Abstracts of Papers Presented at

The 25th International Field Emission Symposium

July 17-22, 1978
Albuquerque, New Mexico

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Hosted by:
Sandia Laboratories
Albuquerque, New Mexico

Abstracts of the contributions in the various topics may be found grouped according to the indicated pages:

<table>
<thead>
<tr>
<th>Topic</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Biological Imaging</td>
<td>365</td>
</tr>
<tr>
<td>Surface Adsorption</td>
<td>366</td>
</tr>
<tr>
<td>E. W. Müller Memorial Medal Competition</td>
<td>368</td>
</tr>
<tr>
<td>Field Emission and Ion Sources</td>
<td>372</td>
</tr>
<tr>
<td>Surface Chemistry</td>
<td>374</td>
</tr>
<tr>
<td>Field Desorption Mass Spectrometry</td>
<td>375</td>
</tr>
<tr>
<td>Field Emission</td>
<td>377</td>
</tr>
<tr>
<td>Mass Transport and Electrical Breakdown</td>
<td>378</td>
</tr>
<tr>
<td>Metallurgy</td>
<td>379</td>
</tr>
<tr>
<td>Atom-Probe</td>
<td>382</td>
</tr>
<tr>
<td>Emission Spectroscopies</td>
<td>386</td>
</tr>
<tr>
<td>Field-electron and Ion Emission</td>
<td>387</td>
</tr>
<tr>
<td>Controlled Thermonuclear Fusion and Ion Implantation</td>
<td>389</td>
</tr>
</tbody>
</table>
List of Past Field Emission Symposia and Their Organizers

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 and 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 and 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
17. 1970 New Haven, Connecticut  F. Hutchinson
18. 1971 Eindhoven, The Netherlands  A. van Oostrom
21. 1974 Marseille, France  M. Drechsler
22. 1975 Atlanta, Georgia  H. E. Grenga, J. J. Hren et al.
25. 1978 Albuquerque, New Mexico  J. A. Panitz and G. L. Kellogg

The 26th International Field Emission Symposium will be held in Berlin (West) September 3-8, 1979. J. H. Block is Chairman of the Organizing Committee.
INTRODUCTION

The International Field Emission Symposium (IFES) is an annual scientific meeting which deals with the study of the production and uses of electrons and ions generated in high electric fields. Each year, participants from a dozen or more countries throughout the world meet informally for about one week to discuss current research efforts of mutual interest. These may range from basic studies of the interaction of adsorbates on metal surfaces probed by high electric fields to practical applications relating to the structural strength of materials or the determination of first-wall impurity profiles in operating Tokamak reactors. Typically, one hundred or more papers are presented at each meeting, equally divided between basic research and applications to problems of practical interest. In order to promote the international character of the symposium, it is held, alternately, in the United States and Europe. In 1978 the 25th International Field Emission Symposium was held on the campus of the University of New Mexico in Albuquerque, and was hosted by Sandia Laboratories. The local organizing committee attempted to provide a broad and varied technical program as well as the opportunity to become acquainted with New Mexico, "The Land of Enchantment".

A special feature of this year's meeting was the presentation of the first annual Erwin W. Müller Memorial Medal to an outstanding young scientist: A. R. Waugh of Cambridge University, England.

The technical program was highlighted by a number of "distinguished guest lectures" and invited papers. Emphasis was placed on forefront areas of research to which field-emission, field-ion, and field-desorption techniques might be profitably applied. In addition, a special session was organized to review past and present attempts to image biological molecules with high-field techniques, and another to review the "state-of-the-art" in liquid metal field-ion sources.

The symposium was particularly fortunate in acquiring partial travel support from the National Science Foundation for several foreign and United States participants (under Grant No. DMR 78-09078), and from the New Mexico Chapter of the American Vacuum Society, which provided partial travel support for a Distinguished Guest Lecturer.

J. A. Panitz
Local Organizing Chairman

G. L. Kellogg
Local Co-chairman
<table>
<thead>
<tr>
<th>Name</th>
<th>Institution</th>
</tr>
</thead>
<tbody>
<tr>
<td>Abe, T.</td>
<td>Tohoku University</td>
</tr>
<tr>
<td>Abitz, R.-G.</td>
<td>Fritz Haber Institute</td>
</tr>
<tr>
<td>Anderson, R.A.</td>
<td>Sandia Laboratories</td>
</tr>
<tr>
<td>Andrén, H.O.</td>
<td>Chalmers Laboratory</td>
</tr>
<tr>
<td>Barofsky, D.F.</td>
<td>Oregon Graduate Center</td>
</tr>
<tr>
<td>Barofsky, E.</td>
<td>Oregon Graduate Center</td>
</tr>
<tr>
<td>Beaver, P.A.</td>
<td>University of Oxford</td>
</tr>
<tr>
<td>Bell, A.E.</td>
<td>Fritz Haber Institute</td>
</tr>
<tr>
<td>Block, J.H.</td>
<td>University of Cambridge</td>
</tr>
<tr>
<td>Boyes, E.D.</td>
<td>Sandia Laboratories</td>
</tr>
<tr>
<td>Brainard, J.</td>
<td>Sandia Laboratories</td>
</tr>
<tr>
<td>Brainard, R.</td>
<td>Sandia Laboratories</td>
</tr>
<tr>
<td>Brenner, S.S.</td>
<td>U. S. Steel Research</td>
</tr>
<tr>
<td>Bronsveld, P.M.</td>
<td>Jülich GmbH</td>
</tr>
<tr>
<td>Campagna, M.</td>
<td>University of New Mexico</td>
</tr>
<tr>
<td>Cappellucci, D.</td>
<td>National Bureau of Standards</td>
</tr>
<tr>
<td>Carroll, J.J.</td>
<td>Tokai University</td>
</tr>
<tr>
<td>Chang, K.S.</td>
<td>University of Chicago</td>
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<tr>
<td>Chen, J.R.</td>
<td>Bell Laboratories</td>
</tr>
<tr>
<td>Chornik, B.</td>
<td>Pennsylvania State University</td>
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<tr>
<td>Cowan, P.I.</td>
<td>Pennsylvania State University</td>
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<tr>
<td>Culbertson, R.J.</td>
<td>University of Oxford</td>
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<tr>
<td>Cutler, R.</td>
<td>University of Chicago</td>
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<td>Delargy, K.M.</td>
<td>Fritz Haber Institute</td>
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<td>DiPoggetto, R.</td>
<td>Université d’Aix-Marseille</td>
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<td>Domke, M.</td>
<td>Princeton Plasma Physics Lab.</td>
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<td>Drechsler, M.</td>
<td>Louisiana State University</td>
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<td>Eaton, H.F.</td>
<td>Fritz Haber Institute</td>
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<td>Ernst, N.</td>
<td>Pennsylvania State University</td>
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<tr>
<td>Feuchtwang, E.T.</td>
<td>Israel Inst. of Technology</td>
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<tr>
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<td>University of Aston</td>
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<tr>
<td>Forbes, R.G.</td>
<td>Sandia Laboratories</td>
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<tr>
<td>Fowler, G.L.</td>
<td>Fritz Haber Institute</td>
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<tr>
<td>Frank, O.</td>
<td>General Electric Co.</td>
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<td>Glaeaver, I.</td>
<td>University of Bonn</td>
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<td>Giessman, U.</td>
<td>Louisiana State University</td>
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<td>Gipson, G.S.</td>
<td>University of Oxford</td>
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<tr>
<td>Godfrey, T.J.</td>
<td>University of Chicago</td>
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<tr>
<td>Gomez, R.</td>
<td>Hewlett-Packard</td>
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<td>Graham, W.R.</td>
<td>Bell Scientific Lab. Inc.</td>
</tr>
<tr>
<td>Groves, T.</td>
<td>University of Bonn</td>
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<td>Heil, H.</td>
<td>Chalmers University</td>
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<tr>
<td>Heinen, H.J.</td>
<td>Pennsylvania State University</td>
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<tr>
<td>Henjered, A.</td>
<td>RCA Laboratories</td>
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<td>Herman, M.</td>
<td>Tohoku University</td>
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<tr>
<td>Hernqvist, K.</td>
<td>Fritz Haber Institute</td>
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<tr>
<td>Hirano, K.I.</td>
<td>Yale University</td>
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<td>Hummel, E.</td>
<td>University of Tokyo</td>
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<td>Hutchinson, F.</td>
<td>Oregon Graduate Center</td>
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<td>Igata, N.</td>
<td>Tokai University</td>
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<td>Isaacson, M.</td>
<td>University Coll. North Wales</td>
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<td>Isabelle, L.M.</td>
<td>Sandia Laboratories</td>
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<td>Iwata, T.</td>
<td>Jülicher GmbH</td>
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<td>Jones, J.P.</td>
<td>Sandia Laboratories</td>
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<td>Kellogg, C.L.</td>
<td>University of Tokyo</td>
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<tr>
<td>Kisker, E.</td>
<td>Jülicher GmbH</td>
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<td>Knott, M.L.</td>
<td>Sandia Laboratories</td>
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<td>Kohyama, A.</td>
<td>University of Tokyo</td>
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<tr>
<td>Kuhlman, E.</td>
<td>Jülicher GmbH</td>
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<tr>
<td>Kurz, E.</td>
<td>Galileo Electro Optics</td>
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<td>Kuswa, G.L.</td>
<td>Sandia Laboratories</td>
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<tr>
<td>Landolt, M.</td>
<td>Bell Telephone Laboratories</td>
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<tr>
<td>Lee, N.J.G.</td>
<td>University of Toronto</td>
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<td>Leisch, M.</td>
<td>University of Graz</td>
</tr>
<tr>
<td>Levine, J.D.</td>
<td>RCA Labs</td>
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<tr>
<td>Lira-Olivares, J.</td>
<td>Simon Bolivar University</td>
</tr>
<tr>
<td>Lucas, A.A.</td>
<td>Facultés Univ. N.D.de la Paix</td>
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<tr>
<td>Mair, G.L.R.</td>
<td>University of Oxford</td>
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<td>Martinka, M.</td>
<td>Pennsylvania State University</td>
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<td>McLane, S.B.</td>
<td>Pennsylvania State University</td>
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<td>Melaed, A.J.</td>
<td>National Bureau of Standards</td>
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<tr>
<td>Miller, M.K.</td>
<td>University of Oxford</td>
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<td>Mills, F.P.</td>
<td>University of Cambridge</td>
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<td>Milton, O.</td>
<td>Sandia Laboratories</td>
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<tr>
<td>Miskovsky, N.M.</td>
<td>Pennsylvania State University</td>
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<tr>
<td>Moaged, K.L.</td>
<td>North Carolina State Univ.</td>
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<tr>
<td>Morikawa, H.</td>
<td>Nagoya Inst. of Technology</td>
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<td>Muller, A.</td>
<td>Fritz Haber Institute</td>
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<td>Nagy, D.</td>
<td>Pennsylvania State University</td>
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<td>Ng, Y.S.</td>
<td>Pennsylvania State University</td>
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<td>Nishikawa, O.</td>
<td>Tokyo Inst. of Technology</td>
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<td>Nordén, H.</td>
<td>Chalmers University</td>
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<td>University of Florida</td>
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<td>Ott, K.H.</td>
<td>University of Bonn</td>
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<td>Panitz, J.A.</td>
<td>Sandia Laboratories</td>
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<td>University Coll. North Wales</td>
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<tr>
<td>Piller, J.</td>
<td>Oregon Graduate Center</td>
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<td>Plummer, R.W.</td>
<td>Hughes Research Laboratories</td>
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<td>Rahinovich, A.</td>
<td>Pennsylvania State University</td>
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<td>Reifenberger, R.</td>
<td>University of Tokyo</td>
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<tr>
<td>Redlich, K.</td>
<td>Oxford University</td>
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<tr>
<td>Robertson, G.H.</td>
<td>University of California</td>
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<td>Röllgen, P.W.</td>
<td>University of Cambridge</td>
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<td>Rye, R.R.</td>
<td>Sandia Laboratories</td>
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<td>Saadat, A.R.</td>
<td>Pahlavi University</td>
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<tr>
<td>Sadakane, Y.</td>
<td>Tohoku University</td>
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<td>Sato, S.</td>
<td>Pennsylvania State University</td>
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<tr>
<td>Schmidt, W.A.</td>
<td>University of Tokyo</td>
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<td>Schmit, J.N.</td>
<td>Fritz Haber Institute</td>
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<td>Schwind, G.A.</td>
<td>University of Liege</td>
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<tr>
<td>Seliger, R.L.</td>
<td>Oregon Graduate Center</td>
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<tr>
<td>Shepherd, S.J.</td>
<td>Hughes Research Laboratories</td>
</tr>
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<td>Shibata, K.</td>
<td>Pennsylvania State University</td>
</tr>
<tr>
<td>Smith, G.D.W.</td>
<td>University of Tokyo</td>
</tr>
<tr>
<td>Somorjai, G.A.</td>
<td>Oxford University</td>
</tr>
<tr>
<td>Southon, M.J.</td>
<td>University of California</td>
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<tr>
<td>Sparks, M.</td>
<td>University of Cambridge</td>
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<td>Spindt, C.</td>
<td>Sandia Laboratories</td>
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<td>Sudraud, P.</td>
<td>Sri International</td>
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<tr>
<td>Sullivan, T.F.</td>
<td>Orsay University</td>
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<tr>
<td>Swanson, L.W.</td>
<td>Pennsylvania State University</td>
</tr>
<tr>
<td>Sykes, L.</td>
<td>Oregon Graduate Center</td>
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<tr>
<td>Teramoto, K.</td>
<td>University of Florida</td>
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<tr>
<td>Tsong, T.T.</td>
<td>Nagoya Inst. of Technology</td>
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<td>Tung, R.T.</td>
<td>Pennsylvania State University</td>
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<tr>
<td>Van de Walle, J.</td>
<td>University of Pennsylvania</td>
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<td>Von Engel, A.</td>
<td>C.N.R.S. University Paris-Sud</td>
</tr>
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<td>Vook, F.L.</td>
<td>Oxford University</td>
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<td>Wagner, A.</td>
<td>Sandia Laboratories</td>
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<td>Wagner, R.</td>
<td>Bell Laboratories</td>
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<td>Walck, S.</td>
<td>University of Göttingen</td>
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<td>Walko, R.J.</td>
<td>University of Florida</td>
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<tr>
<td>Waugh, A.R.</td>
<td>Sandia Laboratories</td>
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<tr>
<td>Williams, P.R.</td>
<td>University of Cambridge</td>
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<td>Yannitiell, D.W.</td>
<td>University of Oxford</td>
</tr>
<tr>
<td>Yashiro, Y.</td>
<td>Louisiana State University</td>
</tr>
<tr>
<td>Nagoya Inst. of Technology</td>
<td>University of Oxford</td>
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SCANNING TRANSMISSION ELECTRON MICROSCOPY

SCANNING TRANSMISSION ELECTRON MICROSCOPY AT NEAR-ATOMIC RESOLUTION: THE PRESENT STATE OF THE ART (Distinguished Guest Lecture)

M. Isaacson

Department of Physics, University of Chicago, Chicago, Illinois

Over the past decade there has been an increased coupling of microscopy (which gives us spatial resolution information) at spatial resolution levels smaller than the wavelength of light. The coupling is best exemplified with the transmission electron microscope. It has been pushed to the extreme such that very fine beams of electrons can be produced which are as small as 2.5 Å in diameter. We are therefore in a situation where we may expect to be able to perform chemical localization at near atomic spatial resolution from a wide variety of objects. One can presently perform spectroscopy of fast electrons transmitted through thin samples from areas approaching 20 Å in size. In addition, individual heavy atoms can be visualized on light element substrates using such atomic dimension electron beams. We will try to review the state-of-the-art of the coupling of microscopy and spectroscopy using fine electron beams and speculate on some of the potential applications in the biological and materials sciences.

BIOLOGICAL IMAGING

PROTEIN ON METAL SURFACES (Distinguished Guest Lecture)

Ivar Giaever

General Electric Company, Schenectady, New York

All the enzymes and many of the hormones in the human body are globular protein molecules. The structure of a protein molecule is closely related to its function. Some of these features will be discussed in an elementary way. Illustrations will be given of how some of the properties of protein molecules can be studied by adsorbing the molecule on metal surfaces. Finally, some problems and difficulties associated with the adsorption of protein onto field emitter tips will be mentioned.

IMAGING OF MOLECULES, PAST AND PRESENT (Invited Paper)

A. J. Melmed


Several types of microscopy have been and are being applied to the quest for detailed information about the morphology and atomic structure of molecules, with special interest in important biological molecules. These will be reviewed with emphasis on the results thus far obtained and the apparent obstacles to improved imaging capability. Particular attention will be given to the field emission (FEEM and FIM) efforts.

FIELD ION IMAGING OF DNA AND OTHER ORGANIC MOLECULES (Invited Paper)

Franklin Hutchinson

Department of Molecular Biophysics and Biochemistry, Yale University, New Haven, Connecticut 06520

Large biological molecules have been imaged on the surface of a field ion tip by surrounding them with layers of metal atoms from an evaporation source. The evaporated atoms striking the molecules to be imaged either do not stick, or are field-desorbed as the electric field is raised to visualize the surface by field ion microscopy. The image of the molecule is formed by the edges of the cavity in the adsorbed metal layer, and resolution is improved as the adsorbed layers become more ordered. Unmistakable images have been obtained of DNA and of transfer RNA molecules to a resolution of about 10 Å. Images of phthalocyanine could not be unequivocally distinguished from defects in evaporated metal layers deposited in the absence of organic molecules. The method is similar in principle to that used by Mueller and Rendulic, who embedded organic molecules in a metal layer electroplated on a field ion tip. The method has similarities to that in which an image is formed by field desorption of easily ionized molecules condensed on a tip bearing the object to be imaged. Field ion microscopy can scan a much smaller area for suitable images than is possible with transmission microscopy, and this imposes a relatively serious limitation.
LOW-FIELD DESORPTION IMAGING OF PROTEINS

J. A. Panitz‡, Ivar Giaever**

‡Sandia Laboratories, Albuquerque, New Mexico; **General Electric Research and Development Center, Schenectady, New York 12301

A novel low-field (< 1 V/R) desorption technique is described for imaging the contour of protein molecules placed on the apex of field-emitter tips. Image contrast relies on a distinct and measurable difference in the desorption field of a species of low ionization potential physisorbed on the molecule, and on the adjacent tip surface below 80 K. Preliminary results are described, using benzene to image Bovine Serum Albumin (BSA) and Immuno-Gamma-Globulin (IgG). Although the imaging procedure appears to be feasible, extreme difficulty in reproducibly placing the proteins on the tip apex, in the desired concentration, has been encountered. These difficulties together with future implications of the technique will also be discussed.

*This article sponsored by the U. S. Department of Energy, Office of Basic Energy Sciences, under Contract AT(29-1)789.


SURFACE ADSORPTION

SINGLE ATOM SELF DIFFUSION ON NICKEL

Raymond T. Tung‡, William R. Graham*†

‡Department of Physics, ††Department of Metallurgy and Materials Science, Laboratory for Research on the Structure of Matter, University of Pennsylvania, Philadelphia, Pennsylvania 19104

Past FIM studies of surface diffusion have been limited to 5d and 4d transition metal substrates. The importance of extending this powerful technique for the study of the atomic mechanisms of surface interactions to the 3d transition series metals is clearly evident. In this paper we present the results from self diffusion studies on several of the low index planes of nickel. We also include some discussion of the specimen preparation techniques which are required to produce nickel surfaces of sufficient perfection for surface diffusion studies.

**Work supported by NSF Grant DMR-75-23254 and by NSF-MRI Grant 76-00678.

BINDING SITES AND BINDING STATES OF Sn ON W

O. Nishikawa

Department of Materials Science and Engineering, Graduate School of Science and Engineering, Tokyo Institute of Technology, O-okayama, Meguro-ku, Tokyo 152 Japan

The binding sites of tin atoms on a tungsten surface are studied by superposing the image of a pseudomorphic tin layer upon the image of a substrate tungsten surface. Superposed micrographs indicate that the lateral locations of the tin atoms correspond to the locations of unoccupied tungsten lattice sites above the surface. Variations in field emission current with increasing evaporation field are also studied for the (011), (111), (112) and (114) planes. Evaporation of adatoms can be noticed by a sharp increase or decrease in the emission current at the field strength corresponding to the binding state of the adatoms. The binding states of the tin atoms on these planes are discussed.

FIELD ION MICROSCOPY OF GALLIUM ON TUNGSTEN

Robert J. Culbertson, T. Sakurai, T. T. Tsong

Physics Department, Pennsylvania State University, University Park, Pennsylvania 16802

The field evaporation of Ga from a W emitter was investigated using a magnetic sector atom probe field ion microscope. A continuous supply of Ga was provided by adjusting both tip temperature and electric field. The Ga coating of a W tip was achieved by dipping the tip in liquid Ga at 325 K in air. A clean W and cap was obtained by field-evaporating the tip at 78 K. By raising the temperature to 300 K Ga atoms diffused from the shank of the tip and field-evaporated from the tip apex at fields (2V/R) well below the evaporation field of W (5.7 V/R). The rate of Ga evaporation could be controlled over a wide range by varying either the
temperature, the electric field or both. The tip temperature was extremely critical for obtaining a steady Ga ion flux. In some cases the Ga flux was sufficiently high to yield a desorption image of the surface, implying that more than 1000 Ga ions/sec are formed at each imaged site. The desorption image has a resolution comparable to that of a field ion image. Unlike ordinary desorption imaging where the surface atoms are removed, only the continuously replenished Ga atoms are evaporated and the W tip surface remains intact.

After annealing the surface at elevated temperatures we found that the field ion image showed patchy regions of a Ga and W ordered alloy structure similar to that found in Pt electroplated W. The evaporation field of this surface was higher than that of Ga atoms migrating from the shank before annealing.

