projection, on the specimen's surface, of the probe hole radius \( r_o \) is given by the relation

\[
r_o = R_o \frac{d}{2} R,
\]

where \( d \) is the diameter of the probe hole and \( R_o \) is the distance between the limiting aperture and the specimen. The number of tungsten ions detected per (110) plane \( \langle N \rangle \) is then given by the relation

\[
N = \eta \pi \sqrt{2} \left( R_o \frac{d}{2} R \right)^2 / 4 \left( a_o R_o \right),
\]

where we have represented the entire detection efficiency by the single quantity \( \eta \). \( a_o \) is the lattice parameter of tungsten \( (3.16 \text{ Å}) \). Equation (2) is based on the assumption that the acceptance angle of the probe hole is determined by the physical dimensions of the atom probe. Electrostatic limitations on the acceptance angle are therefore incorporated in the parameter \( \eta \). Equation (2) can be used to calculate values of \( \eta \) from data obtained in the atom probe.

B. The detection efficiency obtained from fluctuations in the number of atoms per plane

A second means of inferring \( \eta \) from the data is available via values of the experimental standard deviation \( (S) \) of the distribution of \( N \) about \( \langle N \rangle \), where \( \langle N \rangle \) is the experimental least-squares linear fit value for the data from each dissection run. In Sec. III we present a plot of \( \langle N \rangle \) vs \( S \) and then in Sec. IV we compare these data with the model developed below.

Let us now consider a sequence of successive field evaporation events as a single (110) plane is completely pulsed field-evaporated. If single-atom statistics apply, that is, if the detection probabilities of these events are independent and given by \( \eta \) (where \( \eta \) is less than one), then for the \( M \) atoms "covered" by the probe hole the probability distribution describing the number of observed events per plane \( \langle N \rangle \) is a binomial distribution:

\[
P(N) = \frac{M!}{N!(M-N)!} \eta^N (1-\eta)^{M-N}.
\]

The mean value \( (\phi) \) and dispersion \( (\sigma) \) of the binomial distribution are given by

\[
\phi = \eta M
\]

and

\[
\sigma^2 = \eta (1-\eta) M.
\]

These relations can be combined to yield

\[
\phi = \sigma^2 / (1-\eta).
\]
We note that this latter expression is independent of $M$; that is, it is independent of any assumptions regarding the area covered by the probe hole. And since $\eta$ is less than 1, all the data should lie to the left of the curve for $\phi = \sigma^2$ on a plot of $\phi$ vs $\sigma$.

III. RESULTS

The data exhibited in Fig. 4 typify the procedure used to arrive at distributions in the number of tungsten ions detected per (110) plane field evaporated ($N$). We have previously denoted this same quantity $N_m$. In general, the value of $N$ increases as successive planes are field evaporated. This behavior can be simply explained as a dependence on the specimen radius ($R_s$). Distributions in $N$ were obtained using a least-squares fitting procedure to the data from each single dissection run. These linear fits yielded values of $\langle N \rangle$ and then $\langle N \rangle$. The value of $\langle N \rangle$ for each of six successive runs (runs Y-8 through Y-13) on the same specimen are plotted vs $R_s$ and also the applied voltage $V$, as exhibited in Fig. 5. The values of $R_s$ were calculated employing the standard equation $R_s = V/\kappa E$, where $\kappa$ is a geometric factor and $E$ is the electric field; we took $E = 4.4$ V $\text{Å}^{-1}$ and $\kappa = 5$. Figure 5 clearly demonstrates, as expected, that $\langle N \rangle$ is proportional to $R_s^2$; also see Fig. 1 of an article by Moore and Ranganathan. $^{24}$ Although $R_s$, and the number of planes field evaporated are linearly related—via a proportionality factor involving the shank angle—the number of (110) planes field evaporated in a run was never large enough to show curvature in plots of the form exhibited in Fig. 4. Thus, a linear fit is sufficient. The distribution of $N$ about $\langle N \rangle$ for the data shown in Fig. 4 is exhibited in Fig. 6. From such distributions the value of $S$ was obtained. Treating the data in this way removed the effect of an increasing $R_s$ on the measured values of $S$.

