20th
Field Emission Symposium
August 20-23, 1973

The Pennsylvania State University, University Park, Pa.

Sponsored by the Department of Physics
and the National Science Foundation
Preface

We wish to extend our cordial welcome to all the participants of the 20th Field Emission Symposium. Once you have overcome the difficulty of getting here, we hope your stay at the Pennsylvania State University will be thoroughly enjoyable—as well as scientifically profitable.

The Continuing Education Conference Center has been helpful in efficiently organizing administrative aspects, and the Department of Physics has given us its special support. The participation of several colleagues from abroad and a number of graduate students has been assisted by the Solid State and Low Temperature Physics Program of the National Science Foundation under Grant GH-39526. We are grateful that this support has again ensured the international scope of our exchange of experiences and ideas. Much of the advances in our field can be directly traced to the mutual give and take in the papers and discussions of past symposia, and we hope the 20th Field Emission Symposium will be equally productive.

Erwin W. Müller
T. T. Tsong
Organizing Committee
LIST OF PREVIOUS FIELD EMISSION SYMPOSIA

1. 1952  McMinnville, Oregon  W. P. Dyke
2. 1954  Pittsburgh, Pennsylvania  M. Wachtel
3. 1956  Notre Dame, Indiana  J. A. Becker
4. 1957  University Park, Pennsylvania  E. W. Müller
5. 1958  Chicago, Illinois  R. Gomer
6. 1959  Washington, D. C.  L. Marton
7. 1960  McMinnville, Oregon  W. P. Dyke
          F. Charbonnier
8. 1961  Williamstown, Massachusetts  G. Ehrlich
9. 1962  Notre Dame, Indiana  E. A. Coomes
10. 1963  Berea, Ohio  T. George
11. 1964  Cambridge, England  D. G. Brandon
          M. J. Southon
12. 1965  University Park, Pennsylvania  E. W. Müller
14. 1967  NBS Gaithersburg, Maryland  L. Marton
15. 1968  Bonn, Germany  H. D. Beckey
          J. T. McKinney
17. 1970  New Haven, Connecticut  F. Hutchinson
18. 1971  Eindhoven, The Netherlands  A. van Oostrom
19. 1972  Urbana-Champaign, Illinois  G. Ehrlich
PROGRAM OF THE 20TH FIELD EMISSION SYMPOSIUM

Monday, August 20, 1973

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          Dean of College of Science

9:15 am  Opening Remarks:  Erwin W. Müller

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11:00 am  T. E. Feuchtwang:  A New Many-Body Theory for
          Field Emission
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          Ionization Caused by Field Adsorbed Atoms in
          Field Ion Microscopy.
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2:30 pm  Richard G. Forbes:  In Advocacy of Using
          Molecular Wave Mechanics in Field Ion Theory.
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          Page  14

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          Distribution in Field Ionization using the
          Magnetic Atom-Probe.
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<td>Decomposition of Nitric Oxide on Ruthenium</td>
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<td>9:30 am</td>
<td>Novak Drundarov</td>
<td>Obtaining of Tantalum Micromonocrystals in [111] Orientation and Some Effects of Surface Selfdiffusion.</td>
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Chairman: M. J. Southon

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3:30 pm  Coffee Break

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Thursday, August 23, 1973

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SPECTROSCOPY AND ATOM-PROBE

Chairman: A. J. Melmed

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9:30 am  G. K. L. Cranstoun and M. F. King: The Reaction Between Ethylene and Tungsten Studied in the Field-Ion Microscope.

10:00 am  W. A. Schmidt, O. Frank and J. H. Block: Mass Spectrometric Analysis of Field Desorbed Species Formed at a Silver Surface in the Presence of O$_2$, H$_2$O, H$_2$, CO, NH$_3$, CH$_4$, N$_2$, Kr and Xe.

10:30 am  Coffee Break

11:00 am  S. V. Krishnaswamy and E. W. Müller: Premature Field Evaporation in the Atom-Probe

11:30 am  R. J. Lewis and G. D. Smith: Pulse Shape and Timing Errors in FIM Atom Probe.

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4:30 pm Discussions on Atom-Probe Technique.
THE INVITED PAPER

Electronic States at Metal Surfaces

David Penn
National Bureau of Standards
Washington, D. C.
A New Many Body Theory for Field Emission

T. E. Feuchtwang
Department of Physics
The Pennsylvania State University
University Park, Pennsylvania 16802

Plummer's\textsuperscript{1} experimental observation of resonant effects in the high field emission of electrons has motivated a reexamination of this phenomena in terms of current formulations of electron tunneling across barriers. This work, exemplified by Gadzuk's\textsuperscript{2} analysis, is based on the transfer Hamiltonian formalism. The serious conceptual difficulties of the current transfer Hamiltonian formalism have been amply documented,\textsuperscript{3} these difficulties have motivated an extensive search for alternative theories. Recently Caroli et al.\textsuperscript{4} and Feuchtwang\textsuperscript{5} have adapted a Green's function formalism for non-equilibrium processes, due to Keldysh\textsuperscript{6} to develop a new many body theory of tunneling. The theory has the unexpected attractive feature of providing valuable insight into the single particle aspects of tunneling. The adaptation of the new theory to the analysis of resonant and inelastic field emission will be discussed.

\textsuperscript{5}T. E. Feuchtwang, submitted to Phys. Rev.
\textsuperscript{6}L. V. Keldysh, Soviet Physics JETP \textbf{20}, 1018 (1965).
Enhancement of Ionization Caused by Field Adsorbed Atoms in Field Ion Microscopy

D. A. Nolan and R. M. Herman
Physics Department, The Pennsylvania State University
University Park, Pennsylvania 16802

In a previous article\(^1\) we have shown that the increase in He ionization rate observed\(^2\) when inert atoms are field adsorbed\(^3\) above metallic surface atoms results from exchange effects. Theoretically, these effects are realized through the antisymmetrization of the initial and final electronic states appearing in the time-dependent perturbation theory for the ionization process. Using Hartree-Fock orbitals for both the adsorbate and the He atom or ion (undistorted by the presence of the intense electric field or atomic interactions), the enhancement factors for the adsorbed Ne and He range from 90 to 30 and 3, to 5 respectively as field strength is varied from 3 to 5 V/Å at the tip surface. The ratio of enhancement factors for adsorbed Ne vs. adsorbed He shows reasonable agreement with Rendulic's\(^4\) observations (within a factor of 10) although the field dependence of this ratio is not satisfactory.

The latter discrepancy has been resolved through the inclusion of field induced distortions of the atomic orbitals. This is especially important at low field strengths, since the critical distance for ionization is then large, and the overlap matrix elements are substantially altered through the field distortions of distant parts of the atomic orbitals. The inclusion of field-distortions is accomplished through the use of perturbation theory for regions near the nuclei and the WKB approximation in parabolic coordinates for the more distant parts of the orbitals, matching magnitudes and derivatives of the orbitals at various points on the boundaries between the near and distant regions. The calculated enhancement factors for both adsorbed Ne and adsorbed He are significantly reduced in this approximation (Ne, 2-10; He 1-0.4). The ratio of enhancement factors remains roughly unchanged in overall magnitude, although its field dependence is decidedly improved in this approximation.

\(^1\)D. A. Nolan and R. M. Herman, Phys. Rev. B, to be published.
In Advocacy of Using Molecular Wave Mechanics in Field Ion Theory

Richard G. Forbes
University of Aston in Birmingham, Department of Physics

There now seems recognition that gas distribution effects may significantly influence field-ion image contrast. The question arises: How best to describe the imaging-gas behaviour and the actual field-ionizing transition? The two basic possibilities are: A quasi-classical approach in which one takes the atom as a point-behaving object, the transition as an electron transition, and performs an integral over space; and a fully wave-mechanical approach in which one takes the atom as a wave-behaving object, the transition as a molecular (vibronic) transition, and performs an integration over a set of vibrational states. Bohr's Complimentary Principle seems to imply that one or other of these approaches is to be preferred.

There are differences in the theoretical predictions; but these are almost impossible to make usefully precise, because of the general messiness of the field-ion situation. However, epistemological arguments seem to favour the fully wave-mechanical approach:

1) At present the normal practice is to treat the atom quasi-classically when calculating a transition rate, but to invoke Heisenberg's uncertainty principle when discussing image spot size. Greater consistency is achieved by treating the gas atom as in all aspects a wave-behaving object.