Below room temperature Ga atoms evaporate as doubly charged ions with a sharp energy distribution comparable to that of He field ionization within the forbidden zone. The binding energy of Ga atoms on W can be derived from the measurement of the energy deficit of the field evaporated Ga ions. Since the substrate W surface remains intact, the evaporation field can be maintained at a constant value. A retarding potential analyzer with 100 meV resolution of the 2000 eV primary ion energy was incorporated in the atom probe for this purpose. The measured binding energy will be presented as a function of crystallographic orientation of the W substrate and will be discussed based on the adsorption geometry determined from a field ion image.


FIELD-INDUCED DESORPTION OF BENZENE FROM TUNGSTER*

J. A. Panitz

Sandia Laboratories, Albuquerque, New Mexico 87185

A study of the field-desorption properties of benzene from clean, field-evaporated tungsten surfaces is described. Ramped-DC desorption was used to measure the desorption field of benzene at temperatures below 200 K, while imaging Atom-Probe spectroscopy was used to determine the identity of the desorbing species in physisorbed, and chemisorbed layers. Time-gated imaging was used to follow the crystallographic behavior of the predominant physisorbed species, C₆H₆, between 55 K and 200 K.

*This article sponsored by the U. S. Department of Energy, Office of Basic Energy Sciences, under Contract AT(29-1) 789.

†A U. S. Department of Energy Facility.

PROBABILITY DENSITY OF DISPLACEMENT OF ADATOM ON TUNGSTEN SURFACE

Yuji Sadakane, Ken-ichi Hirano

Department of Materials Science, Faculty of Engineering, Tohoku University, Sendai, Japan

By the FIM study of surface diffusion, it has been shown that the migration of the adatom at low temperature is restricted in the edge of the terrace. The edge of the terrace has two kinds of role in the migration of the adatom; reflecting or adsorbing boundary. The role of the edge depends on temperature and time of diffusion treatment. When the distance between the edges of the terrace is a, we define \( W_R(d,a) \), the probability that the adatom makes a displacement d after it makes N jumps by the following equation.

\[
W_N(d,a) = (1-k) W_N^R(d,a) + kW_N^A(d,a)
\]

where the symbol R denotes the case that the adatom is reflected at the edge of the terrace and A the case that the adatom is adsorbed by the edge of the terrace, and k is the adsorbing factor (0 < k < 1), which depends on temperature and time of diffusion treatment. The value of k can be estimated by comparing the experimental values of \( W_N^R(d,a) \) and \( W_N^A(d,a) \) with the theoretical values of them. In the present work, the surface self-diffusion on the W (431) plane has been investigated theoretically and by FIM observation.
ATMING ERROR IN METALLURGICAL SPECIMENS

Jörg Piller

Institute for Metal Physics and SFB 126, University of Göttingen, Hospitalstrasse 12, D-3400 Göttingen, W. Germany

The combined FIM-Atomprobe and Field Desorption Microscope has been applied for the determination of the aiming error in pure metals and in metallurgical alloys.

Previously it has been shown by several authors that there could be considerable differences between the flight paths of the desorbed ions and their related field ions. This is evident, for instance, by the fine detailed structures found in multi-layer field desorption micrographs. On the contrary, it has been demonstrated that under distinct conditions it is possible to aim with the atom probe on single solute or adsorbed atoms which are brightly imaged with a yield near the detection efficiency of the ion detector.

Now it is of interest how the desorbed ions of precipitates are distributed with respect to their field ion image, especially if the precipitates protrude from or are recessed into the specimen surface.

From the desorption images which are gated for the different components of the precipitates and from the field ion images, the extension of the precipitates is measured. The influence of the distorted field above protruding precipitates on the targets of the desorbed ions is compared with those of ions of high indexed planes in pure metals.

These results are related with atom probe measurements where the probe hole is aimed on or nearby the field ion image of the precipitate.


QUANTITATIVE DETERMINATION OF Alloy ELEMENT PARTITIONING IN PEARLITIC STEELS BY ATOM PROBE ANALYSIS

M. K. Miller, P. R. Williams, P. A. Beaven, G. D. W. Smith

Department of Metallurgy and Science of Materials, University of Oxford, Parks Road, Oxford OX1 3PH, England

Previous observations of alloy element redistribution processes in patented and cold drawn pearlitic steel wires\(^1\),\(^2\) have been extended. Quantitative measurements have been made of solute concentration profiles across the ferrite-cementite interface in as-transformed pearlitic steels containing the alloy elements Mn, Cr, and Si.

It has been demonstrated that extensive substitutional solute partitioning occurs over the range of transformation temperatures studied (down to 550 °C).

This result is at variance with previous (lower spatial resolution) electron microprobe analyses, and provides a new insight into the nature of the physical processes occurring during the pearlite transformation. In particular, the existence of a highly effective short-circuit diffusion pathway for substitutional elements in the austenite-pearlite interface would now appear to be clearly established.


A STUDY OF ORDERED Ni\(_4\)Mo SURFACE ON AN ATOMIC SCALE

G. H. Robertson, T. Sakurai, T. T. Tsong

Physics Department, Pennsylvania State University, University Park, Pennsylvania 16802

Field ion microscope (FIM) studies of ordered alloys have great importance because the hemispherical shape of FIM emitters provides a variety of superstructures. The geometrical structure and possible reconstructions can be observed directly on an atomic level.

Previous FIM studies of ordered alloys have found that one of the species is usually not imaged.

We have attempted a new study of an ordered alloy, Ni\(_4\)Mo. Its bulk structure is well established [body-centered tetragonal (BCT) \(c^4/a = 0.6231\)]. This BCT structure contains eight Ni and two Mo atoms in the unit cell, i.e., four Ni and one Mo atoms per Bravais lattice point. The structure may be described as five interpenetrating BCT sublattices—one having a single Mo atom per lattice point and the other four with a
single Ni atom per lattice point. Each sublattice is translated by $1/5 \langle 110 \rangle$ with respect to a neighboring sublattice. Because of this complexity, some net planes described by the same Miller index number [for example, (110) and (011) or (211) and (121)] have different symmetry and structural properties enhancing our interest in this system.

In our FIM study, using a channel plate image intensifier and liquid $H_2$ cooling, we have succeeded for the first time in the observation of Ni and $^2$Mo layers with atomic resolution. While slowly evaporating the (011) surface layer by layer, we observed only two pure Ni layers below each pure Mo layer rather than the four expected from the alloy structure. It also appeared that the number density of a Ni layer is approximately twice that of a Mo layer. This anomaly was not observed on the (110) plane where we observed four pure Ni layers below each pure Mo layer. The basic structural differences between these two planes is the number density and the spacing, $d$, of adjacent layers [$d = 0.8099 \, \text{Å}$ for (110) and $d = 0.6057 \, \text{Å}$ for (011)].

The ready interpretation of this anomaly for the (011) plane is that two adjacent Ni layers reconstruct to form a single layer when the top Mo layer is field-evaporated. Using the atomic resolution of Ni layers we will try to determine the position of Ni atoms with respect to the Mo atoms for a number of net planes, such as (111), (121), (213) and (123). The field emission images from fundamental planes and superlattice planes will be presented to show a unique potential of using this system to study the electronic structure of a superlattice. It may be possible to obtain the density of states of the superlattice by field electron spectroscopy.

1. B. C. LeFevre, H. Grenga, B. Ralph, Phil. Mag. 18, 1193 (1968).

**Analysis of the Electric Field Distribution Inside a Field Ion Microscope by the Finite Element Method**

Gary S. Gipson, D. W. Yannitell, H. C. Eaton

**Department of Mechanical Engineering, Louisiana State University, Baton Rouge, Louisiana 70803**

The electric field distribution inside a field ion microscope is determined by means of the numerical method of finite elements. Using this technique, realistic geometries are considered. Analysis of such models has not been possible by conventional analytical methods because of the intractable nature of the solutions. Features included in the numerical model are microscope walls, a flat screen, and for the first time a cold finger, a dielectric, a specimen holder with shape, and an accelerating shield. It is found that neglecting the tedious geometry can yield inaccurate results. The accelerating shield, located about $10^5$ tip diameters from the specimen tip, is shown to provide a substantial enhancement of the electric field at the specimen apex. This can result in significant changes in the specimen stress distribution and may alter the ion trajectories as well. Also studied is the effect of the specimen shank angle on the electric field. It is found that a variation from $0^\circ$ to $9^\circ$ in emitter shank angle results in approximately a 40% reduction in the field strength near the apex.

**Appearance Potential Measurements on Singly and Doubly Charged Field-Evaporated and Field-Ionized Metal Ions**

Norbert Ernst

Fritz-Haber-Institut der Max-Planck-Gesellschaft, Faradayweg 4-6, 1000 Berlin 33

Experimental appearance potential values $\alpha \sigma_n = e \psi_n + \phi_R$, $n=1, 2$ as derived from the onset voltage $\delta$ of integral energy distribution of $n$-fold charged metal ions are compared with theoretical values $\alpha \sigma_n$ calculated from the image hump model ($e$ elemental charge, $\phi_R$ work-function retarder electrode), $\phi_R$, $\alpha \sigma_n$ is approximately given by:

$$\alpha \sigma_n = \frac{1}{n} \Delta E_n^c (F) + \phi_R$$

where

$$\Delta E_n^c (F) = \sqrt{\frac{3e^3}{4\pi\varepsilon_0}} \sqrt{F}$$

is the critical energy deficit of a $n$-fold charged ion at the emitter surface (work function $\phi_R$) according to the Schottky hump condition ($\varepsilon_0$ diel. const. $8.85 \times 10^{-12} \, \text{As/Vm}$). When the electric field strength $F$ is high enough field evaporation occurs with nearly zero thermal activation energy. $\alpha \sigma_n$ then becomes:

$$\alpha \sigma_n = \frac{1}{n} (\frac{1}{n} I_n + \lambda)$$
Abstracts of 25th International Field Emission Symposium

Experimental results. With copper, the image hump model of field evaporation is verified. $\text{AP}_n$ values of other species mainly reflect field evaporation with nearly zero activation energy (emitter temperatures below 300 K).

<table>
<thead>
<tr>
<th>n species</th>
<th>$\left(\sum_{i=1}^{n} E_i + \lambda\right) \text{[eV]}$</th>
<th>$\text{AP}_n \text{[eV]}$</th>
<th>$F \text{[V/A]}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Cu$^+$</td>
<td>11.0</td>
<td>10.9</td>
<td>2.9</td>
</tr>
<tr>
<td>1 Cu$^+$</td>
<td>9.3</td>
<td>9.3</td>
<td>1.6</td>
</tr>
<tr>
<td>1 Be$^+$</td>
<td>12.6</td>
<td>12.5</td>
<td>5.1</td>
</tr>
<tr>
<td>2 Be$^{++}$</td>
<td>15.5</td>
<td>15.3</td>
<td>4.5</td>
</tr>
<tr>
<td>2 Ni$^{++}$</td>
<td>15.0</td>
<td>14.8</td>
<td>3.4</td>
</tr>
<tr>
<td>2 Fe$^{++}$</td>
<td>14.1</td>
<td>14.0</td>
<td>3.4</td>
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</table>

Calculated and Literature Data

<table>
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<tr>
<th>n species</th>
<th>$\left(\sum_{i=1}^{n} E_i + \lambda\right) \text{[eV]}$</th>
<th>$\text{AP}_n \text{[eV]}$</th>
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<td>14.1</td>
<td>14.0</td>
<td>3.4</td>
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</table>

Experimental Results

<table>
<thead>
<tr>
<th>n species</th>
<th>$\text{AP}_n \text{[eV]}$</th>
<th>$F \text{[V/A]}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Cu$^+$</td>
<td>10.9 ± 0.2</td>
<td>2.9 ± 0.3</td>
</tr>
<tr>
<td>1 Cu$^+$</td>
<td>9.3 ± 0.2</td>
<td>1.6 ± 0.2</td>
</tr>
<tr>
<td>1 Be$^+$</td>
<td>12.5 + 0.1 - 0.3</td>
<td>unknown</td>
</tr>
<tr>
<td>2 Be$^{++}$</td>
<td>15.5 + 0.1 - 0.2</td>
<td>unknown</td>
</tr>
<tr>
<td>2 Ni$^{++}$</td>
<td>15.0 + 0.1 - 0.2</td>
<td>unknown</td>
</tr>
<tr>
<td>2 Fe$^{++}$</td>
<td>13.9 ± 0.1</td>
<td>unknown</td>
</tr>
</tbody>
</table>

Table 1. $\text{AP}_n$ values in brackets are estimated onsets of second high energy distributions observed in the spectra of doubly charged ions.

Raising the emitter temperature above 900 K leads to thermal evaporation of metal atoms with subsequent field ionization, $F_I$, in the gas phase. The first ionization potentials are measured ($\text{AP}_1 < I_1$) corresponding to the main onset of energy distributions. A second high energy distribution with onset down to 5 eV below $I_1$ (Be$^+$) is obviously generated by $F_I$ of electron impact excited atoms.


A COMBINED CONVENTIONAL AND IMAGING ATOM-PROBE AND ITS APPLICATION TO SEgregation Studies

A. R. Waugh, P. P. Mills, M. J. Southon

Department of Metallurgy and Materials Science, University of Cambridge, Pembroke Street, Cambridge CB2 3QZ, United Kingdom

The imaging atom-probe which was constructed in 1975 for metallurgical applications has been modified and updated to combine the elemental mapping capability of the IAP with the quantitative point-analysis capability of the conventional atom-probe. As before, a single 50 mm flat channel-plate is used to detect the gated desorption image with a flight-distance of 125 mm. A second 50 mm channel-plate has now been fitted close to the specimen, which can be tilted through 140° on a specially built stage. The second plate is used to display the entire field-ion image of the specimen and also incorporates a 2 mm diameter probe-hole which defines the entrance aperture of a conventional atom-probe, with provision for energy compensation. A 25 mm chevron channel-plate is used as the detector.

As a result of these modifications it is possible to obtain both quantitative mass spectra from small areas of the specimen and also time-gated desorption images from a large area. Data are collected rapidly as an evaporation-pulse generator has been built which uses a mercury-wetted reed relay operated at 250 Hz. Jitter-free operation at this high rate is made possible by using a solid-state high-voltage switch in place of the conventional charging resistor: output-pulse amplitude is adjustable from 300 to 4000 volts. This type of pulser has been added successfully to all three atom-probes currently in use in Cambridge.

Our prime area of research at present is in segregation: the unique ability of the IAP to detect segregation at grain boundaries with high spatial resolution and single-atom sensitivity has been applied further along the lines described previously. Further observations have been made of oxygen at grain boundaries in molybdenum, including a grain-boundary node, of carbon in iron, and of tin at and near grain-boundaries in iron. The latter system is of particular interest in that significant quantities of a metallic dimer, Sn$_2^{3+}$, are observed, as well as Sn$^+$ and Sn$^{2+}$.
DETERMINATION OF ATOMIC JUMP LENGTH DISTRIBUTIONS FOR SURFACE MIGRATION OF SINGLE ADATOMS

P. L. Cowan*, T. T. Tsong**

*Bell Laboratories, Murray Hill, New Jersey 07974; **Department of Physics Pennsylvania State University, University Park, Pennsylvania 16802

Surface migration of single adatoms on a perfect crystallographic plane occurs by a random sequence of atomic jumps. However, atomic jumps are not necessarily restricted to nearest neighbor sites, as is commonly assumed. Analysis of FIM observations of migrating adatoms is uniquely capable of yielding the distribution of jump lengths and directions. Existing data indicates that W adatoms on W(110) jump predominantly along <111> directions in the plane, but can jump various distances in these directions.


A STUDY OF CHEMISORBED NITROGEN AND OXYGEN ON GROWTH OF THIN GOLD FILMS ON TUNGSTEN

C. Patel

University College of North Wales, Department of Electronic Engineering Science, Dean Street, Bangor, Gwynedd, N. Wales, United Kingdom

When condensed on tungsten in ultrahigh vacuum, metals are known to form monolayer and multilayer films. The presence of sub-monolayer amounts of preadsorbed gas can, however, prevent such films from forming.

In an attempt to establish the roles of chemisorbed gases, detailed studies have been made of the effects of small quantities of nitrogen and oxygen as contaminant gases on the structure of thin gold film on tungsten by observing the change in the work-function with coverage and temperature using probe-hole and simple field emission microscopy.

Adsorption of nitrogen on tungsten followed by gold shows notable changes in the work-function when compared with the 'clean' gold behavior and consequently results in crystallite growth at a gold coverage greater than 28. Heating the composite layers at a temperature T > 800 K causes crystallites to dissolve and gold reverts to 'clean' behavior.

Oxygen adsorption on a gold-bearing tungsten surface produces a decrease in $\phi$ of $\approx -0.45$ eV for spreading temperatures of 600-750 K. This decrease in $\phi$ seems unusual since the oxygen sticking probability on gold is very low for the temperature used. Because the oxygen attacks the tungsten substrate more severely than nitrogen, the gold atoms aggregate much more readily into crystallites than in the case of nitrogen.

The stepwise heating of the following systems W-Au, W-N2, W-N2-Au, W-O2, W-Au-O2-Au, W-O2-Au, have been examined for the desorption characteristics of the chemisorbed gases on morphology of gold overlayers. In both nitrogen and oxygen cases the order of their adsorption plays an important part, and this is found to be noticeable when the stepwise heating of the following systems was performed: W-O2-Au Case 1 and W-Au-O2-Au Case 2. In case 1, $\phi$ increases very sharply between T = 500-750 K and becomes steady in the temperature region T = 900-1400 K. Case 2 shows the opposite effect in the T = 500-750 K temperature region. This behavior is thought to be due to the inversion of dipoles, and will be described in detail.

Co-adsorption of (N2-Au) has been studied on the W(100), (111) and (211) planes. Results will be described and compared with known data of the (N2-W) and (W-Au) system on these planes.

DIFFUSION STUDIES OF ADSORBATES ON TUNGSTEN (110)

Jiann-Ruey Chen, Robert Gomer

Departments of Physics and Chemistry and the James Franck Institute, The University of Chicago, Chicago, Illinois 60637

The correlation of functions of field emission current fluctuations from the (110) plane of tungsten field emitters covered with submonolayer amounts of oxygen, carbon monoxide and xenon, have been measured. At temperatures above the onset of surface diffusion, the experimental correlation functions agree with a theoretical model based on single particle diffu-
sion, and yield diffusion coefficients and activation energies of diffusion on this plane. For oxygen, activation energy of diffusion rises from 14 k cal/mole for O/W < 0.2 to 22 k cal/mole for O/W = 0.56. For virgin CO, no diffusion could be unequivocally established at any coverages, although a weak signal set in at 250°K. For beta CO, diffusion with an activation energy 23 k cal/mole is found, suggesting O is the observed species diffusing. For xenon, an activation energy of ~ 3 k cal/mole was found at θ = 0.5 and θ = 0.9. At high temperatures the decay of the correlation function is slower than predicted by theory. It is suggested that this results from dynamical correlations, or multi-particle density fluctuations, which can occur when several adsorbate particles are simultaneously in motion. At low temperatures, exponential decay of the correlation function is observed and explained as a prediffusive flip-flop of adsorbate particles between binding locations. The temperature-dependence of the mean square fluctuations in the diffusive regime is discussed in terms of adsorbate-adsorbate interactions.


FIELD EMISSION AND ION SOURCES

A BASIC WAVE-MECHANICAL THEORY OF FIELD-ION ENERGY DISTRIBUTIONS

Richard G. Forbes

Department of Physics, University of Aston, Gosta Green, Birmingham, United Kingdom

This paper presents the outline of a new wave-mechanical theory of field ionization and field-ion energy distributions. The theory is a generalization of the approach used by Gomer and Swanson, and avoids the use of the Landau-Zener approximation. The theory may be regarded as a direct fermion analog to the theory of radiative emission as developed by Dirac.

The essential feature of the new theory is that it avoids the "double infinity of final states" problem by enclosing the departing ion in a hypothetical one-dimensional sloping-sided box for which the wave-functions can be determined. The rest of the analysis then proceeds by analogy with the well established theory of Molecular Spectroscopy.

Currently, the theory is in the formal stage of development, but the following results are already clear:

1. A proof can be given that the usual quasi-classical treatment of field-ion imaging should be a valid first approximation, if the electron transition rate-constant is given by an expression involving an electronic overlap integral.

2. The usual quasi-classical treatments of energy distributions should be valid approximations, with the following exceptions:

(a) The shape of the high-energy edge of the main peak;
(b) Discussion of surface-plasmon-assisted field ionization.

In the exceptional cases the influence of overlap factors between vibrational wave-functions needs to be taken into account. For surface-plasmon creation the presence of an additional term in the expression for the energy distribution could prove decisive in rejecting this mechanism of the Jason effect on purely theoretical grounds.

These results are not exactly unexpected. However, the author believes that the present treatment of field ionization, which has long been recognized as a "difficult" subject, has been able to avoid logical errors that appear in previous treatments. Very few, if any, previous treatments are strictly compatible with the basic axioms of quantum mechanics.


FIELD EMISSION SOURCES FOR LARGE AREA DISPLAYS

Jules D. Levine

RCA Laboratories, Princeton, New Jersey 08540

Component and systems aspects of field emitters as electron sources have been studied for large area displays: 10^6 display elements, 10^4 cm^2 display area, and a design cost for under $100. We first investigated UO_2/W eutectics provided by Professor A. T. Chapman. Later, a new field emitter technology was developed using copper and glass fused sheets. This was more suited to mass production. By mechanical or photolithographic means, Cu tips were formed 10 mils apart on a square array. A tip, when appropriately treated and fused with a glass insulator and a copper extractor, yielded 10-100 µA at 400-1000 V. The design value was
1-10 \text{\textmu}A at 200 V, for 200-ft lamberts brightness. Proximity focusing was used. On occasion, beam extraction efficiencies of 100\% were obtained.