The observed values of $\langle N \rangle$ were also found to depend systematically on the position of the channel electron multiplier array (CEMA) with respect to the FIM specimen. This dependence is exhibited in Fig. 7; these data were obtained for a specimen having a shank angle of only $6^\circ$. Thus, the $\langle N \rangle$ values exhibit only a weak dependence on $R_s$. Runs Y-1, Y-2, Y-3, and Y-4 were obtained with successively increasing specimen to CEMA separations and runs Y-5, Y-6, and Y-7 followed with successively decreasing separations. See Sec. IV for a definition of the $R_s^*$ values used in Fig. 7.

Data for a total of 30 separate probing runs, obtained from seven specimens, were analyzed to yield values of $S$ (Table I). Values of the overall average number of ions detected per plane ($\langle N \rangle$) — $\langle N \rangle$ is the average of the $\langle N \rangle$ values obtained in a single run—are plotted as a function of $S$ in Fig. 8. The actual experimental points are indicated by the solid black circles. Table I lists values of $\langle N \rangle$ for 30 and $S$ for 24 different experiments, respectively.

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**Fig. 4.** Number of tungsten ions detected per (110) plane vs the number of planes field evaporated for run M-1. The solid line gives $\langle N \rangle$ values and is a least-squares linear fit to the data points.

**Fig. 5.** $\langle N \rangle$ values vs specimen radius ($R_s$). The $\langle N \rangle$ values and applied voltage ($V$) are for six successive probing runs (runs Y-8 through Y-13). The solid curve is a parabola.

**Fig. 6.** Histogram of the number of planes having $(N - \langle N \rangle)$ values falling within the indicated ranges. A bin width of five ions was arbitrarily selected. The curves were calculated employing the extreme values of $\eta$, $\phi$, and $\sigma$ deduced in Sec. IV B.
IV. DISCUSSION

A. Detection efficiency obtained by geometrically projecting the probe hole

We used Eq. (2) to calculate values of $\eta$ from data obtained in the atom probe. Since only approximate values of $R$ could be obtained directly, this expression was instead fitted to the data in Fig. 7. The fit yields a value of 2.81 cm for $R$ at a CEMA position of $R^* = 5.33$ cm, where $R^*$ is the distance between the CEMA and a fiducial marker outside the atom probe. The average of the two $\langle N \rangle$ values for runs Y-1 and Y-7 is 98. These data were obtained with $d = 5.44$ mm and $R_0 = 360$ Å. A value of $\eta$ equal to 0.2 is obtained via Eq. (2). This is less than the geometric efficiency (the open area of the Chevron detector) of the Chevron detector ($\sim 0.58^{25}$) and indicates that other physical factors are significant. We conclude that this first-order approach to the efficiency problem yields a reasonable result.

B. The detection efficiency obtained from fluctuations in the number of atoms per plane

In Fig. 8 we have also plotted curves of $\phi$ vs $\sigma$ employing Eq. (5) to compare with the experimental plot of the $\langle N \rangle$ vs $S$ data. The result of this figure is that most of the data lie to the right of the curve for $\phi = \sigma^2$ (i.e., $\eta = 0$). Several factors could have caused this. Halting field evaporation due to the mechanical relaxation of the specimen at irregular intervals may have possibly been misinterpreted as the passing of (110) planes in some cases. Also, a slight misalignment of the probe hole with respect to the (110) pole made the steps less distinguishable in some cases than in others. However, we have found that some results for very clearly defined steps do lie to the right of the curve $\phi = \sigma^2$. A physical effect which could have caused this is the evaporation of atoms in groups. In that case, single-atom statistics would no longer apply and the distributions would be broadened. This is presently considered a possible explanation. In any case, the effect appears erratically in the present data. We were unable to correlate it with the controllable experimental parameters.\textsuperscript{26}

The variation in $\eta$ can best be understood with the aid of the (110) tungsten field-desorption images exhibited in Fig. 9. These $W^{3+}$ field-desorption images were taken at U.S. Steel at a specimen temperature of 80 K and a tip-to-screen distance of approximately 12 cm. Figure 9(a) is the result of field-desorbing 0.1 monolayers—note that the ledges of the (110) plane are the main sources of ions which produced this image. After the field desorption of 0.5 monolayers (Fig.
one can still discern the (110) ledges and terraces. At
the point where 1.0 monolayers have been field desorbed
[Fig. 9(c)] the two-dimensional spatial array of image points
is more random—however, note the black region in the cen-
ter of the topmost (110) plane. This black region persists even
after 5.0 monolayers had been field desorbed [Fig. 9(d)].
The implication is that desorption of atoms from the (110) planes
must involve the lateral outward motion of the atoms prior
to their being desorbed as ions. These images show that the
absolute value of $\eta$ is determined by the areal fraction of the
(110) plane covered by the probe hole. The limiting value of $\eta$
should be zero; this is obtained when the probe hole covers
the completely black area.