2) Using a quasi-classical formulation, and adopting a wave-packet method to describe the electron transition, the motion of the gas atom means that the electron wave-functions at different times cannot be linked by the elementary time-dependent Schrödinger equation. A fully wave-mechanical approach circumvents this problem.

3) In a quasi-classical approach it should be meaningful to suppose that the gas atom has an exact path. But the possibility of electron transition raises major conceptual difficulties if one attempts to define the path. This problem does not arise in a fully wave-mechanical approach.

4) The fully wave-mechanical approach, with its usage of occupation numbers rather than number densities
(i.e. gas concentrations) is perhaps better suited to further theoretical development.

5) The fully wave-mechanical approach is conceptually related to modern treatments of chemical reactions and radiations and radiationless transitions.

If the trust of these arguments were accepted, it would follow that the parameter $P_e$ (the electron transition rate) much used in the past field-ion image contrast would, from a strict conceptual viewpoint, not operationally exist as a parameter of physics.

Irrespective of the acceptance or not of the above arguments, it does seem that it would be useful to adopt a rather more chemical approach to the discussion of ionization and imaging theory.

An Alternative Approach to Field Evaporation Theory

Richard G. Forbes
University of Aston in Birmingham, Dept. of Physics

The recent attempt by Page and Ralph,\(^1\) to estimate the number of surface vacancies to be expected as a result of field-induced artifact formation, may be put on a better formal basis and applied to the general problem of field evaporation theory.

The rate-constant for field evaporation, \(k_n\), is often written in the Arrhenius form: \(k_n = A_n \exp(-Q_n/kT)\). By making an appropriate Taylor expansion of the right-hand side, and using an approximation similar to that of Page and Ralph, one obtains the formulas:

\[
S = \frac{\partial \ln k_n}{\partial \ln F} = \mu/kT \tag{1}
\]

\[
\mu = (F_e^2/4\pi) \cdot \Omega \tag{2}
\]

where \(F_e\) is the evaporation field and \(\Omega\) is the atomic volume. \(\mu\) is a convenient parameter for comparing theoretical and experimental results. For Tungsten, values derived (in eV) from the equations and measured\(^2-4\) values of \(S\) are:

<table>
<thead>
<tr>
<th>Experimental</th>
<th>(Brandon(^2))</th>
<th>2.0, 2.0</th>
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<tr>
<td></td>
<td>(Taylor(^3))</td>
<td>2.6, 2.3, 2.0</td>
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<tr>
<td></td>
<td>(Tsong(^4))</td>
<td>1.6, 1.2, 1.1</td>
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| Theoretical: | (eq. 2) | 2.8 |

The experimental results can also be matched by the detailed theoretical models of field evaporation\(^5\). However, the atomic volume approximation seems to work rather better than might be expected, and this leads to speculation about the possible existence of alternative models for the field evaporation process.

\(^2\)D. G. Brandon, Phil. Mag., 14 (1966).
Field Ion Microscope Image at 4.2 K

Shogo Nakamura and Toshiyuki Adachi
The Institute of Scientific and Industrial Research,
Osaka University, Suita, Osaka 565

Recently, it has been found that the field-adsorption of imaging gas on the specimen surface will play important roles for the field-ionization in the field-ion microscope.\(^1,^2\) Different features in the FIM image would be predicted\(^3\) at the liquid helium temperature since the imaging gas will be condensed on the surface of the tip.

The resolution and average brightness at the best image condition were improved compared with liquid hydrogen temperature. Two maxima in the characteristic curves of the image brightness versus the image voltage were observed on the \(\{112\}\) \(\{001\}\) and \(\{111\}\) planes of W tip at the liquid helium temperature.

When the applied voltage was lowered below the best image voltage, on the \(\{100\}\) zone, single or multiple rows of the bright atom spot at the ledge of \(\{011\}\) net planes disappeared and then the atom spots on the \(\{011\}\) net plane edge got bright.

The brightness of the atom spots at the \(\{112\}\) plane edge also decreased when the applied voltage was lowered. Then the atom spots on the \(\{112\}\) plane edge got bright. The cause of unexpected change of the atom spots will be discussed.

The double step at the \(\{112\}\) plane edges were never observed after field evaporation at the liquid helium temperature. However the disturbed structure around \(\{111\}\) planes of Mo were always observed.

The details of construction and the operational characteristics of the liquid helium cooled FIM are presented.

\(^1\)E. W. Müller, S. V. Krishnaswamy and S. B. McLane: Surface Sci. 23 (1970) 112.


\(^3\)Y. C. Chen and D. N. Seidman: Surface Sci. 27 (1971) 231.
Energy Distribution in Field Ionization
Using the Magnetic Atom-Probe

Erwin W. Müller and Toshio Sakurai
Physics Department, The Pennsylvania State University
University Park, Pennsylvania 16802

A new UHV magnetic atom-probe has been designed\(^1\) which provides a movable tip, image display on a channel plate-screen assembly with a probe hole, a 60° focusing magnetic sector field and the display of the line spectrum on a single or chevron type channel plate-screen assembly. With the latter scheme true atom-probe performance, that is potentially single-atom sensitivity, is achieved. By retardation, ion energies as low as 500 eV may be usefully employed, permitting mass resolutions M/AM >1000. For field desorption and field evaporation experiments, the magnetic atom-probe is particularly suited for mass analysis of small tip regions. Presently we are more interested in energy analysis of field ions, for which a resolution of 1 eV is available. Multiple Jason peaks are easily seen. The appearance conditions of the newly discovered ions from the forbidden zone\(^2\), created by electron impact excitation of apex-adsorbed image gas atoms have been studied in some detail as their energy level presents a direct experimental proof of the apex location of the image gas adsorbate. The abundance of He\(^{a+}\) or Ne\(^{a+}\) in He-H\(_2\), Ne-H\(_2\) and He-Ne-H\(_2\) mixtures, as well as corresponding mixtures with Ar and Kr as a source of the exciting electrons will be discussed.

The energy distribution of free-space field ionization of H\(_2\), and more practically D\(_2\), is being used for a much needed voltage-field calibration of field ion emitters.\(^3\) Ratios of the effective field above tip regions with the extremely varying local radii of the field evaporation endform are derived.

\(^1\)E. W. Müller, Laboratory Practice \textbf{22}, 408 (1973).


*Supported by the National Science Foundation
Ion Energy Distribution of Field Ionized 
He, Ne, H₂ Near Fermi Level of W and Pt

by

T. Utsumi
Bell Laboratories
Murray Hill, New Jersey 07974

In order to understand the field ionization process in terms of atom-surface interactions, the detailed analysis of ion energy distribution seems necessary since it is the only way to compare the experimental results with the calculations based on various models such as the one-dimensional WKB method,¹ rearrangement collision-Born approximation,² metal plane wave-atomic wave interaction,³ 6s, 5d metal orbitals-atomic wave interaction,⁴ and the Bardeen type transfer Hamiltonian approach.⁵

In this talk we report our experimental results of ion energy distributions of the main ionization peak (near Fermi level) of He, Ne, H₂ on some key crystal planes of W and Pt, and also we describe the performance of our FIM-probe hole-retarding lens-retarding energy analyzer system.

Total Energy Distributions from CO Covered Tungsten

Peter Young and Robert Gomer
James Franck Institute
The University of Chicago

Some results for the CO/W system have recently been published. Additional work to be reported includes measurements on the (111) plane, and more detailed investigations of temperature and coverage dependences on various planes. The coverage dependence of the Virgin-CO resonance on (110) is also found on (111), but there are interesting quantitative differences which can be explained in terms of differences in substrate geometry. Heating of full layers to moderate temperatures, sufficient to cause partial desorption but not complete conversion from the virgin state, causes the virgin resonance to reappear on (110) and (111). On (111) similar behavior is also found for alpha-CO.

These results will be discussed in terms of a molecular orbital model for CO adsorption.

Energy Analysis of Photo-Stimulated Field Emission*

Richard S. Polizotti, Gert Ehrlich and Ruichen Liu
Coordinated Science Laboratory**
and Department of Metallurgy
University of Illinois at Urbana-Champaign

Attempts to detect the contribution of photo-excited electrons in field emission from a metal\(^1\)\(^-\)\(^4\) have in the past been based on detecting the electrons emitted in phase with the chopped radiation incident upon an emitter. To establish this effect directly, however, it is necessary to examine the energy distribution of the emission: electrons excited by photons of energy hv should begin to make a contribution at an energy hv above the Fermi level. We have carried out such observations on tungsten, irradiated with light from a krypton ion laser (hv=2.409 ev), using a spherical shell retarding potential analyzer. These measurements, which have revealed the presence of photo-stimulated electrons in the expected range above the Fermi energy, will be reported in detail.