Systems studies showed the need for an inexpensive feedback sensor and circuit for each of the 10\textsuperscript{6} elements, which would sample the beam current and also correct for display brightness inhomogeneities due to the erratic nature of the field emission source. Such a sampling circuit was built and tested successfully.\textsuperscript{1}


**CHARACTERIZATION OF THE ION EMISSION FROM A TAYLOR CONE OF LIQUID GOLD**

P. Sudraud, J. Van de Walle, C. Colliex, P. Trebbia

Laboratoire de Physique des Solides, Associé au C.N.R.S., Bât. 510, Université Paris-Sud, 91405 Orsay, France

Various ion emission modes issued from a metallic gold surface under the combined effect of a strong electric field and a high temperature have been investigated.

At a temperature higher than the melting point, and under an electric field greater than a critical value, the free surface of a liquid metal is deformed into a Taylor cone shape, the extremity of which has been visualized in situ in an electron microscope. From the area a few microns large located at the apex of this cone, rather strong positive ion currents can be delivered. Of the order of 1 mA, they are quite stable and can last for hours if an appropriate design allows a permanent flow of matter towards this emitting area.

The characteristics of the beam have been analyzed with the help of a double-focusing mass spectrometer. The mass distribution contains a large variety of ionic species ranging from doubly ionized Au\textsuperscript{++} to molecular Au\textsubscript{n} (fragments with \( n < 7 \) have been detected). The main contribution due to Au\textsuperscript{+} represents about 70\% of the total emission. Superposed on the general decrease with \( n \) of the molecular ions intensity, slight alternances can be detected; species with odd \( n \) are relatively more abundant than those with even \( n \). This is in good agreement with the theoretical predictions of Joyes concerning the stability of noble metal clusters.\textsuperscript{1}

The energy distribution has been measured for all these emission products. The two main ionic species Au\textsuperscript{++} and Au\textsuperscript{+} exhibit a single large peak. Of the order of 45 eV for Au\textsuperscript{++}, the width of this peak is increased to about 70 eV for Au\textsuperscript{+}, and is followed in this case by a long energy deficit tail. The molecular ions also display a clear secondary maximum: its position gradually shifts towards larger energy losses and its relative importance increases regularly with \( n \). These experimental data provide some interesting basis for the understanding of the emission mechanisms in an EHD liquid metal ion source. The contribution of the space charge effects and of the collision processes in a dense plasma extending over a few hundred Ångströms above the liquid surface will be discussed.

Finally, the transition between the low temperature ion emission modes from the solid and the high temperature ones from the liquid has been carefully studied when one raises the temperature across the melting point. Intermediate regimes including the geometrical faceting of the metal surface or the growth of microneedles have been recognized. All these results provide a general frame to classify the different behaviors of a metal surface under these extreme temperature and field environments.


\*Research performed with the help of a grant DRME 76/1184.

**FIELD EMISSION FROM A LIQUID METAL**

L. W. Swanson, G. A. Schwind

Oregon Graduate Center, Beaverton, Oregon 97005

The characteristics of a novel repetitive pulsed electron source employing a liquid Ga/12\% In cathode have been examined. Repetitive current pulses as large as 250 A with an applied voltage of only 10 to 12 kV have been observed. The mechanism involves the formation of a field-stabilized cone of the liquid cathode which forms a sufficiently small apex radius that a regenerative field electron current initiates an explosive emission process of the type described in detail for solid state emitters.\textsuperscript{1–3} If the applied
voltage is held just below threshold, a 100 V by 35 nsec pulse voltage is sufficient to initiate the high current pulse.

Several important differences between the liquid and solid phase explosive emission electron sources have been revealed. First, after the vaporization of a small amount of material from the cone apex during the pulse, the liquid phase source self-heals thereby yielding a repetitive pulse mode operation with a constant applied voltage. Secondly, there is no limit to the life; i.e., the number of pulses, attainable from the liquid emitter. Third, the current pulse rise time and duration are shorter for the liquid metal source. A remarkable feature of the liquid metal electron source when operated in the free running repetitive mode is the lack of measurable time jitter, < 0.1 nsec, between the onset of successive pulses and their uniform amplitude over the rep rate range 40 to 8,000 pps.

With the reverse polarity, this source becomes an intense ion emitter9 '4 Used in a FIM, embodiment patterns with a certain degree of structure can be observed. Mechanisms by which field evaporation from liquid metals occur will be discussed.

GALLIUM FIELD ION EMISSION FROM LIQUID POINT ANODES

G. L. Mair, A. von Engel

A brief summary of the principal processes controlling emission is given followed by an investigation of the two components of the collector current and its superimposed pulses when zero, retarding and accelerating potentials are applied to the collector. The effect of secondary electrons emitted by ion impact on the collector is analyzed as well as the results of suppressing the secondaries by deflection in a moderate electric or magnetic field. Finally observations of the luminosity at the collector surface and near the apex of the wetted point anode, of the formation and life of protrusions and of the flow of neutral atomic particles and droplets are discussed.

SUBMICRON IMAGING OF A LIQUID METAL GALLIUM SOURCE*

R. L. Seliger

Hughes Research Laboratories, Malibu, California 90265

*Abstract not available at time of publication.

SURFACE CHEMISTRY

CATALYTIC CHEMISTRY: A SURFACE SCIENCE VIEWPOINT (Distinguished Guest Lecture)

G. A. Somorjai

Department of Chemistry, University of California, Berkeley, California 94720

The past ten years have seen the development of techniques, the combination of which can be utilized to tackle major problems of heterogeneous catalysis on the molecular scale. Most of these new techniques need only small surface areas (~cm²), but often require high vacuum sample environments. New methods of catalytic research have been developed that use (1) small area samples for structure, composition, and kinetic studies, and (2) mate ultra-high vacuum and high-pressure reaction conditions.

Four examples of studies involving single crystal surfaces of small area will be presented using three types of surfaces: flat (111) orientation, stepped, and kinked surfaces.

A. Determination of the surface structure of adsorbed C₂H₂ and C₂H₄ on the Pt (111) surface for a combination of low-energy electron diffraction (LEED) and high resolution electron energy loss techniques.

B. The mechanisms of H₂-D₂ exchange on stepped platinum crystal surfaces by reactive molecular beam-surface scattering.

C. Effect of oxygen on rates of hydrogenation and dehydrogenation of cyclohexene on kinked platinum crystal surface.

D. Effects of additives C₂H₄, C₃H₆ and potassium on rates and product descriptions during hydrogenation of carbon monoxide over the iron (111) crystal face.
FIELD IONIZATION, FIELD EVAPORATION AND FIELD DESORPTION OF SILVER IONS: A COMPARISON OF APPEARANCE ENERGIES

J. H. Block, R.-G. Abitz, M. Domke, E. Hummel

Fritz-Haber-Institut der Max-Planck-Gesellschaft, Faradayweg 4-6, 1 Berlin 33

Mass spectrometric experiments are performed and integral energy distributions measured for Ag+ ions of various origin. (1) Silver vapor from a Knudsen cell is field-ionized and yields appearance energies (AP) in accordance with gas phase ionization: AP is independent of field strength; AP values represent ionization potentials diminished by kinetic energy contributions of neutrals with nearly Maxwellian distribution. (2) The energy of ions field-evaporated from silver layers at a tungsten emitter (Ag-Ag- or Ag-W-surface bonds) is affected by bond energies. The energy deficits are field- and temperature-dependent. The field dependence is explained in terms of the "charge exchange" or "image hump" model. The temperature dependence results from competing thermal activation, which can be independently determined from rate measurements. With increasing temperature Ag+ ions can pass successively barriers with increasing height at reduced field strength, resulting in reduced energy losses. (3) Field desorption of Ag+ ions performed in a layer of Ag2S at a silver field emitter differs remarkably from usual field evaporation of silver: Although field evaporation occurs at drastically reduced field strength, energy losses rise by 2 eV. This behavior is caused by the shape of the potential wall in front of the emitter. The Madelung potential at the ion-crystal surface shows much steeper variations with distance than the image potential in front of a metal surface does.

FIELD ION APPEARANCE ENERGIES OF CO+ IONS FORMED AT NOBLE METAL SURFACES

W. A. Schmidt, O. Frank

Fritz-Haber-Institut der Max-Planck-Gesellschaft, Faradayweg 4-6, 1 Berlin 33

From measured integral energy distributions of CO+ ions at 80 K tip temperature on surfaces of Cu, Ag and Au, respectively, field ion appearance energies were evaluated and found up to .5 eV higher than the value expected from usual field ionization. Under certain field conditions with gold tips two superimposed energy distributions have been observed. Mathematical evaluation resulted in two appearance energies, (i) the already mentioned high value, and (ii) the value usually expected for CO+. Pronounced differences of the high appearance energies within the group of noble metals were not observed. There were also no differences in the appearance energies if the surfaces were cleaned before ionization or used after prolonged influence of residual gases. Furthermore, no such shifts of the appearance energies were observed for other reactive gases, for example, oxygen.

Although as preliminary interpretation, it is thought that the CO molecule is being ionized in the presence of chemisorbed CO. The ionization takes place then in a certain spatial distance from the surface, and the tunneling electron sees a second inner potential barrier which is easier to overcome than to tunnel through. Other interpretations fail because of the independence of the high appearance energy value from the metal.

FIELD DESORPTION MASS SPECTROMETRY

PROCESSES OF ION FORMATION IN FIELD DESORPTION MASS SPECTROMETRY


Institute of Physical Chemistry, University of Bonn, 5300 Bonn, West Germany

From conventional activated emitters alone no reliable information on the ionization conditions, i.e., the effect of field strength and temperature, on the ion formation can be obtained since the adsorption of substances on surfaces of largely different field strengths (on top, on the shank or in between the field-enhancing microneedles) cannot be sufficiently controlled. However, by using smooth wires and wires with microneedles of different length as field anodes it was possible to differentiate between ion formation either by field ionization or by electrochemical processes in condensed sample layers which are almost field-independent. However, even in the latter case a high external field > 10? V m-1 is needed to induce a charge separation and dissolution of ions. A thermionic emission of metal ions is observed above 800 K. The carbon needles are particularly active...
emission centers suggesting the storage of metal atoms from adsorbed metal salts in the needles.

**APPEARANCE ENERGIES OF FIELD-IONIZED AND FIELD-DESORBED POLYATOMIC SULFUR MOLECULES**

J. H. Block, R.-G. Abitz  
Fritz-Haber-Institut der Max-Planck-Gesellschaft, Faradayweg 4-6, 1 Berlin 33

Sulfur molecules $S_x$ ($2 < x < 8$) are either produced in an electrochemical cell and evaporated onto a tungsten field emitter or are formed by a surface reaction of impinging $S_2$ molecules in a physisorbed layer of the emitter at about 300 K. Field ionization of the $S_x$ vapor and field desorption of $S_x$ surface reaction products are investigated in a quadrupole mass spectrometer and analyzed with regard to mass-to-charge ratio, ion intensity, and integral energy distribution.

For vapor molecules appearance energies (AP) have been expected to reflect ionization energies (I). Experimental values are:

<table>
<thead>
<tr>
<th></th>
<th>AP</th>
<th>I</th>
</tr>
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<tbody>
<tr>
<td>$S_2$</td>
<td>9.33 ± 0.1</td>
<td>9.36 ± 0.05</td>
</tr>
<tr>
<td>$S_5$</td>
<td>8.11 ± 0.15</td>
<td>8.60 ± 0.05</td>
</tr>
<tr>
<td>$S_6$</td>
<td>8.94 ± 0.1</td>
<td>9.00 ± 0.03</td>
</tr>
<tr>
<td>$S_7$</td>
<td>8.50 ± 0.1</td>
<td>8.67 ± 0.03</td>
</tr>
<tr>
<td>$S_8$</td>
<td>8.97 ± 0.1</td>
<td>9.04 ± 0.03</td>
</tr>
</tbody>
</table>

In particular for $S_5$ and $S_7$, deviations are higher than experimental errors. In comparison with vapor molecules, $S_x$ molecules formed by surface reactions display energy losses which are more than 1 eV too small (even at postulated negligible adsorption energy). This deviation is nearly identical for all measured $S_x$ and independent of individual ionization energies. This result indicates that field desorption from the condensed layer involves either adiabatic reaction steps or ionization out of intermediate excited reaction states.

**QUANTITATIVE APPLICATIONS OF FIELD DESORPTION MASS SPECTROMETRY**

Douglas F. Barofsky, Elisabeth Barofsky, Lorne M. Isabelle  
Oregon Graduate Center, 19600 N.W. Walker Road, Beaverton, Oregon 97005

Field desorption mass spectrometry (FDMS) has experienced relatively rapid growth during the last decade in its applications to organic chemistry. However, the use of FDMS has been generally restricted to qualitative analyses of specific compounds or mixtures of compounds. The earliest considerations given to quantitative applications of FDMS were directed towards the problem of defining a suitable means for measuring sensitivity. More recent efforts have been directed toward applying FDMS to quantitative problems in mixture analysis and isotope ratio determinations. FDMS shares with other forms of mass spectrometry the very desirable features of high sensitivity coupled with high specificity. These characteristics are often enhanced by the relatively high molecular ion intensities generally observed in FD mass spectra.

This laboratory has developed an interest in applying FDMS to the quantitative analysis of polynuclear aromatic hydrocarbons contained in high pressure liquid chromatographic fractions.

The present paper presents the results of our efforts, both past and present, in investigating sample handling, desorption rate, focusing, and detection made in conjunction with the precision, accuracy and minimum detectable limit of quantitative FDMS analyses.

**IONIZATION BY ALKALI ION ATTACHMENT ON THE SURFACE OF ELECTROLYTIC SOLUTIONS**

K. H. Ott, F. W. Röllgen  
Institute of Physical Chemistry, University of Bonn, 5300 Bonn, West Germany

Electrolytic solutions prepared by mixing concentrated aqueous salt solutions with low vapor pressure organic compounds were found to be particularly suited for surface ionization of unpolar or weakly polar molecules by alkali ion attachment. To this end the mixtures are deposited on field anodes, i.e., 10 μm W wires. The attachment reaction results from a field-induced and thermally activated charging of the layer with loosely bound alkali ions which are captured by molecules striking the surface.

Optimum results were obtained with emulsifying agents such as polyoxyethyl- enes giving (M + alkali) ions already at room temperature. This is assigned to a membrane effect, i.e., the organic molecules surrounding small $H_2O$-salt droplets are permeable for ions in a high external field.

FIELD EMISSION

THERMALLY ENHANCED FIELD EMISSION FROM A LASER-ILLUMINATED TUNGSTEN TIP

R. Reifenberger†, M. J. G. Lee

Department of Physics, University of Toronto, Toronto, Ontario M5S 1A7, Canada

In order to interpret quantitatively the total energy distribution observed in laser-induced field emission it is necessary to know the temperature of the illuminated field emission tip. We have investigated the emission of electrons from a tungsten field emitter illuminated by the focused beam of a laser operating in the visible region of the spectrum. Data obtained for emission from W(111) are found to be in reasonable agreement with a simple free electron model, and from the data we can estimate the temperature rise of the tip. These estimates are also consistent with a first principles calculation of the temperature rise, based on an experimental investigation of the intensity distribution in the focused laser beam and of the shape of the field emission tip. Data obtained for emission from W(510) do not yield a consistent estimate of the temperature rise of the field emission tip. Some possible explanations for the anomalous emission from W(510) are discussed.

*Supported by the National Research Council of Canada
†Present address: Purdue University, Department of Physics, West Lafayette, Indiana 47907

FIELD EMISSION SPECTROSCOPY OF BISMUTH ON W(100)

J. P. Jones

University of College of North Wales, School of Electronic Engineering Science, Dean St., Bangor, Gwynedd, LL57 1UT, United Kingdom

An examination of the FEED spectra from lead and iron on W(100) led to the proposal that features in the enhancement curves could be identified with energy levels in the isolated atom. To further test this proposal bismuth was chosen as an adsorbate because the atom has a relatively simple energy level structure in the small energy range which is accessible to our retarding field spectrometer.

The initial reduction in $\phi(100)$ by adsorbed bismuth is accompanied by a reduction in the surface state resonance and a weak feature emerges centered at 0.4 eV below the Fermi level. As the coverage approaches one monolayer a stronger resonance emerges at 0.4 eV and is substantially independent of coverage over a range of at least one monolayer. At coverages exceeding two or three monolayers a resonance appears at ~0.6 eV.


A NOVEL METHOD OF PROBE HOLE CURRENT MEASUREMENT

Rocco DiFoggio, Robert Gomer

Departments of Physics and Chemistry and the James Franck Institute, The University of Chicago, Chicago, Illinois 60637

Upon passing through the probe hole, field-emitted electrons are post-accelerated to strike a phosphor-coated glass surface thus producing light which passes through the glass and is detected by a photomultiplier tube. The phosphor is backed by a 500 μ thick evaporated aluminum film to prevent stray light from reaching the PM tube. The advantages of this external detection system over a Channeltron include simpler field emission tube construction, easy replacement of the detection system and a higher permissible bakeout temperature of the vacuum chamber. The overall gain and signal-to-noise are comparable or better than with a Channeltron. Work function measurements done in this way are in good agreement with those done with a Channeltron. As for the choice of a phosphor, an inorganic fast-decay (~400 nanosecond) phosphor such as P47 (Cerium-activated Yttrium Silicate) is bakeable to 450°C, suitable for UHV, and can respond to high frequency fluctuations in field emission currents. Its light output peaks around 3900 μ, which makes it well suited for use with a standard S-II photocathode.

FIELD EMISSION FROM THE SUPERLATTICE PLANES OF ORDERED Ni$_4$W

Joaquin Lira-Olivares

INMETAL and Department of Materials Sciences, Universidad Simón Bolívar, Caracas, Venezuela
We utilized the layered structure presented by the (011), (101) and (112) planes of Ni₄W phase. Such a structure provided similar conditions to those encountered in adsorption experiments (a substrate with adsorbed layers).

Changes in electron current emission were measured after evaporating atomic monolayers from the (011) plane of ordered Ni₄W specimens. Some of the changes were found to be of the same order of magnitude as those expected by work functions changes due to adsorbed atoms (applying the dipolar model). However, there were some large current emission changes noticed when a W monolayer was evaporated from the (011) plane. These changes could be compared to those predicted by Alferieff in this tunneling resonance model.

It was also observed that the intensity of the electron emission current depended on the number of nickel monolayers above the underlying tungsten layer.

MASS TRANSPORT AND ELECTRICAL BREAKDOWN

THE STUDY OF MATTER TRANSPORT ON METAL SURFACES BY FIELD EMITTERS

M. Drechsler

Contre de Recherche des Mécanismes de la Croissance Cristalline CNRS, Université d'Aix-Marseille, Saint-Jérôme, France

*Abstract not available at time of publication.

BUILD-UP PROCESS OF FIELD EMISSION TIP OF TUNGSTEN AND MOLYBDENUM WITH SILICON ADSORPTION

K. Chang, T. Iwata

Department of Electronics, Faculty of Engineering, Tokai University, Hiratsuka, Kanagawa, Japan

The field emission tips of Tungsten (W) and of Molybdenum (Mo) covered by Si show some characteristic buildup (BU) respectively according to the coverage of Si(θ), the temperature of tip (T), and the field strength applied, in FEM. Though the crystallographies of W and of Mo resemble each other, the BU process or the BU pattern is different between that on W and that on Mo.

On W tip a symmetrical protrusion grows on the (110) plane, in the range T = 1200-1400 K and at the coverage θ ≥ 4.1,2 On Mo tip, under almost the same conditions as for the W, it shows a BU of (211) plane, and no BU of (110) plane similar to that of Si/W.

At the same Si coverage and tip temperature as above mentioned, a more sharp protrusion occurs on the (329) plane of W tip when a higher field is applied. On the contrary, a protrusion grows on the (311) plane of Mo tip. In the temperature range beyond 1400 K both the BU of W and of Mo tip covered by Si reveal similar features.

The BU of (110) plane of W tip has been interpreted as due to the growth of Si or Si-W complex on the plane rearranged. In the BU of Si/Mo, there was no evidence of the rearrangement on the (110).

Besides the BU phenomena, the adsorption and crystal growth of Si on W or on Mo shows some differences, respectively, especially for the shape of the nucleus of crystal and for the zone where the nucleation occurs.3 On Mo, when the Si is deposited at room temperature on one side of the tip and diffused over the tip surface by heating at the temperature below 1000 K, some stable Si layers cover the last layer which has been diffused previously, regardless of the plane index. No such multi-layers are observed on the W tip. The interaction between Si and Si on the substrate, therefore, seems to be different on the W opposed to the Mo.

1. The coverage θ of Si was defined from the characteristics of work function vs. Si deposition.

CRYSTAL SHAPE CHANGES BY ADSORPTION

M. Drechsler, A. Müller*

Centre de Recherche des Mécanismes de la Croissance Cristalline, CNRS, Université d'Aix-Marseille, Saint-Jérôme, France; *Fritz-Haber-Institut der Max-Planck-Gesellschaft, Faradayweg 4-6, 1 Berlin 33

The heating of a metal (without electric field) changes the crystal shape by adsorption-influenced diffusion (facetting) as known from many microscopic observations including FEM and FIM. A better study of this phenomenon requires better information on (1) the crystallo-
graphy of the appearing faces, (2) the adsorbed substances, (3) the coverage degrees, (4) the adsorption structures, and (5) the surface energy changes. We have studied this phenomenon in particular for carbon on tungsten. Equilibrium states were produced on tungsten crystals depending on temperature ($1200 < T < 2600$ K) and coverage ($0 < \theta < 10^{15}$ atoms/cm$^2$). Absolute coverages were determined by the method of Piquet et al. Changes of the geometry of the tip crystal were measured using FEM and FIM, to determine the surface-free energies of different faces by Wulff's construction respectively by an analogous equation. The results allow an energy classification of different C on W adsorption sites or states.