A nonbinomial distribution would be obtained if $\eta$ var-
died during the course of the dissection of the (110) plane.
That it is possible to have a variation in $\eta$ can be understood
with the aid of Fig. 9.

A complete description of the detection process would
entail a number of other factors. In order for a field-evapor-
ated ion to be detected the following are required: (1) its
trajectory must pass through the grounded metal probe tube
at the center of the CEMA and screen; (2) it must be properly
focused onto the Chevron ion detector once it emerges from
the probe tube; and finally (3) it must be detected by the
Chevron ion detector. A complete treatment of the detection
process requires that these factors be described individually;
that is, $\eta_1$ is given by the product $\eta_1 \eta_2 \eta_3$, where $\eta_1$ is
the efficiency with which field-evaporated ions pass through
the probe hole, $\eta_2$ is the efficiency with which they are focused
onto the detector, and $\eta_3$ is the efficiency of the Chevron
detector and subsequent electronics. Furthermore, detection
efficiencies depend, as shown in Fig. 9, on the detailed me-
chanisms of field evaporation, which, in turn, can be a
function of the crystallographic plane. There is also a
distribution of velocities parallel to the specimen’s surface of
field-evaporated ions once they are either thermally excited
over and/or through the Schottky hump. The resolution
of the imaging atom probe has been calculated by Pan-
itz, assuming that only thermal contributions to lateral veloci-

ties of field-evaporated ions are important. Only an
insignificant influence on the efficiency would result in the
latter case. Analyses attempting to separate these factors are
not justified based on the present data.

The approach discussed in this section was only applied
to the specific case of the center of the (110) plane of tung-

sen. However, the methodology is reasonably general and
can be applied to other crystallographic positions and/or
planes.

C. Monte Carlo simulation of the detection process

In addition, the influence of a limited number of obser-
vations has to be accounted for; that is, $S$ values should be
compared to $\sigma$ values only in the limit of a large number
of field-evaporated planes. The data in Fig. 8 were obtained
from probing runs of less than 100 planes. To investigate the
influence of a finite number of total planes on $S$, we have
simulated the detection process via a Monte Carlo pro-
dure. The field evaporation of a plane was simulated by tak-
ing a probability of 0.20 for the detection of an event for 500
events. This procedure, performed on a computer, yielded a
value of $N^*$ for the number of detected events, a quantity
analogous to $N$. Repeating this a number of times to simulate
the collapse of a number of planes yielded a set of $N^*$ values
from which values of $\bar{N}^*$ and $S^*$ were obtained for the aver-
age and standard deviation. These are analogous to the
quantities $\langle N \rangle$ and $S$. Since an increasing specimen radius
was not modeled, the linear fitting procedure used to obtain
$\langle N \rangle$ values was not needed for the Monte Carlo case. By
repeating this entire procedure 100 times we obtained a set of
100 $\bar{N}^*$ and $S^*$ values. From these the quantities $\bar{N}^*$, the
average of the $N^*$ values; $\bar{S}^*$, the standard deviation;
$\bar{S}$, the average of the $S^*$ values; and $\Delta (\bar{S}^*)$, the standard
deviation, were obtained. These have no experimental ana-
logs. They are exhibited in Fig. 10 for different total numbers
of planes field evaporated.

The values of $\Delta (S^*)$ was not found to be a sensitive function
of the total number of planes field evaporated if the number exceeded 20 [see Fig. 10(b)]. It is also noted that
$\Delta (S^*)$ is unity for 44 planes field evaporated; during run M-1
a total of 44 planes were field evaporated. This latter value of
$\Delta (S^*)$ was used as the half-width of the error box in Fig. 8.
Incorporating this into our deduction of $\eta$, we conclude that
values of $\eta$ lie in the range 0.11–0.54. We note that a some-
what circular argument was used to deduce these extreme
values, since a value of 0.20 was assumed for $\eta$ in the Monte
Carlo simulations. However, we feel that a more rigorous
analysis is not justified based on the present data. Figure 6
exhibits calculated distribution curves for the extreme values
of $\eta$.