\(^2\)Y. Teisseyre, R. Coelho and R. Haug, Compt. rend. 274, 1202 (1972).

* Supported by the Air Force Office of Scientific Research (AFSC), USAF, under Grant AFOSR 72-2210.

Photon Stimulated Field Emission from Tungsten*

M. J. G. Lee
James Frank Institute, Department of Physics
University of Chicago
Chicago, Illinois

Field emission of hot electrons has been observed from a small region of a tungsten tip illuminated by a focused laser operating in the ultraviolet (3.531 eV) and in the visible (2.602 eV).\textsuperscript{1} In low electric fields, such that emission occurs below the peak of the Schottky barrier, the field dependence of the total hot electron current is consistent with a simple theory based on the barrier penetration factor derived by Murphy and Good. In high fields, the field dependence of the total hot electron current, considered in conjunction with the observed total energy distributions, suggests that the detected electrons of a given total energy passed through the metal surface with a narrow range of normal energies. Some possible interpretations of this result will be discussed.


*This study was supported by a Research and Study Grant from the General Electric Company and benefitted from the general support of the Materials Research Laboratory through the National Science Foundation.
Investigation of Field Electron Emission Noise Spectra

L. W. Swanson
Oregon Graduate Center
Beaverton, Oregon

Further studies of the power spectrum of noise from clean and adsorbate coated field emitters have been carried out. Studies on the clean W surface include the (100), (310), and (112) crystal faces where the spectral density function $W(f)$ has been measured as a function temperature, frequency and probe current. Similar studies have been completed for CO on W(100) and W(310) where structure in the $W(f)$ vs T curves can be related to the onset of diffusion of the various adsorption states. The ability of using these results to calculate diffusion coefficients of clean and adsorbate coated surfaces will be discussed.
Coverage Dependence of Field Emission Flicker Noise for Single Crystal Planes

R. Męclewski*, Ch. Kleint** and R. Błaszczyszyn*
*Institute of Experimental Physics
University of Wrocław, Poland
**Sektion Physik, Karl-Marx-University Leipzig, DDR

The dependence of field emission flicker noise on potassium coverage was measured for single crystal faces of tungsten in sealed-off field emission tubes with Faraday collectors. As the spectra density functions show a complicated behavior,\(^1\) noise power measurements \((\delta T)^2\Delta F(\theta)\) were done at constant temperature for continuous deposition onto the emitter tip and also for increasing temperature starting from monolayer coverage. By magnetic deflection the /011/, /112/, /122/, /111/ and /001/ planes, and the /011/-vicinals could be investigated. All of the measurements gave sharp minima in the noise power exactly at the coverages corresponding to the voltage minima for constant collector current.

The coincidence of noise minima and work function minima is discussed using the surface diffusion models of field emission flicker noise\(^2\),\(^3\) and theories of the work function minimum for electropositive adsorbates. Separate experiments have shown maxima in the surface diffusion activation energy and equilibration temperature at the coverage corresponding to minimum work function. Thus it seems that steric hindrance of elementary surface diffusion processes is a reasonable explanation of the noise power minimum accompanying the work function minimum.


\(^3\)Ch. Kleint, R. Męclewski and R. Błaszczyszyn, Physica, to be published.
Field Emission Work Function Difference  
for Alternating H. C. P. Finite-Size  
Planes  

A. J. Melmed, J. J. Carroll, J. Smit, and E. W. Plummer*  
National Bureau of Standards  
Washington, D. C.  

An alternating structure occurs for consecutive h. c. p. crystal planes when 2h+4k+3l is not an exact multiple of six. Field-ion microscopy techniques allow the controlled preparation and imaging of many crystallographic regions where this condition prevails for the h. c. p. metal ruthenium. In the present work consecutive (100) planes of liquid nitrogen cooled Ru were prepared by field evaporation, imaging with helium in a FIM-FEM tube with a base pressure less than 1 x 10^-10 torr. Probe-hole Fowler-Nordheim measurements were made for consecutive planes as a function of the net plane radius.

The resulting Fowler-Nordheim slopes showed a significant functional dependence upon the net plane radius and the functional dependence was significantly different for the two plane types of the alternating planes. This variation of slope with net plane radius can easily be explained by calculation of the current entering the probe hole from the two types of planes as a function of net plane radius, including consideration of local field effects. All of the features of the experimental measurements have been reproduced by a field emission electron trajectory calculation.1

The following conclusions are made:

(1) The electron trajectory calculation of reference 1 is valid.

(2) Given a maximum allowable error in a determination of Fowler-Nordheim slope, there corresponds a range of allowable plane sizes for a given tip.

(3) There is a work function difference due to surface atomic geometry.

(4) The results strongly imply that the relatively bright and dim (100) Ru field-ion images correspond to the net planes with larger step height (more deeply rippled) and smaller step height (less deeply rippled) respectively.

1J. W. Gadzuk and E. W. Plummer, Rev. Mod. Phys., July 1973

*Present address: Department of Physics, University of Pennsylvania, Philadelphia, Pennsylvania.
Location of Individual Adatoms at a Surface*

W. R. Graham and Gert Ehrlich
Coordinated Science Laboratory**
University of Illinois at Urbana-Champaign

Despite considerable interest in the properties of crystal surfaces, one problem has remained unsolved—the location of an isolated adsorbed atom in relation to the atoms of the underlying lattice. We have applied the field-ion microscope to a determination of the equilibrium position of a single tungsten atom self-adsorbed on a perfect plane of a tungsten crystal. The techniques used for establishing atom locations and for eliminating field effects will be detailed. Although quantitative measurements of the interatomic distances are not yet possible, the preferred sites for binding have been established on the (111) plane.

* Supported by the Air Force Office of Scientific Research under Grant AFOSR 72-2210.

**Operated under JSEP Contract DAAB-07-72-C-0259.
Observation of Indirect Adatom Interaction on Metal Surfaces

T. T. Tsong
Physics Department, The Pennsylvania State University
University Park, Pennsylvania 16802

In previous papers\(^1\) we have reported that Re adatoms on a W(110) plane exhibit oscillatory structures in their interactatomic potential. Here we shall report a preliminary attempt to obtain quantitative data on the pair potential. The potential, in principle, can be determined from measurements of the pair distribution function, which gives directly the potential of mean force. This potential is then related to the pair potential through the Kirkwood integral equation, the Born-Green-Yvon integral equation or some other relations. While a theory for our specific problem is still lacking, it is known that the potential of mean force approximates well the pair interaction potential when the particle density is very small. Our data gives an oscillatory pair potential with its magnitude of the same order as predicted by theories.\(^2\) This technique however suffers a lack of accuracy because of the non-uniform magnification of FIM images and the limited amounts of data obtainable. Nevertheless, it is a straightforward experimental evidence of the indirect adatom interaction on metal surfaces.

\(^1\)T. T. Tsong, Phys. Rev. B6, 417 (1972); B7, 4018 (1973).


*Supported by the National Science Foundation.*
Field-Ion Imaging of Titanium and Effects Due to Hydrogen

J. J. Carroll, J. Smit, and A. J. Melmed
Metallurgy Division
National Bureau of Standards
Washington, D. C.

Titanium has resisted efforts to obtain good field-ion microscope images over extensive regions of the surface developed by field-evaporation. Recently published results\(^1\) show a considerable degree of surface disorder and the presence of tenacious films. We have reported these results, generally, and have found that the use of a specimen preparation technique which minimizes specimen corrosion results in improved field-ion micrographs.

Titanium was field-ion imaged in hydrogen and in neon at various temperatures between 23 and 87 K. Although specimens were slowly evaporating, good micrographs were obtained using micro-channel plate image intensification. Occasionally, plate-like stable inclusions were seen which were probably hydrides. More interesting, however, was the appearance of relatively bright lines\(^1\) or rows of atoms, and associated crystallographic defects when imaging was done with hydrogen. These image features, which were generally in the \(\{110\}\)-\(\{100\}\) regions, disappeared after a small amount of field-evaporation in neon and reappeared upon subsequent re-imaging with hydrogen. We believe that these image features represent incipient hydride formation.