A first attempt is described for a new method to determine adsorption structures by a FEM-FIM technique, i.e., using data on the coverages and the crystallography of the reconstructed faces. A few structure models are presented. The carbon atoms adsorb preferentially along surface steps respectively on surface sites where the adatoms can enter as deep as possible into the surface. The old idea that carbon forms a two-dimensional graphite structure on {334} has probably to be revised. According to a new hypothesis, the (334) faces are formed by a less dense carbon adsorption structure. Sometimes terrace structures appear whose step height exceeds the normal atomic height. Such structures can be explained as part of special equilibrium shapes of minimum potential energy. One of the conclusions is: The study of chemisorption at temperatures where surface self-diffusion occurs is incomplete if this does not include investigations of the reconstructed adsorption structures with its changes of the size and energy of different faces.


EFFECTS OF GLASS ELECTROLYSIS ON ELECTRICAL BREAKDOWN IN HIGH VACUUM* Karl G. Hernqvist

RCA Laboratories, Princeton, New Jersey 08540

*Abstract not available at time of publication.

REGENERATIVE PROCESSES LEADING TO SURFACE FLASHOVER IN VACUUM* R. A. Anderson, J. P. Brainard

Sandia Laboratories, Albuquerque, New Mexico 87185

Charging of the surface of an electrically stressed insulator in vacuum by an avalanche of secondary emission electrons is well established, and there is experimental evidence that the avalanche is involved in the formation of the flashover path. However, the transition from the low-current prebreakdown secondary emission avalanche to a highly conductive plasma channel has not been satisfactorily explained. A model based on electron-stimulated desorption of gas from the insulator surface is presented which accounts for the transition to breakdown in fast-pulsed surface flashover. The time delay preceding breakdown in this model is largely determined by the rate of accumulation of a layer of positive charge, composed of desorbed ions and desorbed neutrals ionized while drifting through the layer of electrons in the avalanche. Eventually the positive charge enhances the electric field near the cathode end of the insulator and a regenerative growth of desorption and ionization occurs. As the electric field rises other regenerative processes become possible, which rapidly lead to breakdown. Time delays according to this model are in good agreement with our experimental results for polymethyl methacrylate and alumina ceramic insulators which were of the order of 10 mm long and broke down in 4 ns at 10$^7$ V/m. In addition, this model accounts for the approximate proportionality we observe between the time delay and the inverse square of applied voltage for time delays between 1 and 20 ns, as well as the more rapid increase in time delay with decreasing voltage beyond 20 ns.


*Work supported by U.S. Dept. of Energy.

FIELD ION MICROSCOPY OF SILICON* T. Sakurai, A. J. Melmed

METALLURGY
We have been investigating oriented silicon whiskers using field ion microscopy (FIM), atom-probe FIM, and field desorption microscopy (FDM), and have found that silicon behaves quite differently from a metal in the presence of a high field. For instance, Si field evaporates as random clusters of Si ions, when evaporated at or below room temperature in vacuum, instead of evaporating in an orderly manner from the surface kink sites. This anomaly in field evaporation is believed to be due to a combination of field penetration in the near-surface bulk and the unique bonding geometry of the tetrahedral diamond structure. We also have observed a strong effect of light illumination on the field ion image intensity and quality, unique to semiconductor emitters. This mysterious effect, known for some years, can be summarized as follows:

1. The intensity of a FI image using H2, He, Ar and Kr imaging gases increases markedly (a factor of up to 10) by infrared illumination when a Si emitter surface is not fully developed.
2. As field evaporation continues, the illumination effect decays out gradually.
3. When the Si surface is completely developed to show a fully ordered pattern there is practically no illumination effect.

We show that this effect results from an increase in surface potential, which causes an increased surface field due to the sharp resistance drop across the surface oxide (SiO2) layer, all due to the well known photoconductivity effect.

A STUDY OF SPINODAL DECOMPOSITION IN Cu-Ti ALLOYS BY MEANS OF THE FIM-ATOM-PROBE AND FDM

R. Wagner

Institute for Metal Physics and SFB 126, University of Göttingen, Göttingen, West Germany

In literature there exists a controversy about the precipitation mechanism in Cu-Ti above 330°C. Some authors propose a classical nucleation and selective growth mechanism, others propose a spinodal mechanism. From a preliminary study of variously heat-treated Cu-Ti alloys with the FIM-atomprobe, we concluded that the decomposition reaction is the spinodal one. The mechanical hardening of these alloys supports this conclusion, and one should be able to quantify the hardening mechanism in modulated structures by measuring the modulation amplitude and wavelength with the FIM atom-probe.

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FIM STUDY OF Mo-Re ALLOYS

K. Teramoto, H. Morikawa, Y. Hashiro, N. Igata, A. Kohyama

Department of Coordinated Science, Nagoya Institute of Technology, Gokiso-cho, Showa-ku, Nagoya 466
Abstracts of 25th International Field Emission Symposium

Japan; \^{}Department of Materials Science, Faculty of Engineering, University of Tokyo, 7-3-1 Hongo, Gunkyo-ku, Tokyo 113 Japan

FIM observations were made of molybdenum alloys (Mo-0.03 at % Re-0.1 at % C, Mo-0.3 at % Re-0.2 at % C, Mo-3 at % Re-0.1 at % C). Many additional bright spots scattered on the usual molybdenum image were characteristic of the alloy images. The concentration of bright spots obtained on (110) and {121} planes was about 10 times larger in the Mo-3%Re alloy than in the Mo-0.3%Re alloy, and also in Mo-0.3%Re alloy than in Mo-0.03%Re alloy, suggesting that these characteristic bright spots came from the rhenium atoms. However, the concentration of bright spots in the Mo-0.3%Re alloy images was about 0.5%, somewhat higher than that of the rhenium atoms themselves. This difference may imply the partial contribution of carbon atoms to the bright spots.

The distribution of bright spots was also studied in a circular area on a (110) plane. A series of images obtained by sequential field evaporation gave a spatial distribution.

Similar observations were carried out on specimens annealed at 1800°C for 1 hr. There was no noticeable difference in concentration and distribution of the bright spots between these as-rolled and annealed specimens.

FIELD-ION MICROSCOPIC STUDY ON PRECIPITATION PROCESSES IN Fe-Cr AND Al-Cu ALLOYS

Tetsuo Abe, Ken-ichi Hirano

Department of Metallurgy, Materials Science and Metal Processing, Faculty of Engineering, Tohoku University, Aoba, Aramaki, Sendai, 980 Japan

The field-ion microscope can provide us with more direct information than can the transmission electron microscope on the atomic processes of phase transformation, such as the spinodal decomposition and formation of the G.P. zones.

In Fe-Cr alloy systems, the spinodal decomposition cannot be fully studied by the transmission electron microscope because it cannot distinguish the Fe-rich zones from the Cr-rich zones because of the similarity of Fe and Cr atoms in the atomic radii and the scattering amplitudes for electrons. However, the field-ion microscope will be a powerful tool because it can provide us with images of individual atoms in which Fe atoms can be distinguished from Cr atoms.

In some Al alloys, the formation process and the structure of the G.P. zones have been proposed based on the X-ray analysis, but the atomic structure of the G.P. zones cannot be displayed clearly by the transmission electron microscope because of the effect of the strain field around the G.P. zones. Here again the field-ion microscope may be useful.

We have succeeded in observing the modulated atomic arrangement in Fe-45 at % Cr alloy formed by the spinodal decomposition and the G.P. zones (monolayer discs of Cu atoms) in Al-1.5 at % Cu alloy.

COMPOSITION OF CARBIDE PARTICLES IN STAINLESS STEELS

H.-O. Andrén, A. Henjingred, H. Nordén

Department of Physics, Chalmers University of Technology, S-402 20 Göteborg, Sweden

The creep strength of stainless steels containing Mc-type particles is at present being investigated. In particular, the effect of adding two carbide-forming elements to the steel is studied. As a part of this study, atom-probe analyses of the carbide particles are being made.

Mc-type particles are non-stoichiometric in that some carbon is missing (the stability range of TiC is 32 to 49% C, to mention one example). Some carbides form ordered structures at certain compositions (e.g. VC0.84), and it is believed that ordering contributes to stability against particle coarsening during high temperature service.

Analyzing these particles in the atom-probe, we have found Ti, V and Nb to evaporate from particles doubly and triply charged (Nb also as Nb4+), but no metal-carbon complex ions of any kind have been detected.

The analyses made so far show that small particles contain considerable amounts of chromium (but little iron). The particles were analyzed using a large acceptance angle which gives a greater yield and thus a more accurate measure of the composition. In this way estimates could also be made of the variation in composition within the particle and at the particle-matrix interface.

ATOM-PROBE STUDIES ON SURFACE SEGREGATION IN THE BINARY ALLOY, TUNGSTEN-MOLYBDENUM
In the past, evidence has been obtained indicating that the surface composition of a binary alloy can differ from the bulk composition. Experimental work mainly with Auger spectroscopy and recently atom-probe studies have been done on this subject. In addition, there is an abundance of theoretical work.

In this case an imaging atom-probe has been used to analyze the top surface layers. A probe diameter of about 50 Å and evaporation rates ranging from 1-5 events per pulse have been used.

A quantitative analysis of the TOF-spectra of unannealed specimens give a surface composition close to the bulk composition. After heat treatment in situ, remarkable changes in the abundance of tungsten and molybdenum ions occur. The obtained surface composition depends on annealing temperature and the duration of the heat treatment. The preliminary results will be discussed with respect to the cited works.


ZONES IN ALUMINUM ALLOYS

E. D. Boyes
University of Cambridge and University of Oxford, United Kingdom

The techniques for imaging and analyzing aluminum-based materials in the FIM/Atom-probe will be reviewed briefly and their applications to the study of specific materials described. The initial low temperature homogeneous precipitation produces zones which may continue to contain some aluminum, in, for example, Al-Cu, Al-Zn-Mg and Al-Mg-Si but not Al-Si alloys, and it has been suggested that both the structure and morphology of the zones may be a sensitive function of the alloy composition.

The advantages of the FIM are:
(a) the sensitivity and relatively simple interpretation of image contrast features a few nm in size, (b) the resolution of size measurements, (c) simplified microanalysis, particularly for matrix elements in those precipitates which are more refractory than the matrix.

The fine-scale distribution \(10^{17} - 10^{19}\) per cc is convenient for FIM/Atom-probe.


ATOM-PROBE

AN "IMAGING ATOM-PROBE" FOR THE ANALYSIS OF ELECTROPLATINGS

Michael Martinka, S. Brooks McIane
Physics Department, Pennsylvania State University, University Park, Pennsylvania 16802

This paper describes the design of an "Imaging Atom-Probe" which will display the spatial distribution of a selected mass constituent of an electrodeposited material. Within the mass spectrometer's flight path, field-evaporated ions are retarded and then post-accelerated to increase flight time and reduce angular timing errors (flat 75 mm chevron detector), respectively.

Particular attention is given to the waveshapes of the evaporation pulse and the detector gating pulse. The gate pulse is applied to a stainless steel support ring at the perimeter of the channel plate, and propagates through a conductive coating toward the center of the detector surface. B. Nahrath, M. Shakhatre and G. Decker have simulated these transmission properties by a 40-element distributed RC system, and they obtained a delay time of 8 nanoseconds between the perimeter and a one-inch-diameter circle about the center. We have directly measured the transmission properties of a 75 mm-diameter Inconel-coated, with a resistance of 10 ohms from perimeter to center, channel plate. Both 7 and 14 ns wide pulses with rise times of 0.5 ns, terminated into 50 ohms, were applied to the channel plate, and waveforms were observed at the terminator with the plate not connected, the channel plate perimeter, and at the center of the detector. The pulse at the
perimeter is attenuated and somewhat broadened. This pulse shape is maintained as the pulse propagates to the center of the detector. Expanded time sweeps, 5 ns/cm, show less than a 1 ns delay time between the perimeter and the center. This was simulated by a capacitor, 130-150 pF, in series with a small resistor, 15-20 ohms, with an RC constant of 2-3 ns. Note, to obtain a rise and fall time of 3 ns at this channel plate the length of transmission line (cap. to ground - 30 pF/ft) from the pulser to the terminator-channel plate system must be kept short, less than one foot.


PERFORMANCE AND APPLICATIONS OF AN IMAGING ATOM-PROBE

M.K. Miller, T. J. Godfrey, P. A. Beaven, P. R. Williams, K. M. Delargy, G. D. W. Smith

University of Oxford, Department of Metallurgy and Science of Materials, Parks Road, Oxford OXI 3PH, England

An Imaging Atom-Probe has been developed for metallurgical research. The design features (1) a 76.2 mm double-curved channel plate assembly with a flight path of 118 mm, (2) a continuously variable nanosecond gate and delay circuit of particularly simple design, and (3) facilities for the direct display and recording of mass spectra.

Experimental work has mainly been directed towards gaining a better understanding of factors controlling the quantitative accuracy of Atom-Probe results. Systematic studies have been carried out of the effects of a number of variables on the mass spectra produced from alloys. The main variables studied were evaporation pulse fraction and pulse repetition rate. Both of these factors require careful control if reproducible results are to be obtained. The effects of residual gases and image gases on the mass spectra have also been investigated. The necessity of carrying out analyses under the best possible vacuum conditions is emphasized.

Initial metallurgical applications of the instrument will also be described. The distribution of alloy elements in steels and nickel superalloys is currently being investigated.

QUANTIFICATION OF ATOM-PROBE FIM DATA

T. T. Tsong, Yee S. Ng

Physics Department, Pennsylvania State University, University Park, Pennsylvania 16802

It is shown that the atom-probe signals of a sample do not give directly the true composition of the sample. A statistical method has been devised to convert the apparent composition to the true composition. The analysis is valid when (a) the field evaporation rate is low enough that each H.V. pulse removes only a small fraction of a surface layer, and (b) the pulse-dc voltage ratio is high enough that field evaporation occurs only during the pulses. Under such conditions, the average number of signals of the ith species detected per each H.V. pulse is given by

\[
\bar{n}_i = \sum_{n=0}^{\infty} \frac{e^{-ted}n!}{n!} \left[1 - (1 - e^{-ted})^{n_i}\right] P_n(n) P_n(\bar{n}),
\]

or

\[
\bar{n}_i = \sum_{n=0}^{\infty} \frac{e^{-ted}n!}{n!} \left[1 - (1 - e^{-ted})^{n_i}\right] P_n(n) P_n(\bar{n}).
\]

In the equation, e represents the transmission coefficient, e_d the detector efficiency, f_i the true fraction of the ith species, and \(\bar{n}\) the average number of atoms field-evaporated per H.V. pulse covered by the probe hole, and

\[
P_n(n) = \frac{n!}{n! (n-n_i)!} e^{-n} f_i^{n_i} (1-f_i^{1-n_i})
\]

and

\[
P_n(\bar{n}) = \frac{\bar{n}^n}{n!} \exp(-\bar{n}).
\]

The apparent fractional abundances \(F_i\)'s are thus related to the true fractional abundances \(f_i\)'s by

\[
F_i = \frac{1 - \exp(-e_i e_d^{f_i} \bar{n})}{N - \sum_{i=1}^{N} \exp(-e_i e_d^{f_i} \bar{n})}
\]

with \(j = 1, 2, \ldots, N\) for an \(N\)-components system.

The details of how to use this sta-
tical analysis in practical problems will be discussed. Using the analysis, the true composition depth profiles of dilute alloys are obtained with a single atomic layer resolution in a surface segregation study. We will demonstrate how misinterpretation of data can result without a proper statistical analysis.

* This work has been supported by NSF Grant No. DMR-76-11418.


ToF ATOM-PROBE INVESTIGATION OF SURFACE SEGREGATION IN DILUTE ALLOYS*

Yee S. Ng, T. T. Tsong, S. B. McLane Jr.

Physics Department, Pennsylvania State University, University Park, Pennsylvania 16802

The surface segregation in dilute alloys can be investigated with the ToF atom-probe FIM with a single atomic layer resolution. The technique has now been successfully applied to study the segregation of Cr atoms to the surface of stainless steel 410. After a surface was developed by field evaporation, it was annealed at 500 ± 50°C for three to five minutes in the absence of an applied field to equilibrate the surface composition. The tip was then quenched down to 80°K. A thermal end form developed after each annealing. Atom-probe signals were subsequently collected by slow field evaporation of the top surface layer and the underlying layers. By using the statistical analysis reported, the apparent composition obtained directly from the atom-probe data was then converted into the true composition.

Cr atoms were found to segregate to the surface of stainless steel 410 at that temperature. The first layer concentration of Cr in the (110) plane was found to be 38.5 ± 12.5% and 63.4 ± 10.2% respectively for samples with average near surface layers Cr concentrations of 6.3 ± 2.1% and 11.9 ± 2.5%. The heat of segregation of Cr to the (110) plane of stainless steel 410 was derived to be 3.43 kcal-mole and 3.92 kcal/mole respectively from the two sets of data when an ideal solution model of surface segregation is adapted. However, our data fit better with a regular solution model.

We have also detected segregation of Cr atoms to the (012) plane of stainless steel 410. Since this plane was not well developed after each annealing, we could not discriminate those signals coming from the top surface layer and those coming from the near surface layers.

In addition, preliminary results from Ni-Cu and other dilute alloys will be presented. Cu atoms were found to segregate to the surface of Ni-Cu alloy at 550 ± 50°C. Multilayer copper composition was obtained with a single atomic layer resolution on the (111) plane of this alloy.

* This work also supported by NSF Grant DMR-76-11418.


ATOM-PROBE AND FIELD ION MICROSCOPY OF SEMICONDUCTORS*

T. T. Tsong, Mike Herman, Yee S. Ng

Department of Physics, Pennsylvania State University, University Park, Pennsylvania 16802

In close cooperation with Dr. A. J. Melmed of NBS, we have made some progress in studying semiconductors and insulators. We focus on the following three subjects:

1. Atom-probe mass spectroscopy: To achieve nano-second pulse field evaporation of semiconductors, we used GaAs emitters heavily doped with Zn (~2 x 10⁻¹⁸ cm⁻³). Although the pulse field evaporation could only be marginally seen by eye, we obtained ToF atom-probe mass spectra with relatively good yield. Molecular ions such as As⁺, As⁺, and Ga₂ have been observed. In hydrogen and oxygen, the number of arsenic hydrides and oxides ions detected greatly exceeded that of gallium hydrides and oxides, inconsistent with the assumption that on GaAs surfaces chemisorption of hydrogen and oxygen occurs only on As sites at low coverages. Other interesting features of this study will be presented.

2. Photoillumination effect: It has been found earlier that field ionization above and field evaporation of semiconductors and insulators are enhanced by photoillumination. We found that this effect is very pronounced for silicon oxide surfaces at low temperatures even at a relatively low light level.
We now believe that photoconductivity is mainly responsible for this effect. The potential drop across an oxide layer depends on the layer thickness, the tip temperature, and the light intensity. It may amount to a few kV. This quantity can be derived quite accurately from I-V curves. In addition the electron-hole recombination rate, which again depends on both the tip temperature and the oxide layer thickness, and which may amount to several seconds, can be measured from the relaxation time of the FI enhancement. By changing the oxide layer thickness the effect of band bending on the electron-hole recombination rate can be investigated. It is important to note that the effect of band bending is exactly the same as that of field penetration, and can therefore be controlled by applied field. Our study should yield significant information on physics of semiconductors and insulator surfaces.

(3) We also found that field emission from a Si tip deviates significantly from the Fowler-Nordheim plot by light illumination. Such effect persists even when a monochromatic light of 12,000 Å (≈ 1 eV < Egap) is used. The effect is explained in terms of field penetration into the near surface layers.

*This work was supported by NSF Grant DMR-76-11418.

1. T. T. Tsong, Yee S. Ng, A. J. Melmed, to be published in Surface Sci.

FIM AND ATOM-PROBE STUDIES OF DEFECTS IN DOPED TUNGSTEN LAMP WIRES

P. A. Beaven, K. M. Delargy, M. K. Miller, P. R. Williams, G. D. W. Smith

University of Oxford, Department of Metallurgy and Science of Materials, Parks Road, Oxford OX1 3PH, England

Previous FIM observations of defects in aluminum-potassium-silicon ('AKS') doped tungsten lamp wire\(^{1,2}\) have been substantially extended. The accumulated evidence shows that a large number of observations in the earlier FIM literature of tungsten concerning the "cross-over effect", "dissociated dislocation loops", "horseshoe dislocations" etc. can be ascribed to the imaging of small defects introduced into the tungsten wire during the doping process. These defects fall into two main classes: small voids (believed stabilized by the presence of potassium) and small solid particles, of hitherto unknown composition.

Each of these classes of defect has been investigated using the T.O.F. Atom-Probe. In the case of the small voids, attempts at chemical analysis have been unsuccessful, owing to ion-optical aberration effects. In some cases, no substrate ions at all were collected from

PROGRESS WITH FIM AND ATOM-PROBE INVESTIGATIONS OF NICKEL-BASED SUPERALLOYS


University of Oxford, Department of Metallurgy and Science of Materials, Parks Road, Oxford OX1 3PH, England

Further investigations of the microstructure and fine-scale composition of nickel-based superalloys have been carried out using TEM, FIM, T.O.F. Atom-Probe and Imaging Atom-Probe techniques. These studies follow the initial work on the determination of γ and γ' phase composition in alloy IN939, reported last year.

New results to be presented concerning alloy IN939 include the following:

(1) Determination of the composition of the ultra-fine secondary precipitates formed during the low temperature (700 °C) final aging treatment. The phase present has been identified as γ', but with a substantially lower cobalt content than in the primary γ' precipitates.

(2) Investigation of the initial stages of decomposition of the solution-treated alloy. Computer methods have been devised to follow the localized composition fluctuations occurring during the early stages of γ' formation. Fluctuations in the concentrations of any number of elements can be followed simultaneously.

(3) Determination of the composition of the primary MC carbide phase. All the major carbide-forming elements (Ti, Zr, Ta, Nb, Cr and W) were found to be present in carbide particles of overall composition MC0.88. Nitrogen and boron were also present, the latter being enriched at the carbide/matrix interface.