**Fig. 9.** Four field-ion desorption images of tungsten formed with $W^{++}$ ions. The
tip-to-screen distance was ~12 cm and the specimen temperature was
80 K. The specimen was oriented such that the image is centered on the
(110) plane. (a) is for 0.1 monolayers field desorbed; (b) is for 0.5 monolayers;
c) is for 1.0 monolayers; and (d) is for 5.0 monolayers.
ACKNOWLEDGMENTS

We wish to thank R. Whitmarsh for enthusiastic technical assistance and Dr. A. Wagner for useful comments on the manuscript. This research was supported by the U.S. Department of Energy under Contract No. DE-AC02-76ER93158. Additional support was received from the National Science Foundation through the use of the technical facilities of the Materials Science Center of Cornell University.

LIST OF SYMBOLS

\(d\) = diameter of probe hole
\(\Delta(N^*)\) = standard deviation in \(\langle N^* \rangle\)
\(\Delta(S^*)\) = Monte Carlo simulation result for the standard deviation in \(S^*\)
\(\eta\) = Chevron detector efficiency
\(M\) = number of tungsten atoms covered by probe hole
\(N\) = number of tungsten ions detected for complete collapse of a single (110) plane
\(N^*\) = Monte Carlo simulation result for the number of atoms detected
\(\overline{N}^*\) = Monte Carlo simulation result for the average number of atoms per plane. This corresponds to the experimental quantity \(\langle N \rangle\).
\(\langle N \rangle\) = average of the \(100 \, N^*\) values generated via the Monte Carlo simulation
\(\overline{N}\) = experimental least-squares linear fit value
\(\overline{N}\) = average of the \(\langle N \rangle\) values, i.e., the experimental average number of atoms per plane
\(\phi\) = mean value of the binomial distribution
\(R\) = distance between limiting aperture and the specimen
\(r_e\) = CEMA position with respect to an arbitrary fiducial mark on the outside of the atom probe
\(S\) = experimental standard deviation
\(S^*\) = Monte Carlo simulation result for the standard deviation in \(N^*\)
\(\overline{S}^*\) = average of the \(100 \, S^*\) values generated via the Monte Carlo simulation
\(\sigma\) = dispersion of the binomial distribution

Nevertheless, the bound at small \(S\) appears as a quite discernible characteristic. We have used the data for run M-1 (see Fig. 8) to infer a range of values of \(\eta\) since this data point is the extreme case; that is, it lies farthest to the left of the curve for \(\phi = \sigma^2\). This data point is enclosed in an error box whose height is equal to the range of \(\langle N \rangle\) values obtained by the least-squares fitting procedure. Values of \(\eta\) deduced from this data point must properly incorporate this range of \(\langle N \rangle\) values, since a single value of \(S\) was obtained for the entire probing run.

We note that \(\Delta(N^*)\) is also not a sensitive function of the number of planes evaporated beyond \(\sim 20\) planes. For more than \(\sim 20\) planes it is only one to two atoms [Fig. 10(a)]. This is a significant result since it is this quantity that must be used in determining the uncertainty of concentrations obtained via the dissection method.

We conclude that for the present conditions an uncertainty in the number of solvent atoms of only one to two atoms needs to be incorporated into error propagation calculations for concentrations obtained using the atom-probe technique, if a binomial distribution in the number of atoms per plane is found to apply. This statement assumes that there are no special problems with the field-evaporation behavior of the solvent atoms.


6 J. T. Tsong, Y. S. Ng, and S. B. McLane, Jr., Chem. Phys. 73, 1464 (1980).


26 In the atom-probe analysis of an Fe–3 wt. % Si alloy Miller and Smith [Ref. 27] found anomalous results for data taken from the center of the (110) plane; in particular, the overall number of ion counts they detected was rather low for this region of the surface. Also, Nishikawa et al. [Ref. 21] measured an $\gamma$ value of 0.1 for the center of the (110) plane of tungsten as opposed to 0.85 for the ledge of the (110) plane.