\(^{1}\)Helen E. Grenga and Rajinder Kumar, Surf. Sci. 29, 447 (1972).
Field Ion Microscopy of Metal Contacts*

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Mechanical contacts between FIM tips of tungsten and platinum at cryogenic temperatures and an outgassed platinum contact plate at room temperature were performed under ultra high vacuum conditions. The contacts were accomplished by means of an electromagnetically controlled approach of the plate to the tip. With this highly refined technique, in contrast to previous results,\textsuperscript{1,2,3,4} the tip can now be repeatedly contacted at light loads \textasciitilde1 mg without inducing gross deformation, the applied load can be resolved to as little as 5 \textmu g (depending upon the current scale used) and the contacted area of the tip can be easily identified against the background of the uncontacted parts of the tip.

In the case of tungsten tips, it has been found that although the contact area increases with an increase in the applied load, damage is confined to the top 3 to 7 (110) atomic layers. The tungsten tips were found to be bent out of the field of view before extended lattice defects could be generated. Platinum tips generally exhibited similar behavior to that of tungsten except that smaller loads were needed to bend the tips, and that twins and twin slices resulting from slip\textsuperscript{4} on the \{111\} planes could occasionally be induced.

Preliminary results of low voltage (0 to 5 Volts emf) electrical contacts between tungsten tips and the platinum plate have indicated that markedly different results from those described above are obtainable.


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Field Ion Microscopic Study of Electric Contact Phenomena

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The field ion microscope was applied to the study of electric contacts. Emitter tips of the microscope were contacted with liquid gallium in vacuum. The threshold closing voltage and opening current to create an amorphous state in the emitter tip were obtained for W-Ga contacts. The characteristic feature of the Re-Ga contact is similar to that of the W-Ga contacts. However, the threshold values could not be obtained because of the Re-Ga reaction. The observed threshold closing voltage is significantly smaller than the conventionally known values. The cause of the disorders at these low closing voltage is explained as the result of the field emission for the positive tip and of the bombardment of the field ionized gallium ions for the negative tip. The increase of the threshold values with the tip radius is also discussed for the closing and opening contacts.
Field-Ion Microscope Observations of Low Energy Ion Bombardment

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and

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An essential preliminary to much surfaces research is the production of a clean specimen surface. One of the most effective methods of surface cleaning is a combination of ion bombardment and heating. However, although such surfaces may actually be chemically clean (as monitored, for example, by Auger electron spectroscopy), their exact topography is not defined and may exert a considerable influence on the subsequent experiment. Moreover, it is well established that ion bombardment introduces considerable surface and sub surface damage not all of which may be removed by subsequent annealing. Clearly, the efficiency of the cleaning process must be balanced against the associated lattice disorder. It is the characterization of these optimum conditions with which the present study is concerned.

The field-ion microscope seems an ideal technique to investigate these effects and here we report on the damage produced in tungsten specimens subjected to low energy inert gas ion bombardment. These in situ experiments were all conducted in a U. H. V. environment to enable "field-off" conditions to prevail during bombardment. These experiments have revealed the differences produced using ions of different species (He⁺, Ne⁺, Ar⁺, Xe⁺) and of different energies in the range 100 eV to 1 KeV. Information has been obtained regarding the depth and type of damage caused by each ion species as a function of incident ion energy.

A parallel study has revealed that ion etching may be a valuable method of producing field-ion microscope specimens. Using a technique similar to that used to prepare transmission electron microscope specimens, field-ion emitters have been produced from materials usually difficult to electropolish. The methodology required for the consistent production of sharp field-ion emitters in this way has been established.
Decomposition of Nitric Oxide on Ruthenium

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The decomposition of nitric oxide on ruthenium is a catalytic process. It is easily seen with the field emission technique, and is one of the few reactions that have been observed in this manner where the mechanism of the reaction can be established. It has been found that nitric oxide is chemisorbed on ruthenium dissociatively. Nitric oxide, deposited on one side of a ruthenium tip in excess of a monolayer, does not exhibit the surface migration usually associated with second layer migration. Instead, migration commences only at about 250 K. This is the same temperature region in which oxygen on ruthenium migrates in a chemisorbed layer with a concentration gradient. A complete monolayer of oxygen gives a work function increment of 1.1 eV over that of clean ruthenium based on comparison of Fowler-Nordheim slopes. A complete layer of nitric oxide, achieved by spreading NO at 250 K showed a $\Delta \phi$ of 0.6 eV. Initial deposition was from a molecular beam on one side of a tip at 4.2 K to several molecular layers.

Nitrogen does not chemisorb on ruthenium. The monolayer shows interesting behavior in that comparison of its F-N slope with clean Ru shows identical slopes but $\Delta (\ln A) = 2.8$ where A is the pre-exponential factor. The nitrogen adlayer is completely desorbed in 15 seconds at 150 K.

The course of the pattern development of a nitric oxide layer that has been spread at 250 K and then heated further shows all the characteristics of that oxygen on ruthenium. The catalysis of the nitric oxide decomposition is related to the strong oxygen-ruthenium and the weak nitrogen-ruthenium interaction.
A Measurement of the Differential Heat of Adsorption of Metallic Adatoms on Single Crystal Faces (Pb/W)

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To measure the isosteric heat of adsorption of metallic adatoms on single crystal faces of a metal, a field emission method is developed.

An adsorption equilibrium is established between an impinging flux of metal atoms (Pb) and the thermal desorption of the adatoms. The isosteric heat of adsorption $q_\theta$ is determined, using a modified form of the Clausius-Clapeyron equation.

$$q_\theta = R \left( \frac{\partial \ln \dot{n}}{\partial \frac{1}{T}} \right)_\theta$$

where $\dot{n}$ is the incident flux of atoms, $\theta$ the degree of coverage, $T$ the temperature of the crystal and $R$ the gas constant.

The measurement is made in a field electron microscope. The degree of coverage is controlled by measuring the field emission current from one face by a probe-hole technique. One value of the current (of $\theta$) can be realised by several equilibrium conditions, i.e., by several pairs of values of $T$ and $\dot{n}$. The curves $\ln \dot{n}$ versus $1/T$ are straight lines. For each value of $\theta$, $q_\theta$ is obtained from the slope of the corresponding curve.

Relative and absolute numbers of atoms per cm$^2$ are determined by experiments at constant flux, using microbalance results of Bauer et al.

Some results are:

For $\theta \ll 1$:

$q_{112} = 82$ kcal/mole, $q_{113} = 85$ kcal/mole,

$q_{111} = 115$ kcal/mole

For $1 < \theta < 2$ $q_{112} = 54$ kcal/mole, $q_{113} = 43$ kcal/mole

The only available theoretical $q$ value (KALAFI) is $q_{111} = 94$ kcal/mole ($\theta \ll 1$).

To control the method, the activation energy of desorption of Pb is measured for the faces (112) and (111). In both cases, 82 kcal/mole are measured ($\theta < 1$). The apparent desorption energy of a face can be smaller than the corresponding adsorption energy (Schmidt and Gomer). This may explain the difference between the two results for (111).
Obtaining Tantalum Micromonocrystals in [111]
Orientation and Some Effects of Surface Selfdiffusion

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From a polycrystal tantalum wire of 0.1 mm in diameter a needle with a 0.01 mm thin neck and a length of 2-3 mm is obtained through electrolitic processing in hydrofluoric acid. After primary recrystallization at a vacuum of 10 Torr and temperatures of 1800-2000 K the tip is covered with several layers of gold. The gold film changes surface energy anisotropy of the primary crystal grains in such a way that after secondary recrystallization at 2300-2500 K the crystal grain with its [111] coinciding with the tip axis growths preferably.

The surface selfdiffusion of such microcrystals has been studied with an FEM. For the activation energy with and without an electric field the results are the following: \( Q_e = 1.8 \pm 0.1 \text{ eV} \) with and \( Q_o = 2.4 \pm 0.2 \text{ eV} \) without an electric field. It is shown that, as a result of the surface selfdiffusion in an electric field, the emission is centered in a narrow area around the [111] axis i. e., a cathode is obtained with the following beam parameters: angular divergence of beam 0.1 - 0.2 sterad.; current density 10 - 10^7 A/cm^2; brightness 10^6 - 10^9 A/cm^2, sterad., and maximum current of 500 mA. The voltage needed for the emission is 2 - 2.5 times lower than on an ordinary tip.
Anomalous Behavior of Surface Self-Migration in the Presence of Carbon, Silicon and Germanium

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The surface self-migration of several transition metals with carbon, silicon, and germanium impurity atoms on the surface has been studied using pulsed field-electron emission techniques. In several possible combinations, i.e., carbon on tungsten, silicon on tungsten, carbon on tantalum and carbon on molybdenum, significant increases in the activation energy for surface self-migration over the values for the pure metals with clean surfaces were found. Several anomalous effects were observed.