Work is also in progress to investigate the effects of prolonged exposure, under stress, at elevated temperatures on the microstructure and properties of superalloys. Highly anisotropic γ' particle shapes have been observed by FIM in a creep specimen of NIMONIC 90.

Similar work is being undertaken on TN939.

the region of the void, despite the presence of a large image gas ion current at the detector, thus demonstrating a substantial trajectory difference between the image gas ions and the substrate ions in the region of the void.

Microanalysis of solid particles was more successful. Particles located both in grain boundary and matrix regions have been analyzed. The overall compositions obtained were in the range W50 to W30, which corresponds approximately to that expected for the β-tungsten phase. Traces of dopant elements were found in solid solution in these particles, supporting earlier suggestions that the β-tungsten phase may be of importance in retaining the trace elements within the microstructure during the wire manufacturing processes. 3


ROUND-ROBIN ATOM-PROBE EXPERIMENT: PRELIMINARY RESULTS
A. J. Melmed, J. J. Carroll, S. S. Brenner*
Surface Science Division, National Bureau of Standards, Washington, D.C. 20234; "U. S. Steel Research Laboratory, Monroeville, Pennsylvania 15146

Following discussions at the 24th IFES in Oxford, we initiated a round-robin experiment intended to provide information about the relative and "absolute" accuracies of atom-probe instruments. A ternary Mo-based alloy was chosen as the specimen material, and specimens were sent to all of the participating laboratories for analysis. Details of the specimen selection and preparation techniques will be described, experimental problems will be discussed, and a preliminary analysis of results will be presented.

EMISSION SPECTROSCOPIES

ANGLE-RESOLVED PHOTO-EMISSION STUDIES OF SURFACES USING SYNCHROTRON RADIATION
(Distinguished Guest Lecture)

E. W. Plummer
Physics Department, University of Pennsylvania, Philadelphia, Pennsylvania

Recent developments in angle-resolved photoelectron spectroscopy using synchrotron radiation will be discussed. Experiments which shed light on the nature of surface and surface reactions will be emphasized. The combination of prolonged light and angle-resolved collections allows the experimentalist to apply simple symmetry rules to determine the symmetry of each electronic state at the surface. This will be illustrated with examples of clean intrinsic surface states and adsorbate energy levels. In contrast, these same symmetry rules can be used with molecular states of known symmetry to determine the bonding configuration. The continuous range of photon energies available using synchrotron radiation allows one to look at resonances in the continuum, for defect effects and to determine the photo-ionization cross-section. How this information can be used to determine the spatial nature of the electronic states at the surface as well as the bonding configuration of adsorbates will be illustrated.

TOTAL ENERGY DISTRIBUTIONS OF FIELD-EMITTED ELECTRONS AT HIGH CURRENT DENSITY
A. E. Bell, L. W. Swanson
Oregon Graduate Center, Beaverton, Oregon 97005

Measurements of the total energy distributions (TED) have been carried out at high-current density J = 10^6 to 10^8 A/cm^2 for a high-work function (built-up (100)W, ψ = 4.5 eV) and low-work function (Zr/(100)W, ψ = 2.5 eV) field emitter. At high values of J, both emitters give values of the full width at half maximum (FWHM) of the TED which exceed the values based on Fowler-Nordheim theory by a few eV. This deviation of the FWHM values increases with J, is independent of temperature and decreases with emitter radius for a specified beam acceptance angle. Studies of these anomalous values of FWHM values extend from 8 to 1975 K. The broadening of the energy distribution occurs on both sides of the Fermi peak and results in a FWHM of up to 3.5 eV for the 2.5 eV work function field emitter compared to an approximately 0.8 eV calculated value. Similar FWHM values were observed for the 4.5 eV work function emitter. The angular intensity
of the field emission beam at the highest FWHM values observed was 0.9 mA/steradian in the case of the 4.5 eV work function emitter and more than 1.4 mA/steradian in the case of the 2.5 eV work function emitter. When the build-up emitter was blunted back so that the emitter field factor decreased from $2.4 \times 10^4 \text{cm}^{-1}$, the FWHM decreased from approximately 3.1 eV to 2.0 eV when the emitter was operated at an angular intensity of $\approx 0.9 \text{mA/steradian}$.

It is believed that Coulomb interaction in the vacuum space a few emitter radii in front of the emitter can account for these results, although internal electron energy broadening mechanisms cannot be ruled out.

BEHAVIOR OF THIN-FILM, FIELD-EMISSION CATHODES IN THE PRESENCE OF SELECTED GASES

C. A. Spindt

Physical Electronics Group, SRI International, Menlo Park, California 94025

The thin-film, field-emission cathode is a relatively low-voltage field emitter, and therefore can be expected to operate over long periods without significant damage due to sputtering. Tests in ultravacuum have now been continuing for over 29,000 hours with no measurable change in voltage-current characteristics, and the cathodes have operated successfully at pressures up to $10^{-5}$ Torr.

We have recently tested the cathode's tolerance to high pressures of specific gases. The voltage-current relationship of a 5000-tip cathode array was established in a $10^{-9}$ Torr vacuum, and the cathode was then exposed to pressures of $10^{-6}$ to $10^{-5}$ Torr of argon, neon, helium, hydrogen, ammonia, methane, hydrogen sulfide, air, water, and oxygen. Exposure time varied from minutes to days for the various gases. In all cases, the emission returned to the initial value when the sample gas was pumped away. We will report the voltage-current characteristics under the influence of the sample gases, as well as the recovery characteristics when the chamber was re-evacuated to $\approx 10^{-9}$ Torr.

A single, 5000-tip cathode was tested in all ten sample gases and in $10^{-9}$ Torr for a total operating time of 1200 hours. At the end of that time, we found that $9\%$ of the tips had failed. A proportionate change in emission current was not noted because the accuracy of the measurement was about $\pm 5\%$.


FIELD-ELECTRON AND ION EMISSION

MAGNETISM AT SURFACE AND INTERFACES BY SPIN-POLARIZED FIELD EMISSION (Invited)

E. Kisker, E. Kuhlmann, M. Campagna, M. Landolt

Institut für Festkörperforschung der Kernforschungsanlage Jülich GmbH, D-5170 Jülich; Bell Telephone Labs, Murray Hill, New Jersey 07974

Magnetic properties of surfaces and interfaces can be studied by complementing a field emission microscope with a probe hole and an electron spin polarization detector. In this talk, we discuss experimental results obtained with single crystal Ni and Fe tips and with epitaxially grown Fe and EuS on W(112). In the case of the evaporated films two components of the polarization vector could be determined. With nickel tips, a negative polarization ($-3\% \pm 1\%$) is observed from (100) planes, and with iron tips $25\% \pm 5\%$ for the (100) planes. By admission of $H_2$ to full coverage the nickel spin polarization was found to vanish within experimental accuracy. A tentative explanation of these results will be presented.

For EuS deposited on tungsten emitters, magnetic interface effects can be studied, and the "spin-filter" action will be discussed.

MANY-BODY AND SPIN-DEPENDENT SURFACE POTENTIAL EFFECTS IN SPIN-POLARIZED FIELD EMISSION*

D. Nagy, P. H. Cutler, E. T. Feuchtwang

Department of Physics, Pennsylvania State University, University Park, Pennsylvania 16802

A spin-dependent generalization of the Appelbaum-Brinkman transfer Hamiltonian method is developed and used to derive an expression for the spin-polarized current density in the field emission from ferromagnets. For a given spin orientation, the current density depends on the spectral amplitudes for that spin orientation. The self-energy operators which determine the spectral amplitudes are approximated by local, energy-independent, spin-diagonal potentials. Although spin-flip processes
are thereby neglected, the detailed variation of the surface potential and an approximate account of many-body effects are included by means of an effective surface potential \( v_{\text{eff}}(z) \), which is the self-consistent surface potential of field-free, spin-polarized jellium in the local density approximation. The calculation of \( v_{\text{eff}}(z) \) by means of the spin-dependent generalization of the Kohn-Sham version of the Hohenberg-Kohn density functional formalism is described and numerical values are obtained for a bulk uniform background density \( p_0 \) and bulk spin polarization \( P \) appropriate for ferromagnetic Ni. With \( p_0 = 4.05 \times 10^{-2} \) electrons/(Bohr unit) and \( P = 0.20 \), a work function \( \gamma \approx 4.7 \) eV is obtained, which is in good agreement with experimental values for Ni. It is also found that the surface potentials for the two spin orientations differ sufficiently to have a small but significant effect on the spin polarization of the emitted current.

*Partial support for this research was provided by NSF Grant DMR 77-00934.


THE EFFECTS OF GEOMETRICAL ASYMMETRY AND MULTIPLE IMAGE INTERACTIONS OF THE TUNNELING PROPERTIES OF POINT CONTACT DIODES

S. J. Shepherd, N. M. Miskovsky, T. E. Sullivan, P. H. Cutler, A. A. Lucas*

Pennsylvania State University, University Park, Pennsylvania 16802;
*Facultés Universitaires N.S. de la Paix, B-5000 Namur, Belgium

Point contact diodes have been utilized as detectors, rectification, and frequency-mixing devices in the infrared. A fundamental application of these devices has been the most precise and accurate determination of the velocity of light. Nevertheless the intrinsic mechanism and the properties of point-contact diodes, although of basic interest, are not unambiguously understood. At present there are two models which attempt to explain the nonlinearity and rectification mechanism of the diodes: (1) Tunneling of electron through an intermediate oxide film from whisker to the metal base, i.e., the configuration is considered to be a metal-oxide-metal (MOM) tunneling junction. (2) The nonlinearity and rectification processes are the result of a thermally enhanced field emission (TFE).

Recently a new explanation of the rectification mechanism was proposed which takes into account explicitly the geometrical shape of the two electrodes. In a model calculation of the tunneling asymmetry, the geometrical asymmetry was introduced by considering a configuration where the metal point electrode is assumed to be a hyperboloid of revolution positioned from a flat base of the same metal. It was found that for a tip radius equal to 100 Å and a tip-base distance of 20 Å the current ratio (\( j_p/j_g \)) may be as high as 5, suggesting that rectification properties of metal point-contact diodes can be explained without requiring material asymmetry.

In the work to be reported the analysis of the geometrical rectification mechanism has been extended to include (i) effects of the electron image interaction; (ii) different geometrical configurations of the junction; (iii) the important multiple image interactions due to the close spacing of the metallic electrodes.

To include the effect of the image and multiple image interactions we have obtained, using a Green's function formalism, an exact solution of the potential field for hyperboloidal tip with a planar anode. This is used to construct the potential barrier in the calculation of the tunneling current as a function of bias voltage. Following Schmeit's et al., these calculations use a free electron model and WKB transmission coefficient. Direct comparison with the triangular barrier model of Schmeit's yields the corrections due to all image-force interactions.

We have also considered a diode configuration of two identical metallic spheres closely spaced (i.e., \( > 10 \) Å). A complete solution has been obtained for the potential distribution, including the multiple-image interactions using Green's function formalism. The diode model with spherical electrodes has especial importance because experiments using this arrangement (i.e., spherical electrodes) have been conducted at NBS and I-V characteristics for spacings of the order of 10-20 Å have been obtained. Although some features of the measured curves exhibit qualita-
tive agreement with planar MOM theory, there is an obvious need for I-V characteristics based on more realistic diode geometries, which the present analysis gives promise of providing.

*This research was supported in part by IRIS (Institute for Research in Interface Science) of the Belgian Ministry for Science Policy.


FIELD PENETRATION AND BAND BENDING IN THE SPACE CHARGE REGION OF SEMICONDUCTORS*

T. T. Tsong

Physics Department, Pennsylvania State University, University Park, Pennsylvania 16802

The present theory of band bending in the surface space charge region of semiconductors, developed by Shockley, Brattain and Bardeen, Kingston and Neustadt and others, contains an approximation. The theory fails completely when the amount of band bending becomes comparable to (\(E_c - E_F\)) in a positive field and to (\(E_F - E_V\)) in a negative field. The amount of band bending and the internal field existing in many interface phenomena, field emission and field ionization are far beyond the validity of this approximation.

The approximation is removed and the resulting Poisson's equation is solved numerically. The field penetration as well as the details of the band bending, valid in all field ranges of interest, are calculated. The approximate theory is shown to be a particular case of our new calculation under extremely low fields. The amount of band bending and the depth of field penetration from the new calculation are presented.

The effect of field penetration on the field ionization of gases above semiconductor surfaces, and the field evaporation of semiconductor surfaces, are also discussed.

*Supported by NSF Grant DMR 76-11418.

CONTROLLED THERMONUCLEAR FUSION AND ION IMPLANTATION

SURFACE PHYSICS AND MATERIALS PROBLEMS IN TFTR* (Distinguished Guest Lecture)

H. F. Dylla

Plasma Physics Laboratory, Princeton University, Princeton, New Jersey 08540

The Tokamak Fusion Test Reactor (TFTR) is presently under construction at the Princeton Plasma Physics Laboratory. The device is projected to reach conditions of energy breakeven by injection of greater than 20 MW of 120 keV neutral deuterium beams into a tritium target plasma. Before TFTR becomes operational in 1981, a number of crucial materials problems involving plasma-surface interactions require resolution. Of primary concern among these problems is an understanding of impurity generation mechanisms and the development of conditioning techniques and/or wall materials which might minimize impurity effects. A related problem concerns hydrogen isotope trapping and re-emission from surfaces exposed to plasma. The re-emission of hydrogen isotopes during a discharge strongly affects the evolution of the plasma density. An understanding of the trapping mechanism is particularly important to minimize the tritium wall loading. A variety of surface analytical techniques have been employed to diagnose these problems on existing tokamaks and recent measurements from the Princeton Large Torus (PLT) will be reviewed.

*This work was supported by the U. S. Department of Energy Contract No. EY-76-C-02-3073.
SURFACE ANALYSIS OF FIELD-EMITTER
SAMPLES EXPOSED TO THE PLASMAS
OF PLT AND ISX*

G. L. Kellogg, J. A. Panitz

Sandia Laboratories, Albuquerque,
New Mexico 87185

In an attempt to help characterize the plasma-wall interactions in operating tokamak reactors, the Imaging Atom-Probe mass spectrometer (IAP), the Field-Ion Microscope (FIM), and the Transmission Electron Microscope (TEM) have been used to analyze the surface and near-surface region of field-emitter samples which were placed at the wall position and exposed to the plasmas of the Princeton Large Torus (PLT) and the Impurities Study Experiment (ISX) tokamak fusion reactors. Measurements of the extent of damage to the specimen surfaces, the composition and thickness of deposited surface films, and the depth distribution of low energy plasma species implanted into the near-surface region of the samples have been carried out.

Three sets of samples (two in PLT and one in ISX) have been investigated thus far. Although each set was subjected to different exposure conditions (e.g., type and number of plasma discharges, limiter material used, position of samples with respect to limiters), some consistent results from the analysis of the samples have been found. For example, all of the tips which had direct plasma exposure showed evidence for a change in surface morphology (i.e., lattice damage or deposited surface films), whereas those tips which were placed in the reactor at the same time but shielded from line-of-sight exposure to the plasma were apparently unchanged. Mass spectra obtained with the IAP showed that plasma and impurity species become trapped within the damaged regions and deposited films, but are not implanted into the substrate bulk.

The implications of these results (and others to be presented) as they relate to the plasma-wall interactions in operating tokamaks will be discussed.

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N. Igata, S. Sato, K. Shibata, A. Kohyama

Department of Materials Science,
University of Tokyo, 7-3-1 Hongo
Bunkyo-ku, Tokyo 113 Japan

Hitherto we have investigated FIM observation of tungsten irradiated with C ions. In this study the purity of the specimen was 99.95% annealed at 1800°C for 2 hrs. For comparison of radiation damage with other charged particles, tungsten wires were irradiated with 80 keV He⁺, 300 keV O⁺ and 300 keV Ar⁺ ions. The irradiation temperature was 30°C for He⁺ and 30-200 C for O⁺ and Ar⁺. The size distribution of vacancy clusters was investigated after each irradiation. In the case of He⁺ irradiation the larger vacancy clusters between 10-15 Å was not observed. This would be due to less primary knock-on energy in He⁺ irradiation compared with the case of O⁺ and Ar⁺ irradiation. The swelling AV/V showed saturation tendency in the earlier stages above 0.5 dpa in the case of O⁺ and Ar⁺ irradiation, and the value was higher than in the case of He⁺ irradiation. This would be also due to higher primary knock-on energy, and the interstitials almost escaped from the surface in the earlier stage in the case of Ar⁺ and O⁺ irradiation.

A DISCRETE-CHARGE MODEL OF A
FIELD-ION EMITTER SURFACE

Richard G. Forbes

Department of Physics, University of Aston, Gosta Green, Birmingham, United Kingdom

This paper explores some of the properties of a basic version of a new type of charged-surface model in which the surface atoms in a charged surface are represented in terms of an array of superimposed monopoles and dipoles, together with a distant array of monopoles of the opposite sign. (The distant array is necessary for electrostatic self-consistency.)

The model is a development of earlier work by the present author and by Tsong and Müller, and can be regarded as a significant step forward from the normal description of charged surfaces provided by classical electrostatics. The need for such a model seems an unrealized consequence of the Hellman-Feynman theorem.

Exploration of the model brings out
a fundamental inadequacy in the concept of "applied field" as used in field-ion theory. The usual treatment is compatible neither with classical continuous-charge-distribution models nor with discrete-charge models. This fact has significant consequences for theories of field adsorption and field evaporation.

The following tentative conclusions have been drawn from the analysis of an infinite square array:

1. The monopole contribution to field adsorption is less important than the dipole contribution, though it should not be neglected in detailed treatments.

2. Existing calculations of localized field-adsorption binding energies are not electrostatically self-consistent.

3. For a surface atom with a given external field above it, there may exist a theoretical maximum for its polarization energy that is independent of the proper SI polarizability of the surface atom.

4. Non-linearity in the local field near the surface should in principle be taken into account in field-ion theory.

5. Significant negative field-induced work-function corrections may exist.

6. Estimates of "surface-atom polarizability" drawn from surface diffusion experiments in high fields may be unreliable.

7. Calculations of critical distances may need revision, and the whole question of "where is the emitter's electrical surface" needs re-examination.

The analysis also raises in the author's mind much more general doubts about the usefulness of flat-surface models of a charged surface. It seems much more likely that a good quantum-mechanical theory of charged surfaces would emerge from localized-orbital-type models.
The 25th
International Field Emission Symposium
July 17-22, 1978

The University of New Mexico
105 Education Building
Albuquerque, New Mexico

Hosted by

Sandia Laboratories, Albuquerque, New Mexico

Cover: The logo of the 25th International Field-Emission Symposium includes a 21 K field-ion image of iridium (8.55 kV, BIV) in a Zia Sun motif. The Zia Sun Symbol is often associated with New Mexico, and appears on the State flag. It has its origins in the ancient glyphs of the Pueblo Indians.
The Local Organizing Committee would like to welcome all participants and friends of the 25th International Field Emission Symposium to Albuquerque, New Mexico. We have attempted to provide you with a broad and varied technical program, as well as the opportunity to become acquainted with New Mexico - "The Land of Enchantment." Consequently, an evening session has been planned to allow a free afternoon in an otherwise hectic schedule. A special feature of this year's meeting will be the presentation of the first annual Erwin W. Müller Memorial Medal to an outstanding young scientist.

The technical program will be highlighted by a number of "distinguished guest lectures" and invited papers. Our attempt is to bring to your attention forefront areas of research to which field-emission, field-ion, and field-desorption techniques might be profitably applied. In addition, a special session has been organized to review past and present attempts to image biological molecules with high-field techniques.

We would especially like to thank the National Science Foundation for providing travel support for several foreign and United States participants under Grant #DMR78-09078, the University of New Mexico (and Mr. Edmund B. Kasner, in particular) for administration of this NSF grant, the Office of Basic Energy Sciences of the United States Department of Energy which provided additional support, and the New Mexico Chapter of the American Vacuum Society, which provided partial travel support for a Distinguished Guest Lecturer (H. F. Dylla).

This symposium would not have been possible without the combined efforts of many individuals. Our particular thanks go to Arlene Padilla and Myrna Walla who provided expert secretarial and clerical support and to Tillie Pierce and "Mac" McHarney who designed the Symposium artwork and prepared master copies for printing. In addition, numerous support facilities at Sandia provided services which made organizing this meeting so much easier.

J. A. Panitz
Local Organizing Committee Chairman, 25th International Field Emission Symposium
Local Organizing Committee of the 25th International Field Emission Symposium
July 17-22, 1978

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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
17. 1970 New Haven, Connecticut  F. Hutchinson
18. 1971 Eindhoven, The Netherlands  A. van Oostrom
19. 1972 Urbana-Champaign, Illinois  G. Ehrlich
21. 1974 Marseille, France  M. Drechsler
22. 1975 Atlanta, Georgia  H. E. Grenga, J. J. Hren et al.
23. 1976 University Park, Pennsylvania  E. W. Müller &
                                           T. T. Tsong

PROGRAM OF THE 25th INTERNATIONAL FIELD EMISSION SYMPOSIUM

Monday, July 17, 1978

8:30 a.m.  Registration

9:15 a.m.  J. A. Panitz: Opening Remarks

9:30 a.m.  Morgan Sparks (President, Sandia Laboratories)
            Opening Address: An Introduction to Sandia Laboratories

10:15 a.m. Coffee

10:45 a.m. Distinguished Guest Lecture
            M. Isaacson: Scanning Transmission Electron Microscopy at Near-Atomic Resolution; the Present State-of-the-Art

11:30 a.m. Lunch

SESSION 1: SPECIAL SESSION ON BIOLOGICAL IMAGING

1:00 a.m.  Distinguished Guest Lecture
            Ivar Giaever: Protein on Metal Surfaces

1:45 a.m.  A. J. Melmed (Invited): Imaging of Molecules, Past and Present

2:25 p.m.  F. Hutchinson (Invited): Field-Ion Imaging of DNA and Other Organic Molecules

3:10 p.m.  J. A. Panitz, Ivar Giaever: Low-Field Desorption Imaging of Proteins

3:30 p.m.  Coffee
### Monday, July 17, 1978 (continued)

**SESSION 2: SURFACE ADSORPTION**

<table>
<thead>
<tr>
<th>Time</th>
<th>Session</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>4:00 p.m.</td>
<td>R. T. Tung, W. R. Graham: Single Atom Self Diffusion on Nickel</td>
<td>6</td>
</tr>
<tr>
<td>4:20 p.m.</td>
<td>O. Nishikawa: Binding Sites and Binding States of Sn on W</td>
<td>7</td>
</tr>
<tr>
<td>4:40 p.m.</td>
<td>R. J. Culbertson, T. Sakurai, T. T. Tsong: Field Ion Microscopy of Gallium on Tungsten</td>
<td>8</td>
</tr>
<tr>
<td>5:00 p.m.</td>
<td>J. A. Panitz: Field-Induced Desorption of Benzene from Tungsten</td>
<td>9</td>
</tr>
<tr>
<td>5:20 p.m.</td>
<td>Dinner</td>
<td></td>
</tr>
</tbody>
</table>
### SESSION 3: E. W. MÜLLER MEMORIAL MEDAL COMPETITION

<table>
<thead>
<tr>
<th>Time</th>
<th>Presentation</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>9:00 a.m.</td>
<td>Jörg Piller: Aiming Error in Metallurgical Specimens</td>
<td>10</td>
</tr>
<tr>
<td>9:20 a.m.</td>
<td>M. K. Miller, P. R. Williams, P. A. Beaven, G. D. W. Smith: Quantitative Determination of Alloy Element Partitioning in Pearlitic Steels by Atom Probe Analysis</td>
<td>11</td>
</tr>
<tr>
<td>9:40 a.m.</td>
<td>G. H. Robertson, T. Sakurai, T. T. Tsong: A Study of Ordered Ni₄Mo Surface on an Atomic Scale</td>
<td>12</td>
</tr>
<tr>
<td>10:05 a.m.</td>
<td>G. S. Gipson, D. W. Yannitell, H. C. Eaton: Analysis of the Electric Field Distribution Inside a Field Ion Microscope by the Finite Element Method</td>
<td>13</td>
</tr>
<tr>
<td>10:20 a.m.</td>
<td>Coffee</td>
<td></td>
</tr>
</tbody>
</table>

### SESSION 4: E. W. MÜLLER MEMORIAL MEDAL COMPETITION

<table>
<thead>
<tr>
<th>Time</th>
<th>Presentation</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>10:50 a.m.</td>
<td>N. Ernst: Appearance Potential Measurements on Singly and Doubly Charged Field Evaporated and Field Ionized Metal Ions</td>
<td>14</td>
</tr>
<tr>
<td>11:10 a.m.</td>
<td>A. R. Waugh, P. F. Mills, M. J. Southon: A Combined Conventional and Imaging Atom-Probe and Its Application to Segregation Studies</td>
<td>15</td>
</tr>
<tr>
<td>11:50 a.m.</td>
<td>C. Patel: A Study of Chemisorbed Nitrogen and Oxygen on Growth of Thin Gold Films on Tungsten</td>
<td>17</td>
</tr>
<tr>
<td>12:10 p.m.</td>
<td>J. R. Chen and R. Gomer: Diffusion Studies of Adsorbates on W</td>
<td>18</td>
</tr>
<tr>
<td>12:30 p.m.</td>
<td>Lunch</td>
<td></td>
</tr>
<tr>
<td>4:30 p.m.</td>
<td>Dinner</td>
<td></td>
</tr>
</tbody>
</table>
Evening | SESSION 5: FIELD EMISSION AND ION SOURCES
--- | ---
7:00 p.m. | R. G. Forbes: A Basic Wave-Mechanical Theory of Field-Ion Energy Distributions 19
7:20 p.m. | J. D. Levine: Field Emission Sources for Large Area Displays 20
7:40 p.m. | Refreshments
8:10 p.m. | P. Sudraud, J. Van de Walle: Characterization of Ion Emission from a Taylor Cone of Liquid Gold 21
8:30 p.m. | L. W. Swanson, G. A. Schwind: Field Emission from a Liquid Metal 22
8:50 p.m. | G. L. R. Mair and A. von Engel: Gallium Field Ion Emission from Liquid Point Anodes 23
9:10 p.m. | R. L. Seliger: Submicron Imaging of a Liquid Gallium Source 24
**SESSION 6: SURFACE CHEMISTRY**

<table>
<thead>
<tr>
<th>Time</th>
<th>Event</th>
</tr>
</thead>
<tbody>
<tr>
<td>9:00 a.m.</td>
<td>Distinguished Guest Lecture</td>
</tr>
<tr>
<td></td>
<td>G. Somorjai: Catalytic Chemistry: A Surface Science Viewpoint</td>
</tr>
<tr>
<td>9:45 a.m.</td>
<td>J. H. Block, R. -G. Abitz, M. Domke, E. Hummel: Field Ionization, Field Evaporation and Field Desorption of Silver Ions, a Comparison of Appearance Energies</td>
</tr>
<tr>
<td>10:00 a.m.</td>
<td>W. A. Schmidt, O. Frank: Field Ion Appearance Energies of CO⁺ Ions Formed at Noble Metal Surfaces</td>
</tr>
<tr>
<td>10:25 a.m.</td>
<td>Coffee</td>
</tr>
</tbody>
</table>

**SESSION 7: FIELD DESORPTION MASS SPECTROMETRY**

<table>
<thead>
<tr>
<th>Time</th>
<th>Event</th>
</tr>
</thead>
<tbody>
<tr>
<td>10:55 a.m.</td>
<td>F. W. Röllgen, U. Giessmann, H. J. Heinen: Processes of Ion Formation in Field Desorption Mass Spectrometry</td>
</tr>
<tr>
<td>11:15 a.m.</td>
<td>J. H. Block, R. -G. Abitz: Appearance Energies of Field Ionized and Field Desorbed Polyatomic Sulfur Molecules</td>
</tr>
<tr>
<td>11:35 a.m.</td>
<td>D. F. Barofsky, E. Barofsky, L. M. Isabelle: Quantitative Applications of Field Desorption Mass Spectroscopy</td>
</tr>
<tr>
<td>11:55 p.m.</td>
<td>K. H. Ott, F. W. Röllgen: Ionization by Alkali Ion Attachment on the Surface of Electrolytic Solutions</td>
</tr>
<tr>
<td>12:15 p.m.</td>
<td>Lunch</td>
</tr>
<tr>
<td>Time</td>
<td>Session Title</td>
</tr>
<tr>
<td>--------</td>
<td>-------------------------------------------------------------------------------</td>
</tr>
<tr>
<td>2:00 p.m.</td>
<td>R. Reifenberger, M. J. G. Lee: Thermally Enhanced Field Emission from a Laser Illuminated Tungsten Tip</td>
</tr>
<tr>
<td>2:20 p.m.</td>
<td>J. P. Jones: Field Emission Spectroscopy of Bismuth on W(100)</td>
</tr>
<tr>
<td>2:40 p.m.</td>
<td>R. DiFoggio and R. Gomer: A Novel Method of Probe Hole Current Measurement</td>
</tr>
<tr>
<td>2:50 p.m.</td>
<td>J. Lira-Olivares: Field Emission from the Superlattice Planes of Ordered Ni₄W</td>
</tr>
<tr>
<td>3:10 p.m.</td>
<td>Coffee</td>
</tr>
<tr>
<td>3:40 p.m.</td>
<td>M. Drechsler (Invited): The Study of Matter Transport on Metal Surfaces by Field Emitters (A Review)</td>
</tr>
<tr>
<td>4:10 p.m.</td>
<td>K. Chang, T. Iwata: Build-up Process of Field Emission Tip of Tungsten and Molybdenum with Silicon Adsorption</td>
</tr>
<tr>
<td>4:30 p.m.</td>
<td>M. Drechsler, A. Müller: Crystal Shape Changes by Adsorption</td>
</tr>
<tr>
<td>4:50 p.m.</td>
<td>K. G. Hernqvist: Effects of Glass Electrolysis on Electrical Breakdown in High Vacuum</td>
</tr>
<tr>
<td>5:10 p.m.</td>
<td>R. A. Anderson, J. P. Brainard: Regenerative Processes Leading to Surface Flashover in Vacuum</td>
</tr>
</tbody>
</table>
SESSION 10: METALLURGY

9:00 a.m.  T. Sakurai, A. J. Melmed: Field Ion Microscopy of Silicon  


9:40 a.m.  R. Wagner: A Study of Spinodal Decomposition in Cu-Ti-Alloys by Means of the FIM-Atomprobe and FDM  

10:00 a.m. K. Teramoto, H. Morikawa, Y. Yashiro, N. Igata, A. Kohyama: FIM Study of Mo-Re Alloys  

10:20 a.m. Coffee  

SESSION 11: METALLURGY  

10:50 a.m. T. Abe, K-i. Hirano: Field-Ion Microscopic Study on Precipitation Processes in Fe-Cr and Al-Cu Alloys  


11:30 a.m. M. Leisch: Atom Probe Studies on Surface Segregation in the Binary Alloy Tungsten-Molybdenum  

11:50 a.m. E. D. Boyes: Zones in Aluminum Alloys  

12:10 p.m. Lunch  

SESSION 12: ATOM-PROBE  

2:00 p.m.  M. Martinka, S. B. McLane, Jr.: An "Imaging Atom-Probe" for the Analysis of Electroplatings  

2:20 p.m.  M. K. Miller, T. J. Godfrey, P. A. Beaven, P. R. Williams, K. M. Delargy: Performance and Application of an Imaging Atom Probe
Thursday, July 20, 1978 (continued)

SESSION 12: ATOM-PROBE (continued)

2:40 p.m.  T. T. Tsong, Y. S. Ng: Quantification of Atom-Probe FIM Data  51

3:00 p.m.  Y. S. Ng, T. T. Tsong, S. B. McLane, Jr.: ToF Atom-Probe Investigation of Surface Segregation in Dilute Alloys  52

3:20 p.m.  Coffee

SESSION 14: ATOM-PROBE

4:10 p.m.  T. T. Tsong, M. Herman, Y. S. Ng: Atom-Probe and Field Ion Microscopy of Semiconductors  53

4:30 p.m.  K. M. Delargy, P. A. Beaven, M. K. Miller, G. D. W. Smith: Progress with FIM and Atom-Probe Investigations of Nickel-Based Superalloys  54

4:50 p.m.  P. A. Beaven, K. M. Delargy, M. K. Miller, P. R. Williams, G. D. W. Smith: FIM and Atom Probe Studies of Defects in Doped Tungsten Lamp Wires  55

5:10 p.m.  Atom-Probe Workshop
            A. J. Melmed, J. J. Carroll, S. S. Brenner: Round Robin Atom-Probe Experiment: Preliminary Results  56

7:00 p.m.  First bus leaves Hokona Hall for Sandia Crest

8:00-9:00 p.m. Social Hour (cash bar) Sandia Crest

9:00-10:00 p.m. Banquet, Summit House Restaurant, Sandia Crest

10:30 p.m. Presentation of the First Annual Erwin W. Müller Memorial Medal and Business Meeting
Friday, July 21, 1978

8:00 a.m.  Sandia Laboratories Tour
12:45 a.m. Lunch for tour participants at the Coronado Club, Kirtland Air Force Base

SESSION 15: EMISSION SPECTROSCOPIES

2:45 p.m. Distinguished Guest Lecture
E. W. Plummer: Angle-Resolved Photo-Emission Studies of Surfaces using Synchrotron Radiation

3:30 p.m. A. E. Bell, L. W. Swanson: Total Energy Distributions of Field-Emitted Electrons at High Current Density

3:50 p.m. C. A. Spindt: Behavior of Thin-Film, Field-Emission Cathodes in the Presence of Selected Gases

4:10 p.m. Coffee

SESSION 16: FIELD-ELECTRON AND ION EMISSION

4:30 p.m. E. Kisker, E. Kuhlmann, M. Campagna, M. Landolt (Invited): Magnetism at Surfaces and Interfaces by Spin Polarized Field Emission


5:10 p.m. S. J. Shepherd, P. H. Cutler, N. M. Miskovsky, T. E. Sullivan, A. A. Lucas: The Effects of Geometrical Asymmetry and Multiple Image Interactions on the Tunneling Properties of Point Contact Diodes

5:30 p.m. Dinner
Saturday, July 22, 1978

SESSION 17: CONTROLLED THERMONUCLEAR FUSION AND ION IMPLANTATION

9:00 a.m.  Distinguished Guest Lecture
           H. F. Dylla: Surface Physics and Materials Problems in TFTR  63

9:45 a.m.  G. L. Kellogg, J. A. Panitz: Surface Analysis of Field-Emitter Samples Exposed to the Plasmas of PLT and ISX  64

10:05 a.m. N. Igata, S. Sato, K. Shibata, A. Kohyama: Field Ion Microscopy of Tungsten Irradiated with He, O and Ar Ion Particles  65

10:25 a.m. Coffee

10:55 a.m. R. G. Forbes: A Discrete-Charge Model of a Field-Ion Emitter Surface  66

SESSION 18: POST DEADLINE PAPER

11:20 a.m. Yuji Sadakane, K-i. Hirano: Probability Density of Displacement of Adatom on Tungsten Surface  67
<table>
<thead>
<tr>
<th>Name</th>
<th>Page</th>
<th>Name</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Abe, T.</td>
<td>45</td>
<td>Igata, N.</td>
<td>44, 65</td>
</tr>
<tr>
<td>Abnitz, R. G.</td>
<td>26, 29</td>
<td>Isaacson, M.</td>
<td>1</td>
</tr>
<tr>
<td>Anderson, R. A.</td>
<td>40</td>
<td>Isabelle, L. M.</td>
<td>30</td>
</tr>
<tr>
<td>Andren, H-O</td>
<td>42, 46</td>
<td>Iwata, T.</td>
<td>37</td>
</tr>
<tr>
<td>Barofsky, D. T.</td>
<td>30</td>
<td>Jones, J. P.</td>
<td>33</td>
</tr>
<tr>
<td>Barofsky, E.</td>
<td>30</td>
<td>Kellogg, G. L.</td>
<td>64</td>
</tr>
<tr>
<td>Beaven, P. A.</td>
<td>11, 50, 54, 55</td>
<td>Kisker, E.</td>
<td>60</td>
</tr>
<tr>
<td>Bell, A. E.</td>
<td>58</td>
<td>Kohyama, A.</td>
<td>44, 65</td>
</tr>
<tr>
<td>Block, J. H.</td>
<td>26, 29</td>
<td>Kuhlman, E.</td>
<td>60</td>
</tr>
<tr>
<td>Boyes, E. D.</td>
<td>48</td>
<td>Landolt, M.</td>
<td>60</td>
</tr>
<tr>
<td>Brainard, J. P.</td>
<td>40</td>
<td>Lee, M. J. G.</td>
<td>32</td>
</tr>
<tr>
<td>Brenner, S. S.</td>
<td>56</td>
<td>Leisch, M.</td>
<td>47</td>
</tr>
<tr>
<td>Carroll, J. J.</td>
<td>56</td>
<td>Levine, J. D.</td>
<td>20</td>
</tr>
<tr>
<td>Chang, K.</td>
<td>37</td>
<td>Lira-Olveres, J.</td>
<td>35</td>
</tr>
<tr>
<td>Chen, J. R.</td>
<td>18</td>
<td>Lucas, A. A.</td>
<td>62</td>
</tr>
<tr>
<td>Cowan, P. L.</td>
<td>16</td>
<td>Mair, G. L. R.</td>
<td>23</td>
</tr>
<tr>
<td>Culbertson, R. J.</td>
<td>8</td>
<td>Martinina, M.</td>
<td>49</td>
</tr>
<tr>
<td>Cutler, P. H.</td>
<td>61, 62</td>
<td>Melmed, A. J.</td>
<td>3, 41, 56</td>
</tr>
<tr>
<td>Cumpagna, M.</td>
<td>60</td>
<td>McLane, Jr., S. B.</td>
<td>49, 52</td>
</tr>
<tr>
<td>Delargy, K. M.</td>
<td>50, 54, 55</td>
<td>Miller, M. K.</td>
<td>11, 50, 54, 55</td>
</tr>
<tr>
<td>DiFogglo, R.</td>
<td>34</td>
<td>Mills, P. F.</td>
<td>15</td>
</tr>
<tr>
<td>Domke, M.</td>
<td>26</td>
<td>Miskovsky, N. M.</td>
<td>62</td>
</tr>
<tr>
<td>Drechsler, M.</td>
<td>36, 38</td>
<td>Morikawa, H.</td>
<td>44</td>
</tr>
<tr>
<td>Dylla, H. F.</td>
<td>63</td>
<td>Müller, A.</td>
<td>30</td>
</tr>
<tr>
<td>Eaton, H. C.</td>
<td>13</td>
<td>Nagy, D.</td>
<td>61</td>
</tr>
<tr>
<td>Ernst, N.</td>
<td>14</td>
<td>Nisikawa, O.</td>
<td>7</td>
</tr>
<tr>
<td>Feuchtwang, E. T.</td>
<td>61</td>
<td>Nordén, H.</td>
<td>46</td>
</tr>
<tr>
<td>Forbes, R. G.</td>
<td>19, 66</td>
<td>Ng, Y. S.</td>
<td>51, 52</td>
</tr>
<tr>
<td>Frank, O.</td>
<td>27</td>
<td>Ott, K. H.</td>
<td>31</td>
</tr>
<tr>
<td>Giaever, I.</td>
<td>2</td>
<td>Panitz, J. A.</td>
<td>5, 9, 64</td>
</tr>
<tr>
<td>Giesmann</td>
<td>28</td>
<td>Patel, C.</td>
<td>17</td>
</tr>
<tr>
<td>Gipson, G. S.</td>
<td>13</td>
<td>Piller, J.</td>
<td>10</td>
</tr>
<tr>
<td>Godfrey, T. J.</td>
<td>50</td>
<td>Plummer, E. W.</td>
<td>57</td>
</tr>
<tr>
<td>Gomcr, R.</td>
<td>18</td>
<td>Reifenberger, R.</td>
<td>32</td>
</tr>
<tr>
<td>Graham, W. R.</td>
<td>6</td>
<td>Robertson, G. H.</td>
<td>12</td>
</tr>
<tr>
<td>Heinen, H. J.</td>
<td>28</td>
<td>Röllgen, F. W.</td>
<td>28, 31</td>
</tr>
<tr>
<td>Henjered, A.</td>
<td>46</td>
<td>Sadakane, Y.</td>
<td>67</td>
</tr>
<tr>
<td>Herman, M.</td>
<td>53</td>
<td>Sakurai, T.</td>
<td>8, 12, 41</td>
</tr>
<tr>
<td>Hernqvist, K. G.</td>
<td>39</td>
<td>Sato, S.</td>
<td>62</td>
</tr>
<tr>
<td>Hirano, K-i</td>
<td>45, 67</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hummel, E.</td>
<td>26</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hutchinson, F.</td>
<td>4</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Name</td>
<td>Page</td>
<td></td>
<td></td>
</tr>
<tr>
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</tr>
<tr>
<td>Schmidt, W. A.</td>
<td>27</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Schwind, G. A.</td>
<td>22</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Seliger, R. L.</td>
<td>24</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Shepard, S. J.</td>
<td>62</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Shibata, K.</td>
<td>65</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Smith, G. D. W.</td>
<td>11, 54, 55</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Somorjai, G.</td>
<td>25</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Southon, M. J.</td>
<td>15</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Spindt, C. A.</td>
<td>59</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sudraud, P.</td>
<td>21</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sullivan, T. E.</td>
<td>62</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Swanson, L. W.</td>
<td>22, 58</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Teramoto, K.</td>
<td>44</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tsong, T. T.</td>
<td>12, 16, 51, 52</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tung, R. T.</td>
<td>61</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Van de Walle, J.</td>
<td>21</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Von Engel, A.</td>
<td>23</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Wagner, R.</td>
<td>43</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Waugh, A. R.</td>
<td>15</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Williams, P. R.</td>
<td>11, 50, 55</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Yannitell, D. W.</td>
<td>13</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Yashiro, Y.</td>
<td>44</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Over the past decade there has been an increased coupling of microscopy (which gives us spatial resolution information) with microprobing (which gives us compositional information) at spatial resolution levels smaller than the wavelength of light. The coupling is best exemplified with the transmission electron microscope. It has been pushed to the extreme such that very fine beams of electrons can be produced which are as small as 2.5 Å in diameter. We are therefore in a situation where we may expect to be able to perform chemical localization at near atomic spatial resolution from a wide variety of objects. One can presently perform spectroscopy of fast electrons transmitted through thin samples from areas approaching 20 Å in size. In addition, individual heavy atoms can be visualized on light element substrates using such atomic dimension electron beams. We will try to review the state-of-the-art of the coupling of microscopy and spectroscopy using fine electron beams and speculate on some of the potential applications in the biological and materials sciences.
notes
Protein on Metal Surfaces

Ivar Giaever

All the enzymes and many of the hormones in the human body are globular protein molecules. The structure of a protein molecule is closely related to its function. Some of these features will be discussed in an elementary way. Illustrations will be given of how some of the properties of protein molecules can be studied by adsorbing the molecules on metal surfaces. Finally, some problems and difficulties associated with the adsorption of protein onto field emitter tips will be mentioned.
Several types of microscopy have been and are being applied to the quest for detailed information about the morphology and atomic structure of molecules, with special interest in important biological molecules. These will be reviewed with emphasis on the results thus far obtained and the apparent obstacles to improved imaging capability. Particular attention will be given to the field emission (FEEM and FIM) efforts.
FIELD ION IMAGING OF DNA AND OTHER ORGANIC MOLECULES

Franklin Hutchinson
Department of Molecular Biophysics and Biochemistry
Yale University
New Haven, Connecticut 06520

Large biological molecules have been imaged on the surface of a field ion tip by surrounding them with layers of metal atoms from an evaporation source. The evaporated atoms striking the molecules to be imaged either do not stick, or are field desorbed as the electric field is raised to visualize the surface by field ion microscopy. The image of the molecule is formed by the edges of the cavity in the absorbed metal layer, and resolution is improved as the adsorbed layers become more ordered. Unmistakable images have been obtained of DNA and of transfer RNA molecules to a resolution of about 10 Å. Images of phthalocyanine could not be unequivocally distinguished from defects in evaporated metal layers deposited in the absence of organic molecules. The method is similar in principle to that used by Mueller and Rendulic, who imbedded organic molecules in a metal layer electroplated on a field ion tip. The method has similarities to that in which an image is formed by field desorption of easily ionized molecules condensed on a tip bearing the object to be imaged. Field ion microscopy can scan a much smaller area for suitable images than can transmission microscopy, which is a serious limitation.
notes
LOW-FIELD DESORPTION IMAGING OF PROTEINS*

J. A. Panitz
Sandia Laboratories,† Albuquerque, NM 87185

and

Ivar Giaever
General Electric Research and Development Center
Schenectady, NY 12301

ABSTRACT

A novel low-field (< 1V/Å) desorption technique is described for imaging the contour of protein molecules placed on the apex of field-emitter tips. Image contrast relies on a distinct and measurable difference in the desorption field of a species of low ionization potential physisorbed on the molecule, and on the adjacent tip surface. Preliminary results are described, using benzene to image Bovine Serum Albumin (BSA) and Immuno-Gamma-Globulin (IgG). Although the imaging procedure appears to be feasible, extreme difficulty in reproducibly placing the proteins on the tip apex, in the desired concentration, has been encountered. These difficulties, together with future implications of the technique will also be discussed.