1) In three of the metal-impurity systems which exhibited large increases in activation energy, the evidence indicates that lattice steps several atom layers high are observed at the edge of the (110) plane. As migration of material from these planes progressed, the multilayer edges appeared to move as a unit across the plane's surface. This multiplayer phenomenon is not observed for migration on clean metal surfaces.

2) The activation energy for the carbon on tungsten system and the silicon on tungsten system showed two distinctly different regimes when plotted as a function of temperature. In both cases, the activation energy at lower temperatures was two to three times that for clean tungsten. As the temperature was increased, a fairly sharp break in the curve occurred (at about 1975°K for silicon on tungsten and 2250°K for carbon on tungsten) above which the activation energy decreased to nearly that for the clean metal.

3) Simultaneously with the transition from high to low activation energy, the multiple step height phenomenon reverted to single atom layer steps as observed for the clean metal.

4) For both silicon on tungsten and carbon on tungsten (433) planes appeared as dark "holes" in the emission patterns. These planes are not developed on the patterns of clean tungsten. A critical phenomenon was associated with the appearance of these planes. At a reproducible (coverage dependent) temperature (1250-1550°K), the planes disappeared, reappearing abruptly when the emitter was cooled below the critical temperature. Also, collapsing
or receding step edges were observed on these planes much like those seen on (110) planes.

The interpretation of the several effects noted is that the impurity atoms on the surface layers bond with the metal atoms to form surface phases of the respective refractory metalloids. The abrupt changes of surface structure and properties at "critical" temperatures seem to be surface phase transformations.

Thus a given metal-impurity system should have its own unique surface phase history related to but different than that for the bulk alloys.
Tip Blunting by Evaporation and Surface Diffusion

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The blunting of an field emitter tip by surface self-diffusion has been studied experimentally (for example by E. W. Mueller, Dyke et al., Warner and more recently by the authors) as well as theoretically (Herring, Nichols and Mullins). It is of interest to study the blunting, if evaporation of the tip substance occurs additionally. Therefore, the increase of the curvature radius near the tip apex is calculated for some metals (W, Mo, Pt, Ni, Cu) as a function of time, temperature (evaporation and surface diffusion) and cone angle. The evaporation should lower the blunting and finally result in the appearance of an upper radius limit.

To verify some of the predictions, experiments are made under conditions where the evaporation rate is not negligible. Profile changes of Mo tips during annealing are measured by a field electron emission and scanning microscope technique. The predicted lowering of the blunting rate as well as the existence of a radius limit are confirmed.

The described phenomenon must be of importance in some cases of surface diffusion measurements.

The blunting rate should also be lowered and a radius limit should exist if an evaporation occurs as a consequence of a reaction of a gas on the tip surface. Therefore we measured the radius increase of tungsten tips during annealing in the presence of oxygen of $1.5 \times 10^{-5}$ Torr and $2.5 \times 10^{-5}$ Torr, where evaporation of tungsten oxides occur. Also in this case the predictions are confirmed.

If the initial radius is greater than the limit radius the evaporation must be dominant and the result should be a decrease of the tip radius during annealing. This also agrees with experiments on tips of W in oxygen and on tips of Mo in vacuum.
Remolding of a Field Emission Cathode

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The remolding\textsuperscript{1} conditions that provide high angular confinement of field electrons from \langle013\rangle and \langle001\rangle oriented tungsten emitters will be described in detail. Investigations were also made on the effect of polarization on the thermal-field induced surface migration of metal atoms during the remolding. Surface migration energy, which is estimated from the remolding rate at 75 MV/cm, was 2.25 eV.

Experimental results show that the end form of the thermal-field build-up of crystal planes on tungsten and rhenium emitters is determined by the anisotropic feature of the surface free energy, which is influenced by the adsorption of gases and polarization of surface metal atoms caused by the remolding field.

Discussion will be given on possible causes of the thermal-field build-up of the (001) crystal plane of tungsten emitter exposed to \(\text{CO}_2\) or \(\text{O}_2\) gases of about \(10^{-7}\) torr.

A Preliminary Study of Carbon Fibre Field Emitters

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Several workers\textsuperscript{1,2} have found in recent years that Carbon Fibres can be made into field emitters that will emit a relatively stable current in vacua of the order of $10^{-8}$ torr. This could give them an advantage over metallic emitters in certain applications. We have produced carbon fibre emitters by a simple preparation technique, and have measured the long-term stability of the total emission current under this kind of vacuum condition. The results are similar to those of other workers.\textsuperscript{1,2}

Carbon is interesting in that its electronic properties are intermediate between those of semiconductors and metals. Semiconductors have given field emission energy distribution broader than those from metals\textsuperscript{3,4}; Salomon and Braun obtained a half-width of the order of .6 eV for Cadmium Sulphide. We have measured the energy distribution from a carbon fibre emitter with the retarding potential analyser described by Salmon.\textsuperscript{5} The half-width at room temperature is about .2 eV, which is comparable with the half-width obtained from tungsten emitters. Thus carbon fibres behave in this respect like metallic emitters and not like semiconductors.

Our work reinforces the impression\textsuperscript{1,2} that carbon fibre emitters might be suitable for use in practical electron guns for high-resolution applications.


The Behaviour of the Carbon Field Emitter as an Electron Source

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There are considerable advantages to be gained by using tungsten field emitters rather than hot cathodes as electron sources. Unless, however, the vacuum within the device is better than $10^{-10}$ torr, the emission from the tungsten soon becomes erratic, leading to a final breakdown of the source.

Much effort has been expended to find a material which will behave as a field emission source and have a reasonable lifetime in the pressure range $10^{-8}$ to $10^{-6}$ torr, the span of pressure normally found in commercially made devices. Our investigations would indicate that such a material has been found, namely carbon in the form of carbon fibres.

The fibres are seven microns in diameter and have a resistivity of $2 \times 10^{-4}$ Ω-cm. These are etched to a fine point by using a controlled corona discharge in air. Using this method fibres can be reproducibly etched to give 1 μA at 500 ± 50 volts. The fibres will emit electrons continuously at pressures between $10^{-8}$-10^{-7} torr for many thousands of hours at current levels as high as 50 μA, and have been tested for 500 hours at 100 μA. They all break down catastrophically at 400 μA. The cone angle of the emitted beam lies between 90° and 60°. The emission is noisy particularly at higher pressures and obeys a 1/f relationship.

A simple scanning microscope with a resolution of approximately 2,000 Å using the carbon fibre as an electron source has been constructed and gives stable pictures at a system pressure of $5 \times 10^{-7}$ torr.
Contrast from Martensitic Interfaces in Field-Ion Micrographs*

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The matching of planes which was first shown to occur across twin boundaries\textsuperscript{1} has been found to be applicable to martensitic interfaces as well. The examples of the f.c.c-h.c.p. boundary in cobalt and the f.c.c-b.c.t boundary in steel will be treated in detail. This m:n ring matching contrast effect is useful in the identification of small regions of retained austenite as well as internal twins within the martensite crystals.

A simple derivation involving the use of the concept of the reciprocal lattice will be indicated. This permits the determination of m and n values and also the limits of application of the m:n matching theory for twin boundaries.


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Some Initial Studies of Precipitation in Refractory Metal Systems

by

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The early stages of Group IV metal (Ti, Zr, Hf) carbide and nitride precipitates in molybdenum and tungsten based alloys have been studied by field-ion microscopy and comparative high resolution transmission electron microscopy. This work has two purposes:

1) to obtain a better understanding of the field-ion imaging characteristics of two phase materials using matrices whose imaging characteristics are well established.

and

2) to provide a model system for the study of interstitial-substitutional interactions in b.c.c. metals e.g. carbide and nitride precipitation in low alloy steels.

Molybdenum-TZM alloy wire (0.5 wt% Ti, 0.1% Zr, 0.015C) that has been carbidized in an atmosphere of propane and ammonia produces contrast attributed to platelets coherent with the matrix. The contrast is consistent with the electron microscope observations that Ti(C,N) precipitates as coherent plates on $\{310\}_{\text{Mo}}$ habit planes.