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†A U. S. Department of Energy Facility.
Single Atom Self Diffusion on Nickel*

Raymond T. Tung† and William R. GrahamΔ
Laboratory for Research on the Structure of Matter
University of Pennsylvania
Philadelphia, Pennsylvania 19104

Abstract

Past FIM studies of surface diffusion have been limited to 5d and 4d transition metal substrates. The importance of extending this powerful technique for the study of the atomic mechanisms of surface interactions to the 3d transition series metals is clearly evident. In this paper we present the results from self diffusion studies on several of the low index planes of nickel. We also include some discussion of the specimen preparation techniques which are required to produce nickel surfaces of sufficient perfection for surface diffusion studies.

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† Department of Physics.
Δ Department of Metallurgy and Materials Science.
notes
The binding sites of tin atoms on a tungsten surface are studied by superposing the image of a pseudomorphic tin layer upon the image of a substrate tungsten surface. Superposed micrographs indicate that the lateral locations of the tin atoms correspond to the locations of unoccupied tungsten lattice sites above the surface.

Variations in field emission current with increasing evaporation field are also studied for the (011), (111), (112) and (114) planes. Evaporation of adatoms can be noticed by a sharp increase or decrease in the emission current at the field strength corresponding to the binding state of the adatoms. The binding states of the tin atoms on these planes are discussed.
notes
Field Ion Microscopy of Gallium on Tungsten*

Robert J. Culbertson, T. Sakurai and T. T. Tsang, Physics Department, The Pennsylvannia State University, University Park, PA 16802

The field evaporation of Ga from a W emitter was investigated using a magnetic sector atom probe field ion microscope. A continuous supply of Ga was provided by adjusting both tip temperature and electric field. The Ga coating of a W tip was achieved by dipping the tip in liquid Ga at 325K in air. A clean W end cap was obtained by field evaporating the tip at 78K. By raising the temperature to 300K Ga atoms diffused from the shank of the tip and field evaporated from the tip apex at fields (2V/Å), well below the evaporation field of W (5.7V/Å). The rate of Ga evaporation could be controlled over a wide range by varying either the temperature, the electric field or both. The tip temperature was extremely critical for obtaining a steady Ga ion flux. In some cases the Ga flux was sufficiently high to yield a desorption image of the surface, implying that more than 1000 Ga ions/sec are formed at each imaged site. The desorption image has a resolution comparable to that of a field ion image. Unlike ordinary desorption imaging where the surface atoms are removed, only the continuously replenished Ga atoms are evaporated and the W tip surface remains intact.

After annealing the surface at elevated temperatures we found that the field ion image showed patchy regions of a Ga and W ordered alloy structure similar to that found in Pt electroplated W. The evaporation field of this surface was higher than that of Ga atoms migrating from the shank before annealing.

Below room temperature Ga atoms evaporate as doubly charged ions with a sharp energy distribution comparable to that of He field ionization within the forbidden zone. The binding energy of Ga atoms on W can be derived from the measurement of the energy deficit of the field evaporated Ga ions. Since the substrate W surface remains intact, the evaporation field can be maintained at a constant value. A retarding potential analyzer with 100 meV resolution of the 2000 eV primary ion energy was incorporated in the atom-probe for this purpose. The measured binding energy will be presented as a function of crystallographic orientation of the W substrate and will be discussed based on the adsorption geometry determined from a field ion image.


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notes
FIELD INDUCED DESORPTION OF BENZENE FROM TUNGSTEN*

J. A. Panitz
Sandia Laboratories, † Albuquerque, NM 87185

ABSTRACT

A study of the field-desorption properties of benzene from clean, field-evaporated tungsten surfaces is described. Ramped-DC desorption was used to measure the desorption field of benzene at temperatures below 200°K, while Imaging Atom-Probe spectroscopy was used to determine the identity of the desorbing species in physisorbed, and chemisorbed layers. Time-gated imaging was used to follow the crystallographic behavior of the predominant physisorbed species, C6H6, between 55 K and 200°K.

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†A U. S. Department of Energy Facility.
notes
Aiming Error in Metallurgical Specimens

Jörg Piller *

Institute for Metalphysics and SFB 126
University of Goettingen, Hospitalstr. 12
D 3400 Goettingen, W-Germany

The combined FIM - Atomprobe and Field Desorption Microscope /1/ has been applied for the determination of the aiming error in pure metals and in metallurgical alloys.

Previously it has been shown by several authors that there could be considerable differences between the flight paths of the desorbed ions and their related field ions. This is evident for instance by the fine detailed structures found in multi-layer field desorption micrographs. In contrary it has been demonstrated that under distinct conditions it is possible to aim with the atom probe on single solute or adsorbed atoms which are brightly imaged with a yield near the detection efficiency of the ion detector.

Now it is of interest how the desorbed ions of precipitates are distributed in respect to their field ion image, specially if the precipitates protrude from or are recessed into the specimen surface.

From the desorption images which are gated for the different components of the precipitates and from the field ion images the extension of the precipitates is measured. The influence of the distorted field above protruding precipitates on the targets of the desorbed ions is compared with these of ions of high indexed planes in pure metals.

These results are related with atomprobe measurements where the probe hole is aimed on or nearby the field ion image.


* Competing for the E.W. Müller Medal
notes
QUANTITATIVE DETERMINATION OF ALLOY ELEMENT PARTITIONING IN PEARLITIC STEELS BY ATOM PROBE ANALYSIS

M.K. Miller*, P.R. Williams, P.A. Beaven and G.D.W. Smith

University of Oxford, Department of Metallurgy and Science of Materials, Parks Road, Oxford OX1 3PH, England.

Previous observations of alloy element redistribution processes in patented and cold drawn pearlitic steel wires (1,2) have been extended. Quantitative measurements have been made of solute concentration profiles across the ferrite-cementite interface in as-transformed pearlitic steels containing the alloy elements Mn, Cr and Si. It has been demonstrated that extensive substitutional solute partitioning occurs over the range of transformation temperatures studied (down to 550°C). This result is at variance with previous (lower spatial resolution) electron microprobe analyses, and provides a new insight into the nature of the physical processes occurring during the pearlite transformation. In particular, the existence of a highly effective short-circuit diffusion pathway for substitutional elements in the austenite-pearlite interface would now appear to be clearly established.

References


* Competing for the E.W. Müller Medal
notes
A Study of Ordered Ni\textsubscript{4}Mo Surface on an Atomic Scale.

G.H. Robertson, T. Sakurai, and T.T. Tsong, Physics Department, The Pennsylvania State University, University Park, PA 16802

Field ion microscope (FIM) studies of ordered alloys have great importance because the hemispherical shape of FIM emitters provides a variety of superstructures. The geometrical structure and possible reconstructions can be observed directly on an atomic level. Previous FIM studies of ordered alloys have found that one of the species is usually not imaged.

We have attempted a new study of an ordered alloy, Ni\textsubscript{4}Mo. Its bulk structure is well established [body centered tetragonal (BCT) \(c/a = 0.6231\)]. This BCT structure contains eight Ni and two Mo atoms in the unit cell, i.e., four Ni and one Mo atoms per Bravais lattice point. The structure may be described as five interpenetrating BCT sublattices, one having a single Mo atom per lattice point and the other four with a single Ni atom per lattice point. Each sublattice is translated by \(1/5 <110>\) with respect to a neighboring sublattice. Because of this complexity, some net planes described by the same Miller index number [for example, (110) and (011) or (211) and (121)] have different symmetry and structural properties enhancing our interest in this system.

In our FIM study, using a channel plate image intensifier and liquid H\textsubscript{2} cooling, we have succeeded for the first time in the observation of Ni and Mo layers with atomic resolution. While slowly evaporating the (011) surface layer by layer, we observed only two pure Ni layers below each pure Mo layer rather than the four expected from the alloy structure. It also appeared that the number density of a Ni layer is approximately twice that of a Mo layer. This anomaly was not observed on the (110) plane where we observed four pure Ni layers below each pure Mo layer. The basic structural differences between these two planes is the number density and the spacing, \(d\), of adjacent layers [\(d = 0.8099\) Å for (110) and \(d = 0.6057\) Å for (011)]. The ready interpretation of this anomaly for the (011) plane is that two adjacent Ni layers reconstruct to form a single layer when the top Mo layer is field evaporated. Using the atomic resolution of Ni layers we will try to determine the position of Ni atoms with respect to the Mo atoms for a number of net planes, such as (111), (121), (213) and (123). The field emission images from fundamental planes and superlattice planes will be presented to show a unique potential of using this system to study the electronic structure of a superlattice. It may be possible to obtain the density of states of the superlattice by field electron spectroscopy.


*Competing for the E.W. Müller Medal.
notes
ANALYSIS OF THE ELECTRIC FIELD DISTRIBUTION INSIDE A FIELD ION MICROSCOPE BY THE FINITE ELEMENT METHOD

Gary S. Gipson**, D. W. Yannitell, and H. C. Eaton

Department of Mechanical Engineering
Louisiana State University
Baton Rouge, Louisiana 70803

ABSTRACT

The electric field distribution inside a field ion microscope is determined by means of the numerical method of finite elements. Using this technique, realistic geometries are considered. Analysis of such models has not been possible by conventional analytical methods because of the intractable nature of the solutions. Features included in the numerical model are microscope walls, a flat screen, and for the first time a cold finger, a dielectric, a specimen holder with shape, and an accelerating shield. It is found that neglecting the tedious geometry can yield inaccurate results. The accelerating shield, located about 10⁵ tip diameters from the specimen tip, is shown to provide a substantial enhancement of the electric field at the specimen apex. This can result in significant changes in the specimen stress distribution and may alter the ion trajectories as well. Also studied is the effect of the specimen shank angle on the electric field. It is found that a variation from 0° to 9° in emitter shank angle results in approximately a 40% reduction in the field strength near the apex.
notes
Appearance potential measurements on singly and doubly charged field evaporated and field ionized metal ions

Norbert Ernst

Fritz-Haber-Institut der Max-Planck-Gesellschaft, Faradayweg 4-6, 1 Berlin 33

Experimental appearance potential values $\text{AP}_n (=e\phi + \Delta E_n^+ (F))$ as derived from the onset voltage $\delta$ of integral energy distribution of $n$-fold charged metal ions are compared with theoretical values $\text{AP}_n$ calculated from the image hump model /1,2/ (e elemental charge, $\phi_R$ workfunction retarder electrode). $\text{AP}_n$ is approximately given by:

$$i\text{AP}_n \approx \frac{1}{n} \Delta E_n^+ (F) + \phi_E$$

(1)

where $\Delta E_n^+ (F) = \left( \sqrt{n^2 e^3} - \frac{e}{4 \pi \varepsilon_0} \right)$ is the critical energy deficit of a $n$-fold charged ion at the emitter surface (work function $\phi_E$) according to the Schottky hump condition ($\varepsilon_0$ dielectric constant). When the electric field strength $F$ is high enough field evaporation occurs with nearly zero thermal activation energy. $i\text{AP}_n$ then becomes:

$$i\text{AP}_n \approx \frac{1}{n} (\sum I_n + \Lambda)$$

(2)

$I_n$ is the $n$-th ionization potential and $\Lambda$ the binding energy of the atom to the emitter surface. In table I calculated and literature data are compared with experimental results. With copper, the image hump model of field evaporation is verified. $\text{AP}_n$-values of other species mainly reflect field evaporation with nearly zero activation energy (emitter temperatures below 300 K).

<table>
<thead>
<tr>
<th>n</th>
<th>species</th>
<th>$I_n$</th>
<th>$\text{AP}_n$</th>
<th>$\text{F}[V/\bar{A}]$</th>
<th>$\text{AP}_n[GeV]$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Cu$^+$</td>
<td>11,0</td>
<td>10,9</td>
<td>2,9</td>
<td>2,9 ± 0,3</td>
</tr>
<tr>
<td></td>
<td>Be$^{++}$</td>
<td>12,6</td>
<td>12,5</td>
<td>5,1</td>
<td>1,6 ± 0,2</td>
</tr>
<tr>
<td>2</td>
<td>Be$^{++}$</td>
<td>15,5</td>
<td>15,3</td>
<td>4,5</td>
<td>unknown</td>
</tr>
<tr>
<td></td>
<td>Ni$^{++}$</td>
<td>15,0</td>
<td>14,8</td>
<td>3,4</td>
<td>15,5 ± 0,1-0,2</td>
</tr>
<tr>
<td></td>
<td>Fe$^{++}$</td>
<td>14,1</td>
<td>14,0</td>
<td>3,4</td>
<td>13,9 ± 0,1</td>
</tr>
</tbody>
</table>

$\text{AP}_n$-values in brackets are estimated onsets of second high energy distributions observed in the spectra of doubly charged ions.

Raising the emitter temperature above 900 K leads to thermal evaporation of metal atoms with subsequent field ionization, FI, in the gas phase. The first ionization potentials are measured ($\text{AP}_n \approx I_n$) corresponding to the main onset of energy distributions. A second high energy distribution with onset down to 5 eV below $I_1$ (Be$^+$) is obviously generated by FI of electron impact excited atoms.

/1/ E.W. Müller, Phys. Rev. 102, 618 (1956)
/2/ T.T. Tsong, W.A. Schmidt and O. Frank, Surface Sci. 65 109 (1977)

"*Competing for the E.W. Müller Medal."
A COMBINED CONVENTIONAL AND IMAGING ATOM-PROBE AND
ITS APPLICATION TO SEGREGATION STUDIES

A.R. Waugh*, P.F. Mills & M.J. Southon
Dept. of Metallurgy and Materials Science, University of Cambridge,
Pembroke Street, Cambridge, CB2 3QZ, U.K.

The imaging atom-probe which was constructed in 1975 for metallurgical applications (1) has been modified and updated to combine the elemental mapping capability of the IAP with the quantitative point-analysis capability of the conventional atom-probe. As before, a single 50mm flat channel-plate is used to detect the gated desorption image with a flight-distance of 125mm. A second 50mm channel-plate has now been fitted close to the specimen, which can be tilted through 140° on a specially-built stage. The second plate is used to display the entire field-ion image of the specimen and also incorporates a 2mm diameter probe-hole which defines the entrance aperture of a conventional atom-probe, with provision for energy compensation. A 25mm chevron channel-plate is used as the detector.

As a result of these modifications it is possible to obtain both quantitative mass spectra from small areas of the specimen and also time-gated desorption images from a large area. Data are collected rapidly as an evaporation-pulse generator has been built which uses a mercury-wetted reed relay operated at 250 Hz. Jitter-free operation at this high rate is made possible by using a solid-state high-voltage switch in place of the conventional charging resistor: output-pulse amplitude is adjustable from 300 to 4000 volts. This type of pulser has been added successfully to all three atom-probes currently in use in Cambridge.

Our prime area of research at present is in segregation: the unique ability of the IAP to detect segregation at grain boundaries with high spatial resolution and single-atom sensitivity has been applied further along the lines described previously (2). Further observations have been made of oxygen at grain boundaries in molybdenum, including a grain-boundary node, of carbon in iron, and of tin at and near grain-boundaries in iron. The latter system is of particular interest in that significant quantities of a metallic dimer, Sn$_2^{3+}$, are observed, as well as Sn$^+$ and Sn$^{2+}$.


* Competing for the E.W. Muller Medal
Surface migration of single adatoms on a perfect crystallographic plane occurs by a random sequence of atomic jumps. However, atomic jumps are not necessarily restricted to nearest neighbor sites as is commonly assumed. Analysis of FIM observations of migrating adatoms is uniquely capable of yielding the distribution of jump lengths and directions. Existing data indicates that W adatoms on W(110) jump predominantly along <111> directions in the plane, but can jump various distances in these directions.

*Competing for the E. W. Müller Medal.

**This research done at The Pennsylvania State University was supported by NSF Grant No. DMR 76-11418.
A STUDY OF CHEMISORBED NITROGEN AND OXYGEN ON GROWTH OF
THIN GOLD FILMS ON TUNGSTEN

C. PATEL

University College of North Wales, Department of Electronic Engineering Science, Dean Street, Bangor, Gwynedd N.Wales, United Kingdom.

When condensed on tungsten in ultra-high vacuum, metals are known to form monolayer and multilayer films. The presence of sub-monolayer amounts of preadsorbed gas can however prevent such films from forming.

In an attempt to establish the roles of chemisorbed gases, detailed studies have been made of the effects of small quantities of nitrogen and oxygen as contaminant gases on the structure of thin gold film on tungsten by observing the change in the work-function with coverage and temperature using probe-hole and simple field emission microscopy.

Adsorption of nitrogen on tungsten followed by gold shows notable changes in the work-function when compared with the 'clean' gold behaviour and consequently results in crystallite growth at a gold coverage greater than 2θ. Heating the composite layers at a temperature T > 800K causes crystallites to dissolve and gold reverts to 'clean' behaviour.

Oxygen adsorption on a gold-bearing tungsten surface produces a decrease in $\phi$ of $\approx -0.45$eV for spreading temperatures of 600-750K. This decrease in $\phi$ seems unusual since the oxygen sticking probability on gold is very low for the temperature used. Because the oxygen attacks the tungsten substrate more severely than nitrogen thus the gold atoms aggregate much more readily into crystallites than in the case of nitrogen.

The stepwise heating of the following systems W-Au, W-N₂, W-N₂-Au, W-O₂, W-Au-O₂-Au, W-O₂-Au have been examined for the desorption characteristics of the chemisorbed gases on morphology of gold overlayers. In both nitrogen and oxygen cases the order of their adsorption plays an important part, this is found to be noticeable when the stepwise heating of the following systems were performed: W-O₂-Au Case 1 and W-Au-O₂-Au Case 2. In Case 1 $\phi$ increases very sharply between T = 500-750K and becomes steady in the temperature region T = 900-1400K, Case 2 shows the opposite effect in the T = 500-750K temperature region. This behaviour is thought to be due to inversion of dipoles - and will be described in detail.

Co-adsorption of (N₂-Au) has been studied on the W(100), (111) and (211) planes. Results will be described and compared with known data of the (N₂-W) and (W-Au) system on these planes.

"++ Competing for the E.W. Müller Medal."
notes
The correlation of functions of field emission current fluctuations from the (110) plane of tungsten field emitters covered with submonolayer amounts of oxygen, carbon monoxide, and xenon, have been measured. At temperatures above the onset of surface diffusion, the experimental correlation functions agree with a theoretical model based on single particle diffusion, and yield diffusion coefficients and activation energies of diffusion on this plane. For oxygen, activation energy of diffusion rises from 14 kcal/mole for O/W \leq 0.2 to 22 kcal/mole for O/W = 0.56. For virgin CO, no diffusion could be unequivocally established at any coverages, although a weak signal set in 250°K. For beta CO, diffusion with an activation energy 23 kcal/mole is found, suggesting O is the observed species diffusing. For Xenon, an activation energy of \sim 3 k \text{cal/mole} was found at $\Theta = 0.5$ and $\Theta = 0.9$. At high temperatures the decay of the correlation function is slower than predicted by theory. It is suggested that this results from dynamical correlations, or multi-particle density fluctuations, which can occur when several adsorbate particles are simultaneously in motion. At low temperatures, exponential decay of the correlation function is observed, and explained as a prediffusive flip-flop of adsorbate particles between binding locations. The temperature dependence of the mean square fluctuations in the diffusive regime is discussed in terms of adsorbate-adsorbate interactions.

*Competing for the E. W. Müller Medal.

1. R. Gomer, Surface Sci. 38, 373 (1973)
A basic Wave-mechanical theory of Field-ion energy distributions

Richard G. Forbes

University of Aston, Department of Physics, Gosta Green, Birmingham, UK

This paper presents the outline of a new wave-mechanical theory of field ionization and field-ion energy distributions. The theory is a generalisation of the approach used by Gomer and Swanson (1), and avoids the use of the Landau-Zener approximation. The theory may be regarded as a direct fermion analogue to the theory of radiative emission as developed by Dirac.