The tungsten-based alloy system contains 4% Re, 0.4% Hf, and 0.02% C. On thermodynamic grounds, the formation of hafnium carbide is favoured. The carbide coarsening rate in the alloy is controlled by the substitutional diffusion of the carbide forming solute, and, as a result, fine well-dispersed stable precipitates of hafnium carbide are expected.

The alloy has so far been studied at three stages: in the as-received condition, after annealing to just below the solvus, and in the solutioned and quenched form. The microstructures for the first two states are similar, containing approximately spherical particles, although the distribution and size of the particles are not uniform. In the solutioned and quenched condition small coherent platelets are observed, and the orientation relationship is consistent with that expected on a criteria of good matching between hafnium carbide particles and the matrix.
Some aspects of the contrast theory for understanding the images of small particles in the field ion microscope will be presented.
FIM Study of Precipitation-Strengthened High Strength Steel

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A field ion microscope study on high strength steel containing small amounts of V was made to elucidate the morphological differences of precipitates in specimens RH and RL hot-rolled at higher (800°C) and lower (700°C) temperatures respectively.

Hydrogen ion images showed that in both rolled specimens existed two kinds of precipitates: granular ones (50~200Å in diameter) imaged as bright spots on {001}$_{\alpha}$ along <011>$_{\alpha}$, and plate-shaped ones imaged as dark thin bands on {001}$_{\alpha}$. The former in the RL specimens were smaller in size and more uniformly dispersed than those of RH specimens. In one RL specimen, some granular precipitates were observed near a dislocation loop.

When tips of the both types of specimens were heated at about 500°C under vacuum (~10$^{-7}$ Torr) for about 15 minutes, the granular precipitates disappeared whereas the dark thin bands representing the plate-shaped precipitates were transformed to rows of bright spots.

The net plane edges of matrix ferrite of RH specimens were imaged more clearly than those of RL specimens.

The He-H$_2$ mixture gave clearer and stabler ion images, but somewhat smaller ones for precipitates than pure H$_2$ gas did.
FIM-studies of Disordered and Ordered Alloys in the System of Au-Pt at Different Temperatures

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In addition to previous FIM-investigations of alloys with bcc crystal structure now the system Au-Pt with fcc structure has been studied at different temperatures with Ne ($10^{-6}$-10^{-3} Torr) using a 3" channel plate.

In agreement with the new diagram of constitution of the Au-Pt system the ordered phase Au$_3$Pt has been verified when this compound is quenched from 950°C to room temperature. All other binary compositions show disordered structure. The superstructure of Au$_3$Pt is indicated by the regularity of its FIM image and also by the different evaporation rates of the crystal for the alternating layers of gold and gold-platinum netplanes observed especially for (001), (102) and (011). The FIM-picture of the Au$_3$Pt surface coincides well with that simulated with a computer.
Field-Ion Microscope Study of Ordered Domain Structure and Ordering Process in Ni$_4$Mo Alloy

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Field-ion microscope studies on ordered domain structure and ordering process in Ni$_4$Mo alloy were made.

The kind of ordered domains in the FIM images showing a domain structure and consequently the type of ordered domain boundaries were successfully identified by a simple method in this research, considering the crystallographic relations between individual ordered domains.

The domain structure in the ordered Ni$_4$Mo ($\beta$) consists of ordered domains with six distinct orientations. A pair of three domains are antiparallel to each other. Three domains in each group can be brought about from one another by rotating successively by $2\pi/3$ (radians) about [111]$_{\text{FCC}}$ axis.

FIM images of the Ni$_4$Mo alloy which was ordered to various extent were analyzed by utilizing the above crystallographic relation and also taking into account the following factors, namely (i) the B.C.T. symmetry (which is a property of the ordered Ni$_4$Mo structure), (ii) $\{111\}_{\text{FCC}}$ dark regions and $<110>_{\text{FCC}}$ dark bands$^1$ (which have appeared in FIM images at all stages of ordering), (iii) the relative size of ring steps (which is in order of decreasing d spacing)$^2$, (iv) the arrangements of bright spots in (hkl)$_{\text{BCT}}$ plane, and (v) the number of net ring steps between two poles.

The results analyzed suggest that six kinds of domains of $\beta$ nucleate with almost equal probability in the $\alpha$ matrix (FCC).

The ordering at temperatures below the nose temperature (about 750°C) of TTT diagram proceed homogeneously, but above the nose temperature the ordering process is sensitive to the heating rate to the annealing temperature from room temperature after quenching from 1100°C ($\alpha$-region)$^3$. On higher heating rate ordering mechanism is nucleation and growth, and on slower heating rate the ordering mechanism is similar to that at low temperature.


FIM Studies on Cobalt: Influence of Hydrogen, Nitrogen and Oxygen

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In addition to previous FIM investigations of O. Nishikawa and E. W. Müller high purity cobalt (99.999%) has been studied at 21 K and 78 K using neon with different addition of helium, hydrogen, nitrogen and oxygen. The addition of hydrogen is disadvantageous. A content of helium increases too much the contrast in the FIM image for regions with higher field strength. Nitrogen has no remarkable influence. A partial pressure of oxygen (< 10⁻⁶ Torr) increases the field evaporation rate when the end form of the tip is performed. The chemisorption of oxygen diminishes the resolution but stabilizes the mechanical properties of the tip by hindering slide processes. The density of stacking faults is found to be very small for the pure cobalt. No stacking faults are found for cobalt with (0001) orientation but high densities of stacking faults when this plane is no more perpendicular to the axis of the tip.

Reference:

Adsorption of Potassium on Tungsten and Molybdenum

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The adsorption of potassium has been studied on single planes of rotatable tungsten and molybdenum field emitters. Work function, $\phi_{hkl}$, versus surface concentration of potassium, $n_{hkl}$, has been measured for coverages up to $5 \times 10^{15}$ atoms/cm$^2$. The measurements were made both at liquid nitrogen temperature /immobile layers/ and at elevated temperatures /at which surface diffusion occurred/. In both cases similar curves are obtained: the work function decreased linearly at first, then moves slowly, passes through a minimum, and then attains a constant value /the saturation value/. Some planes require higher surface concentrations than others to reach saturation. The shapes of the $\phi_{hkl}$ versus $n_{hkl}$ curves are different for various planes at coverages of $\theta > 1$. The values of the work function minima and corresponding potassium surface concentrations were determined extremely precisely.

The results obtained for tungsten and molybdenum are similar. Since the difference in lattice constants is small it seems that the geometry of the plane rather than the electronic structure of the substrate influences on the concentration dependence of the work function.

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The Reaction Between Ethylene and Tungsten
Studied in the Field-Ion Microscope

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The adsorption of ethylene on tungsten has been studied in the F. I. M. at ethylene pressures from $1 \times 10^{-3}$ torr to $5 \times 10^{-8}$ torr, the tungsten being either at 78K or heated.

Heating the tungsten in high pressures of ethylene to temperatures exceeding about 1000°C ultimately produces surfaces which are ascribed to the carbides WC and W₂C. Heating to these temperatures for short periods of time produces surfaces which are interpreted as showing the initial stages of the carbide layers.

Adsorption at lower gas pressures or lower temperatures produces no signs of carbide formation. Colour-superposition photography has been used to determine the "active" sites for adsorption and the reactions observed are consistent with the first stages of the reaction by which carbide is formed under more rigorous conditions.
Mass Spectrometric Analysis of Field Desorbed Species 
Formed at a Silver Surface in the Presence of 
O₂, H₂O, H₂, CO, NH₃, CH₄, N₂, Kr and Xe.

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Stimulated by earlier FEM/FIM investigations in the 
silver-oxygen system it was of particular interest to 
analyze the surface compounds mass spectrometrically. Sur-
prisingly, no silver-oxygen compound were found if molecular 
oxygen was offered to the tip surface at field strength 
high enough for field desorption. However, with atomic 
oxygen formed by secondary reactions from the well known 
H₂O⁺ reaction, AgO⁺ and AgO₂⁺ were detectable showing posi-
tively charged silver-oxygen compounds to be existent.

Another remarkable result of the mass analyses of field 
desorption products in the presence of H₂O was the forma-
tion of the ions Ag(H₂O)⁺, N = 1, 2, 3, 4, appearing with 
high abundances. Because of this behaviour, the ability of 
a silver surface to form compounds with other gases was 
studied. With the gases H₂, CO, CH₄ and N₂, the ions AgH₂⁺ 
and Ag(H₂)₂⁺, AgCO⁺ and Ag(CO)₂⁺, AgCH₄⁺, and AgN₂⁺ can be 
formed, respectively. No compounds were detected in the 
presence of Kr or Xe.

Arguments are given to explain the ions with attached 
gas molecules as complex ions of the type [Ag Xₙ⁺]. To 
check the proposed interpretation ammonia was used because 
in the chemistry of complexes the [Ag(NH₃)₂]⁺ complex is 
well known. The expected result was observed even if under 
low temperature conditions ions of the type [Ag(NH₃)₂]⁺ n 
NH₃ were detectable (n = 1, 2, . . . , 6).

As a consequence, this interpretation requires the 
assumption that surface atoms exposed to evaporation field 
strengths are in at least a highly polarized or even 
ionic state. Then, with such field-treated surface atoms, 
chemical reactions are possible which are different from 
those known from chemisorption at zero field conditions.
Premature Field Evaporation in the Atom-Probe*

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Since some time we have realized that delayed time signals affecting the mass resolution are caused by energy deficits of ions field evaporating before the pulse has fully matured. A partial retardation technique has been introduced to measure effective energy deficits. These were found to amount to the order of 1% of the total ion energy, but not to show the mass and charge dependence predicted by the Lucas theory of surface plasmon excitation losses. To support our interpretation we consider the motion of a prematurely evaporating ion in the time- and space-dependent field between the tip and the ground electrode. An analytical approach would involve the solution of a non-linear second order differential equation. We have resorted to a numerical method, using the IBM computer model 370/165 and representing the potential between the electrodes by the confocal paraboloid configuration. The results arising from two different pulse shapes, in which the voltage either approaches the top level exponentially from below, or exponentially from an overshoot, are presented. In the first case, energy deficits due to premature evaporation decrease with increasing ion mass, all apparent masses being shifted upwards. In the second case, mass dependence of energy deviation is opposite, and apparent masses are shifted down. A more complex situation arises when different ion species from one tip evaporate at different field levels. An atom-probe and pulser design assuring the shortest possible rise time and employing low evaporation rates are important for operating the atom-probe under optimal conditions of mass resolution.

1 E. W. Müller, Ber. der Bunsenges. 25, 979 (1971).
   Laboratory Practice 22, 408 (1973).

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Pulse Shape and Timing Errors in FIM Atom Probe

by

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A computer model has been developed to determine the distance, time and energy dependence of ion acceleration in the FIM Atom Probe, based on simple assumptions regarding the pulse shape, and a realistic form of the inter-electrode potential. The sources of energy deficit and late arrival of ions at the detector are analysed in detail, and the magnitude of these effects are calculated for a variety of working conditions. It is shown that two classes of error arise in the spectra, namely peak shifts toward higher mass numbers, and peak broadening. The former can be allowed for in data analysis, but the latter may produce an appreciable loss of resolution in the spectra. Optimum pulse characteristics are proposed for various combinations of inter-electrode spacings and applied potentials.
Measurement of Energy Losses and an Energy Deficit Compensated ToF Atom-Probe*

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A spread of ion energies affecting the resolution of the ToF atom-probe seems inevitable when the acceleration is due to the superposition of the pulse to the dc voltage. Realistic pulse head times are too long to avoid premature evaporation, and an additional energy spread is introduced when field evaporation is thermally activated or gas-promoted. Measurements of ion energy deviations are useful for performance evaluation of the atom-probe and for elucidating complex surface processes of field evaporation in the presence of adsorbates.

Our atom-probe has been modified by the incorporation of an energy dispersing 90° electrostatic deflection system of 50 cm radius and the display of the energy spectrum on a phosphor screen behind a chevron stack of two channel plates. Every single ion can be photographically recorded as a spot which is much brighter than the background noise. A magnetic crossfield provides a two-dimensional display of ion energy with a resolution of the order of 10 eV, and of ion mass with a resolution sufficient to discriminate between image gas adsorbates such as He+ or Ne+ and the variously charged metal ion species. Some unexpected results with the energy deviations of these ions will be reported.

The ion energy dispersion obtained by the electrostatic deflection can be employed to compensate for the inevitable and variable energy losses due to pulse shape limitations by the simple expediency of providing a slightly shorter flight path for the slower ions. In our energy deficit compensated ToF atom-probe1 this is achieved by tilting the flat double channel plate detector by an appropriate angle. The energy deviations range that can be compensated is of the order of 200 to 400 V, conveniently covering the usual deviations, and the precision of compensation is only negligibly affected by focusing problems. Thus a considerably improved resolution of the ToF atom-probe at relaxed pulse shape requirements is attainable.


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Field Desorption Spectrometry*

by

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In this paper we describe an extension of the highly successful time-of-flight atom-probe technique developed by E. W. Müller and co-workers. By reducing the spectrometer drift distance to only 10 cm, the entire FIM image can be displayed on a curved chevron CEMA detector, and therefore, all ion species originating at the surface can be examined at one time. Single atom probing can be accomplished at one or several different crystallographic locations during a single evaporation event by positioning one or several external photomultipliers over areas of interest. Since the desorption image can be directly viewed and photographed the evaporation rate in vacuum can be precisely determined, and aiming problems are completely eliminated. The use of a triode lens similar to that of Brenner and McKinney results in an ion energy determined only by a stable dc supply, and therefore, severe evaporation pulse requirements can be relaxed. Finally, the small size of the device which does not need differential pumping, permits the use of inexpensive, bakeable high vacuum components, and permits rapid pumpdown to $10^{-10}$ Torr. Recent results obtained with the new instrument will be reviewed as well as a discussion of resolution which now approaches $\Delta m/n = 0.8$ at $m/n = 63$. Several unique features of the instrument useful to conventional atom-probe spectroscopy will also be presented including a module which provides an oscilloscope sweep so that the detected species are displayed on a linear mass scale.

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Miscellaneous Results and Effects Relating to the Use of the Atom-Probe for Metallurgical Applications

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The Cambridge atom-probe has been used for a number of short investigations over the past year, none of which warrant presentation on their own. This paper will therefore be in the form of a progress report in which the following topics will be briefly discussed.

1. The construction of a specimen changing air-lock and heater assembly so that specimens may be replaced within a few minutes into a vacuum of the order of $5 \times 10^{-10}$ torr.

2. The problem of residual hydrogen in the specimen itself and how it can bias results by the preferential etching of one or more components in a complex alloy. Examples from the Ni-Al and Co-Ta systems will be given. It will be shown that heating the specimen to a few hundred degrees C prior to analysis can remove most of the hydrogen, although it has to date proved impossible to remove all hydrogen from pure Ta.

3. The relative abundances of the various charge states of ions evaporating from different positions on a tungsten specimen. In general it has been observed that the higher charge state appears at higher evaporation fields. The case of Al evaporating from the pure metal and from a single crystal of Ni$_3$Al will be discussed.

4. The determination of the composition of precipitate particles in the Co-Ta system, and in various steels.

5. Parameters affecting the mass resolution of the instrument. It has been found that the presence of hydrogen or of the image gas, and the evaporation rates the apparent resolution is about 1 in 300, defined as the full width at half maximum.

6. Development of techniques for looking at clustering in long data sequences.

7. Modifications to the data acquisition and control systems.
A Combination Digital 5 ns Timer Measuring Four Sequential Events As Well As Ion Rates*

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Four sequential events can be timed from an initial point with a 5 ns resolution over a range from 10 ns to 100 µs. Further the time of the first recorded event may be delayed until any time within the first 100 µs. In time of flight mass spectrometry this allows 4 masses of interest to be measured in any selected mass interval by ignoring lighter masses.

Accuracy of 0.001% in timing over the entire range is derived by using a controlled oven crystal oscillator time base with a frequency of 200 MHz which is stable within 3 parts per million over a 24 hr period.

When not being actually used as a timer the same single counter chain can be used as a digital ion rate detector for rates from 100 ions/sec to 9,999,900 ions/sec. This detector is helpful in alignment of the detector, in centering a bright atom over the probe hole, and as a pressure indicator.

The circuits emphasize economy in the use of a single power supply for all of the integrated circuits and the use of only 9 MECL 10,000 series integrated circuits, 5 MECL II integrated circuits, and the remaining integrated circuits are all transistor-transistor logic.

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Growth of Tips in the Direction of Normals to The Closepacked Faces by Heating in the Presence of the Electric Field

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Growth of tips from host material in the directions normal to the \{110\}, \{100\} and \{111\}-type faces for tungsten and to the faces of crystallographical zone \<11\overline{2}0> for rhenium was investigated. The growth of such kind, first described in paper\(^1\), was studied by means of a field emission microscope. The latter could be operated both as high vacuum field electron microscope and as helium field ion microscope.

The heating of tips in the presence of the electric field leads both to some known phenomena such as: faceting with closepacked planes (so called "build-up"), micro-roughening of tips surfaces and to the overall growth of the tips and, in general, the growth in the directions normal to the close-packed planes. Macroscopical outgrowth, being formed in this way, have dimensions comparable with the tip radius. They are accordingly faceted and covered with microprotrusions. In order to reveal the macrooutgrowths by means of field emission microscopy it is necessary to remove the microprotrusions. These were either smoothed by gentle heating, or removed by cold field evaporation.

The growing macrooutgrowths form a system of asymmetrically located terraces or ledges. This is due to nonuniform distribution of the field and hence of surface supersaturation and to the statistical nature of nucleating atom layers. Thus the field erosion of the tip was observed, while it had been faceted before with close-packed planes as a result of build-up. The macrooutgrowths have correspondingly the field evaporation endform or annealing form after cold field evaporation or heating without field. The radius of the tip after thermo-field treatment and subsequent heating without field until the annealing form is attained, is often less than before the treatment. Such sharpening extends the resources of field emission microscopy.

Determination of the Shape of Field Ion Tips*

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In order to obtain the shape of a field ion tip, a method extending the use of digitizing and computer graphics was used. The functions describing the projections along the various zone lines was obtained by measurements of the positions of poles with respect to the projection center. Also, the functional relationship between linear distance from the projection center and distance along the projection axis was determined. Then using the above functions, Euler's method of numerical integration was applied to obtain the geometry of the field ion tip.

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Computer Graphics Plotting of FIM Data to Depict Grain Boundary Topography*

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A computer graphics technique for portraying grain boundary topography has now been developed. It utilizes a Calcomp plotter to plot surfaces defined by coordinates obtained by digitizing field ion micrographs. As has been previously reported\(^1\), the digitized data are transformed to specimen coordinates by assuming a spherical tip shape and a stereographic projection. The consequences of these assumptions were not, however, previously fully analyzed. An evaluation of the assumptions has since been made and will be reported.

The grain boundaries are depicted by plotting cross sectional contours and actually two separate methods are used depending on the nature of the surfaces. When the surfaces are free of reentrant angles, orthogonal cross sectional contours are used. When the surfaces are reentrant, one set of contours corresponds to the grain boundary traces seen in the field ion micrographs. Several grain boundary plots utilizing both techniques will be given.

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**Now with the Engineering Science Department, Tennessee Technological University, Cookeville, Tennessee.

1. 19th Field Emission Symposium, University of Illinois, (1972).
COMPUTER SIMULATION OF FIELD-EMISSION IMAGES

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It is rather difficult to interpret field-emission images and therefore all abilities field emission microscope have not been fully realised. While quantitative theory of field-ion image forming, which based on computer simulation, is being successfully developed, but information extracted from field-emission image is to a great extent of qualitative character. Therefore it is worth while elaborating a method of computer simulation of field-emission images.

Results of computer simulation of field-emission image of tungsten tip are given in this work. In the first approximation the following model has been used:

1. Shape of a tip is hemispherical.
2. Dependence of the work function \( \phi \) from packing density of surface atoms \( n \) is defined as empirical formula, which has been received on the basis of analysis of the many experimental data.
3. Local field emission current density was defined by Fowler-Nordheim equation.
4. Stereographic projection surface in screen was adopted.
5. Field above the surface of the tip was assumed as:

\[
P = P_0 \frac{1 - b \cos \theta}{1 - \cos \theta}
\]

\( P_0 \) - field on the top of the tip; \( \theta \) - angle from central axis of the tip; \( b \) - constant.

Initial date of the program is as follows:

a) radius of the tip and its orientation;
b) lattice parameter \( a \)
c) maximum of the sum \( \sum_m = h^2 + k^2 + l^2 \) of indexes of planes appearing on field-emission image;
d) "spread" constant;
e) value of \( P_0, \beta \), and empirical dependence \( \phi(n) \) for a given material.

The results of field-emission images model calculation are given in fig. 1 with the following parameters:

fig. 1 - \( P = 0.5V/A \) = const; \( \sum_m = 20 \); \( r_0 = 300a \)

fig. 2 - \( P = 0.5V/A \); \( \sum_m = 16 \); \( r_0 = 300a \); \( \beta = 0.2 \)

Orientation is the same in both cases: plane (011) is in the middle of the specimen.

Besides it was made computer "photometrical" models in definite cristallographical directions (fig. 2).

Comparison of curvers (fig. 2) with experimental results of photometrical field-emission images has been used for exact definition of values \( \sum_m \) and \( b \).

One can see that the model of computer calculation of field-emission images represented above give results, which agree with already known experimental.
DYNAMIC OF THE INITIAL STAGE OF FIELD-ION IMAGES FORMING
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The initial stage of the tungsten specimens field-ion images forming was studied by field-ion microscope, supplied with image intensifier on the ground three-stage cascade photoelectric tube and with cinematographic registration system. Much repeated standard experiment involved in:

a) polishing (by field evaporation) of the specimen surface and obtaining its stable field-ion image;

b) decreasing tip potential to value at which field-ion image disappeared completely, - the microscope screen in this case was fully shadowed or a very weak light was noticed in the background from residual gas ions; field ionization of the ions require considerably lesser electric fields (indicated value was varied);

c) "momentary" return to initial potential, corresponding to the conditions of the best image of specimen surface, since simultaneous turn on registering cinematograph.

Obtained thus series of the field-ion images microphotographs were worked up; both the color superposition technique, and their photometrical along chosen directions. Their comparative analysis allowed to obtain qualitative picture of the process and determine some of its quantitative (times of the processes) characteristics.

Using the speed of the images registration guaranteed the revealing of single bright spot rise (i.e., every two neighboring microphotographs of given series do not differ on the number of the bright spots on them or indicated difference is equal to unit).

Two parts of the first stage of the field-ion images forming were distinguished under analysis:

1) intensive desorption of the residual gas atoms and molecules, which were adsorbing at time, lowering of the tip potential;

2) increase of number of imaging atoms of specimen itself.

The information obtained for the first part cannot apparently be used enough efficiently because the essence of desorbing molecules is not clear (though conducted on the concrete case analysis of residual gas partial pressure approaches us to unambiguity). At the time intended increase of time intervals $\Delta t$ estimates precision for the second part may be seem very useful for the further development of the field-ion images forming theory generally and process lying on his ground in particular.

For example the small fragments from series of field-ion images for first ($\Delta t = 0.5$ sec, $U=14,0$kv) and second ($\Delta t= 0.5$ sec, $U=13.5$ kv) parts is shown in fig. 1 and fig. 2 correspondently.
ELECTRON SCATTERING PHENOMENA AND THE FIELD ELECTRON ENERGY DISTRIBUTION

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Field Electron Energy Distributions (FEED) have been measured as a function of temperature for a number of crystallographic directions on a clean tungsten emitter. This is of interest because the angular distribution of the d electron wave functions in tungsten has a strong maximum in the [100] direction. The data are interpreted in terms of an electron scattering model; the parameters in the free-electron FEED equation were determined by extrapolation of the data to 0°K, at which temperature the electron scattering phenomena disappear. A qualitative theoretical model has been proposed which includes the probability of non-scattering of transported electrons in the supply function of the FEED equation for transition metals. Both the s electron - d hole and s electron - phonon interaction have been considered. The measured FEED in different directions seems to agree with both the theoretically predicted s → d transitions (N. F. Mott theory) and conduction experiments in transition metals. Along the direction where it is predicted that there is mainly emission from the s band, the experimentally observed variation of the monoenergetic electron current at the Fermi level is a linear and decreasing function of temperature above the Debye temperature. N. F. Mott predicted this effect as a redistribution of s electrons when they undergo transitions to the narrow d band. However, the monoenergetic current along [001] (mainly d electrons) exhibits a slightly increasing temperature dependence at higher temperatures which is significantly different from that observed for mainly s electrons. This character can be explained by s → d transitions in terms of two band electron structure. The Nottingham effect should probably be considered taking into account the above-mentioned scattering phenomena which seems to change the average energy of emitted electrons.