The essential feature of the new theory is that it avoids the "double infinity of final states" problem by enclosing the departing ion in a hypothetical one-dimensional sloping-sided box for which the wave-functions can be determined. The rest of the analysis then proceeds by analogy with the well-established theory of Molecular Spectroscopy.

Currently, the theory is in the formal stage of development, but the following results are already clear:

1. A proof can be given that the usual quasi-classical treatment of field-ion imaging should be a valid first approximation, if the electron transition rate-constant is given by an expression involving an electronic overlap integral.

2. The usual quasi-classical treatments of energy distributions should be valid approximations, with the following exceptions:

   (a) The shape of the high-energy edge of the main peak;

   (b) Discussion of surface-plasmon-assisted field ionization.

In the exceptional cases the influence of overlap factors between vibrational wave-functions needs to be taken into account. For surface-plasmon creation the presence of an additional term in the expression for the energy distribution could prove decisive in rejecting this mechanism of the Jason effect on purely theoretical grounds.

These results are not exactly unexpected. However, the author believes that the present treatment of field ionization, which has long been recognised as a "difficult" subject, has been able to avoid logical errors that appear in previous treatments. Very few, if any, previous treatments are strictly compatible with the basic axioms of quantum mechanics.

notes
Component and systems aspects of field emitters as electron sources have been studied for large area displays: $10^6$ display elements, $10^4$ cm$^2$ display area, and a design cost for under $100$. We first investigated UO$_2$/W eutectics provided by Professor A. T. Chapman. Later, a new field emitter technology was developed using copper and glass fused sheets.$^1$ This was more suited to mass production. By mechanical or photolithographic means, Cu tips were formed 10 mils apart on a square array. A tip, when appropriately treated and fused with a glass insulator and a copper extractor, yielded 10-100 μA at 400-1000 V. The design value was 1-10 μA at 200 V, for 200-ft lamberts brightness. Proximity focusing was used. On occasion, beam extraction efficiencies of 100% were obtained.

Systems studies showed the need for an inexpensive feedback sensor and circuit for each of the $10^6$ elements, which would sample the beam current and also correct for display brightness inhomogeneities due to the erratic nature of the field emission source. Such a sampling circuit was built and tested successfully.$^1$

notes
CHARACTERIZATION OF THE ION EMISSION FROM A TAYLOR CONE OF LIQUID GOLD*

P. Sudraud, J. Van de Walle, C. Collieux, P. Trebbia
Laboratoire de Physique des Solides
Associe au C.N.R.S., Bât. 510
Université Paris-Sud
91405 ORSAY - France.

Various ion emission modes issued from a metallic gold surface under the combined effect of a strong electric field and a high temperature have been investigated.

At a temperature higher than the melting point, and under an electric field greater than a critical value, the free surface of a liquid metal is deformed into a Taylor cone shape, the extremity of which has been visualized in-situ in an electron microscope. From the area a few microns large located at the apex of this cone, rather strong positive ion currents can be delivered. Of the order of 1mA, they are quite stable and can last for hours if an appropriate design allows a permanent flow of matter towards this emitting area.

The characteristics of the beam have been analysed with the help of a double-focusing mass spectrometer. The mass distribution contains a large variety of ionic species ranging from doubly ionized Au\(^{++}\) to molecular Au\(_n^+\) (fragments with \(n \leq 7\) have been detected). The main contribution due to Au\(^+\) represents about 70\% of the total emission. Superposed on the general decrease with \(n\) of the molecular ions intensity, slight alternances can be detected; species with odd \(n\) are relatively more abundant than those with even \(n\). This is in good agreement with the theoretical predictions of Joyes concerning the stability of noble metal clusters.

The energy distribution has been measured for all these emission products. The two main ionic species Au\(^{++}\) and Au\(^+\) exhibit a single large peak. Of the order of 45 eV for Au\(^{++}\), the width of this peak is increased to about 70 eV for Au\(^+\) and is followed in this case by a long energy deficit tail. The molecular ions also display a clear secondary maximum: its position gradually shifts towards larger energy losses and its relative importance increases regularly with \(n\). These experimental data provide some interesting basis for the understanding of the emission mechanisms in an EHD liquid metal ion source. The contribution of the space charge effects and of the collision processes in a dense plasma extending over a few hundred angströms above the liquid surface will be discussed.

Endly, the transition between the low temperature ion emission modes from the solid and the high temperature ones from the liquid has been carefully studied when one raises the temperature across the melting point. Intermediate regimes including the geometrical faceting of the metal surface or the growth of micro-needles have been recognized. The relation between the topographical properties of the surface and the ion emission patterns has been established. All these results provide a general frame to classify the different behaviours of a metal surface under these extreme temperature and field environment conditions.


* Research performed with the help of a grant DRME 76/1184.
FIELD EMISSION FROM A LIQUID METAL

by

L. W. Swanson
and
G. A. Schwind

Oregon Graduate Center
Beaverton, Oregon 97005

ABSTRACT

The characteristics of a novel repetitive pulsed electron source employing a liquid Ga/12% In cathode have been examined. Repetitive current pulses as large as 250 A with an applied voltage of only 10 to 12 kV have been observed. The mechanism involves the formation of a field-stabilized cone of the liquid cathode which forms a sufficiently-small apex radius that a regenerative field electron current initiates an explosive emission process of the type described in detail for solid state emitters. If the applied voltage is held just below threshold, a 100 V by 35 nsec pulse voltage is sufficient to initiate the high current pulse.

Several important differences between the liquid and solid phase explosive emission electron sources have been revealed. First, after the vaporization of a small amount of material from the cone apex during the pulse, the liquid phase source self heals thereby yielding a repetitive pulse mode operation with a constant applied voltage. Secondly, there is no limit to the life; i.e., the number of pulses, attainable from the liquid emitter. Third, the current pulse rise time and duration are shorter for the liquid metal source. A remarkable feature of the liquid metal electron source when operated in the free running repetitive mode is the lack of measurable time jitter, < 0.1 nsec, between the onset of successive pulses and their uniform amplitude over the rep rate range 40 to 8,000 pps.

With the reverse polarity, this source becomes an intense ion emitter. Used in a FIM, embodiment patterns with a certain degree of structure can be observed. Mechanisms by which field evaporation from liquid metals occur will be discussed.

notes
GALLIUM FIELD ION EMISSION FROM LIQUID POINT ANODES

G.L.R. Mair and A. von Engel

Department of Engineering Science,
Oxford University,
Parks Road,
Oxford, OX1 3PJ.

Abstract

A brief summary of the principal processes controlling emission is given followed by an investigation of the two components of the collector current and its superimposed pulses when zero, retarding and accelerating potentials are applied to the collector. The effect of secondary electrons emitted by ion impact on the collector is analysed as well as the results of suppressing the secondaries by deflection in a moderate electric or magnetic field. Finally observations of the luminosity at the collector surface and near the apex of the wetted point anode, of the formation and life of protrusions and of the flow of neutral atomic particles and droplets are discussed.
notes
SUBMICRON IMAGING OF A LIQUID METAL GALLIUM SOURCE

R. L. Seliger
Hughes Research Laboratories
Malibu, California 90265

ABSTRACT

A liquid-metal gallium ion source is imaged by a unity magnification, single gap, accelerating lens with a post-lens deflector to form a focused scanning probe. Experiments with this system are being performed to evaluate the suitability of liquid-metal ion sources for high resolution ion probe formation. We report the dependence of the probe diameter \( d_p \) and probe current \( I_p \) on the lens acceptance half-angle \( \alpha_o \) and the total source emission current \( I_s \). The results range between \((d_p, I_p) = (500 \text{ nm}, 1 \text{ nA})\) at \((\alpha_o, I_s) = (6 \text{ mrad}, 10 \mu\text{A})\) and \((d_p, I_p) = (60 \text{ nm}, 68 \text{ pA})\) at \((\alpha_o, I_s) = (1.2 \text{ mrad}, 5 \mu\text{A})\). Spot diameters were measured by sputtering thin gold films with the focused beam. The current density and brightness at the target for the 60 nm diameter, 55 keV probe are \(2.4 \text{ A/cm}^2\) and \(4.8 \times 10^6 \text{ A/cm}^2\text{-sr}\) respectively. Since the experimental system does not yet include a stigmator, magnetic shielding nor substantial vibration isolation, the resolution performance limit of the liquid-metal gallium source has probably not yet been reached.

* This work was supported by the Defense Advanced Research Projects Agency

† To be presented at the 25th International Field Emission Symposium, University of New Mexico, Albuquerque, New Mexico, July 17-21, 1978.
The past ten years have seen the development of techniques the combination of which can be utilized to tackle major problems of heterogeneous catalysis on the molecular scale. Most of these new techniques need only small surface areas (~cm$^2$), but often require high vacuum sample environments. New methods of catalytic research have been developed that use (1) small area samples for structure, composition, and kinetic studies, and (2) mate ultra-high-vacuum and high-pressure reaction conditions.

Four examples of studies involving single crystal surfaces of small area will be presented using three types of surfaces: flat (111) orientation, stepped, and kinked surfaces.

A. Determination of the surface structure of adsorbed C$_2$H$_2$ and C$_2$H$_4$ on the Pt (111) surface for a combination of low-energy electron diffraction (LEED) and high-resolution electron energy loss techniques.

B. The mechanisms of H$_2$-D$_2$ exchange on stepped platinum crystal surfaces by reactive molecular beam-surface scattering.

C. The effect of oxygen on the rates of hydrogenation and dehydrogenation of cyclohexene on kinked platinum crystal surfaces.

D. The effects of additives, C$_2$H$_4$, C$_3$H$_6$ and potassium on the rates and product descriptions during the hydrogenation of carbon monoxide over the iron (111) crystal face.
Field ionization, field evaporation and field desorption of silver ions, a comparison of appearance energies

by

J.H. Block, R.-G. Abitz, M. Domke and E. Hummel

Fritz-Haber-Institut der Max-Planck-Gesellschaft, Faradayweg 4-6, 1 Berlin 33

Mass spectrometric experiments are performed and integral energy distributions are measured for Ag$^+$-ions of various origin. 1) Silver vapor from a Knudsen cell is field ionized and yields appearance energies (AP) in accordance with gas phase ionization: AP is independent on field strength; AP-values represent ionization potentials diminished by kinetic energy contributions of neutrals with nearly Maxwellian distribution. 2) The energy of ions field evaporated from silver layers at a tungsten emitter (Ag-Ag- or Ag-W-surface bonds) is effected by bond energies. The energy deficits are field and temperature dependent. The field dependence is explained in terms of the "charge exchange" or "image hump" model. The temperature dependence results from competing thermal activation, which can be independently determined from rate measurements. With increasing temperature Ag$^+$-ions can pass successively barriers with increasing height at reduced field strength, resulting in reduced energy losses. 3) Field desorption of Ag$^+$-ions performed in a layer of Ag$_2$S at a silver field emitter differs remarkably from usual field evaporation of silver: Although field evaporation occurs at drastically reduced field strength, energy losses are rising by 2 eV. This behavior is caused by the shape of the potential wall in front of the emitter. The Madelung potential at the ion-crystal surface shows much steeper variations with distance than the image potential in front of a metal surface does.
notes
Field Ion Appearance Energies of CO$^+$ Ions formed at Noble Metal Surfaces

by

W.A. Schmidt and O. Frank

Fritz-Haber-Institut der Max-Planck-Gesellschaft
Faradayweg 4-6, 1000 Berlin 33, West-Germany

From measured integral energy distributions of CO$^+$ ions at 80 K tip temperature on surfaces of Cu, Ag, and Au, respectively, field ion appearance energies were evaluated and found up to 0.5 eV higher than the value expected from usual field ionization. There is a slight increase of the appearance energy with field strength. Under certain field conditions with gold tips two superimposed energy distributions have been observed. Mathematical evaluation resulted in two appearance energies, (i) the already mentioned high value and (ii) the value usually expected for CO$^+$. Pronounced differences of the high appearance energies within the group of noble metals were not observed. There were also no differences in the appearance energies if the surfaces were cleaned before ionization or used after prolonged influence of residual gases. Furthermore, no such shifts of the appearance energies were observed for other reactive gases, for example oxygen.

Although as preliminary interpretation, it is thought that the CO molecule interacts with surfaces of Cu, Ag or Au, cleaned or not completely cleaned, resulting in a binding energy. This binding energy affects the appearance energy. Theoretical treatment might be applied in analogy to field evaporation theory. Another interpretation, assuming CO ionization being non-adiabatic can be excluded because of the results with gold.
notes
Processes of Ion Formation in Field Desorption Mass Spectrometry

F.W. Röllgen, U. Giessmann, H.J. Heinen, and H.D. Beckey
Institute of Physical Chemistry, University of Bonn, D 5300 Bonn, W. Germany

From conventional activated emitters alone no reliable information on the ionization conditions i.e. the effect of field strength and temperature on the ion formation can be obtained, since the adsorption of substances on surfaces of largely different field strengths i.e. on top, on the shank or in between the field enhancing microneedles cannot be sufficiently controlled. However, by using smooth wires, wires with microneedles of different length and single tips as field anodes it was possible to differentiate between various supply, ionization and desorption mechanisms. These different processes and their dependence on the field strength, anode temperature, surface morphology, and the nature of adsorbate will be discussed.
notes
Appearance Energies of Field Ionized and Field Desorbed Polyatomic Sulfur Molecules

by

J.H. Block and R.-G. Abitz

Fritz-Haber-Institut der Max-Planck-Gesellschaft, Faradayweg 4-6, 1 Berlin 33

Sulfur molecules $S_x$ ($2 < x < 8$) are either produced in an electrochemical cell and evaporated onto a tungsten field emitter or are formed by a surface reaction of impinging $S_x$ molecules in a physisorbed layer of the emitter at about 300 K. Field ionization of the $S_x$ vapor and field desorption of $S_x$ surface reaction products are investigated in a quadrupole mass spectrometer and analyzed with regard to mass to charge ratio, ion intensity, and integral energy distribution.

For vapor molecules appearance energies (AP) have been expected to reflect ionization energies (I).

Experimental values are:

<table>
<thead>
<tr>
<th></th>
<th>AP</th>
<th>I</th>
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<tbody>
<tr>
<td>$S_2$</td>
<td>9.33 ± 0.1</td>
<td>9.36 ± 0.05</td>
</tr>
<tr>
<td>$S_5$</td>
<td>8.11 ± 0.15</td>
<td>8.90 ± 0.03</td>
</tr>
<tr>
<td>$S_6$</td>
<td>8.94 ± 0.1</td>
<td>8.97 ± 0.1</td>
</tr>
<tr>
<td>$S_7$</td>
<td>8.97 ± 0.1</td>
<td>8.04 ± 0.03</td>
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In particular for $S_5$ and $S_7$, deviations are higher than experimental errors.

In comparison with vapor molecules, $S_x$ molecules formed by surface reactions display energy losses which are more than 1 eV too small (even at postulated negligible adsorption energy). This deviation is nearly identical for all measured $S_x$ and independent on individual ionization energies. This result indicates that field desorption from the condensed layer involves either diabatic reaction steps or ionization out of intermediate excited reaction states.
Field desorption mass spectrometry (FDMS) has experienced relatively rapid growth during the last decade in its applications to organic chemistry. However, the use of FDMS has been generally restricted to qualitative analyses of specific compounds or mixtures of compounds. The earliest considerations given to quantitative applications of FDMS were directed towards the problem of defining a suitable means for measuring sensitivity. More recent efforts have been directed toward applying FDMS to quantitative problems in mixture analysis and isotope ratio determinations. FDMS shares with other forms of mass spectrometry the very desirable features of high sensitivity coupled with high specificity. These characteristics are often enhanced by the relatively high molecular ion intensities generally observed in FD mass spectra.

This laboratory has developed an interest in applying FDMS to the quantitative analysis of polynuclear aromatic hydrocarbons contained in high pressure liquid chromatographic fractions.

The present paper will present the results of our efforts, both past and present, in investigating sample handling, desorption rate, focusing, and detection made in conjunction with the precision, accuracy and minimum detectable limit of quantitative FDMS analyses.
IONIZATION BY ALKALI ION ATTACHMENT ON THE SURFACE OF ELECTROLYTIC SOLUTIONS

K.H. Ott and F.W. Röllgen
Institute of Physical Chemistry, University of Bonn,
D 5300 Bonn, W. Germany

Electrolytic solutions prepared by mixing concentrated aqueous salt solutions with low vapour pressure organic compounds were found to be particularly suited for surface ionization of unpolar or weakly polar molecules by alkali ion attachment. To this end the mixtures are deposited on field anodes i.e. 10 μm W wires. The attachment reaction results from a field induced and thermally activated charging of the layer with loosely bound alkali ions which are captured by molecules striking the surface.

Optimum results were obtained with emulsifying agents giving (M + alkali)$^+$ ions already at room temperature. This is assigned to a membrane effect i.e. the organic molecules surrounding small H$_2$O-salt droplets are permeable for ions in a high external field.
notes
The interpretation of the total energy distribution observed in photo-induced field emission depends on knowing the temperature rise of the illuminated tip. We are investigating the temperature rise of a tungsten field emitter by studying the current observed in direct field emission when the tip is illuminated by a focused laser beam of wavelength \( \lambda = 647.1 \) nm. Our data, when analyzed on the basis of a simple theoretical model similar to that discussed by Körnendi\(^1\), determine accurately the Fermi energy of the field emitter. Above the Fermi energy, our data are found to be in good agreement with the theoretical model, and from the data we can estimate the temperature rise of the tip. Below the Fermi energy, the uncertainty in our data is considerably greater because of increased shot noise. An analysis of our preliminary data in this energy range shows a sizeable discrepancy from the predictions of the model mentioned above. Recently Sullivan, Cutler, and Lucas\(^2\) have suggested that thermally-enhanced field emission may explain the remarkable rectification characteristics of metal whisker diodes at infrared frequencies. In order to establish an experimental basis for an important aspect of their theory, work is underway to relate the observed temperature rise of the field emission tip for a given incident laser power to the predictions derived in their paper.

Field Emission Spectroscopy of Bismuth on W(100)

J. P. Jones,
University College of North Wales,
School of Electronic Engineering Science,
Dean St., Bangor, Gwynedd, LL57 1UT.

Abstract

An examination of the FEED spectra from lead and iron on W(100) led to the proposal\(^1\) that features in the enhancement curves could be identified with energy levels in the isolated atom. To further test this proposal bismuth was chosen as an adsorbate because the atom has a relatively simple energy level structure in the small energy range which is accessible to our retarding field spectrometer.

The initial reduction in \(\Phi(100)\) by adsorbed bismuth is accompanied by a reduction in the surface state resonance and a weak feature emerges centred at 0.4eV below the Fermi level. As the coverage approaches one monolayer a stronger resonance emerges at 0.4eV and is substantially independent of coverage over a range of at least one monolayer. At coverages exceeding two or three monolayers a resonance appears at \(\sim 0.6\) eV.

These effects will be illustrated and their possible origin discussed.

\(^1\)J. P. Jones and E. W. Roberts, submitted to Surface Science.
notes
A NOVEL METHOD OF PROBE HOLE CURRENT MEASUREMENT

Rocco Di Foggio and Robert Gomer
The Departments of Physics and Chemistry
and The James Franck Institute
The University of Chicago
Chicago, Illinois 60637

Upon passing through the probe hole, field emitted electrons are post-accelerated to strike a phosphor coated glass surface thus producing light which passes through the glass and is detected by a photomultiplier tube. The phosphor is backed by a 500 Å thick evaporated aluminum film to prevent stray light from reaching the PM tube. The advantages of this external detection system over a Channeltron include simpler field emission tube construction, easy replacement of the detection system and a higher permissible bakeout temperature of the vacuum chamber. The overall gain and signal-to-noise are comparable or better than with a Channeltron. Work function measurements done in this way are in good agreement with those done with a Channeltron. As for the choice of a phosphor, an inorganic fast-decay (~400 nanosecond) phosphor such as P47 (Cerium activated Yttrium Silicate) is bakeable to 450°C, suitable for UHV, and can respond to high frequency fluctuations in field emission currents. Its light output peaks around 3900 Å which makes it well suited for use with a standard S-11 photocathode.
FIELD EMISSION FROM THE SUPERLATTICE PLANES OF
ORDERED Ni\textsubscript{4}W

JOAQUIN LIRA-OLIVARES
INMETAL and Department of Materials Sciences -
Universidad Simón Bolívar, Caracas - Venezuela

We utilized the layered structure presented by the (011),
(101) and (112) planes of Ni\textsubscript{4}W phase. Such a structure
provided similar conditions to those encountered in adsorp-
tion experiments (a substrate with adsorbed layers).

Changes in electron current emission were measured after
 evaporating atomic monolayers from the (011) plane of or-
dered Ni\textsubscript{4}W specimens. Some of the changes were found to
be of the same order of magnitude as those expected by
work functions changes due to adsorbed atoms (applying the
dipolar model). However, there were some large current e-
mission changes notice when a W monolayer was evaporated
from the (011) plane. These changes could be compared to
those predicted by Alferieff in this tunneling resonance
model.

It was also observed that the intensity of the electron e-
mission current depended on the number of nickel monolayers
above the underlying tungsten layer.
THE STUDY OF MATTER TRANSPORT ON METAL SURFACES

BY FIELD EMITTERS

(A REVIEW)

M. Drechsler
Centre de Recherche des Mechanismes de la Croissance Cristalline
CNRS, Universite d'Aex-Marseille, Saint-Jerome, France
Build-up Process of Field Emission Tip of Tungsten and Molybdenum with Silicon Adsorption

K. Chang and T. Iwata

Department of Electronics, Faculty of Engineering, Tokai University, Hiratsuka, Kanagawa, JAPAN

The field emission tips of Tungsten (W) and of Molybdenum (Mo) covered by Si show some characteristic build-up (BU) respectively according to the coverage of Si(e), the temperature of tip(T), and the field strength applied, in FEM. Though the crystallographies of W and of Mo resemble each other, the BU process or the BU pattern is different between on W and on Mo.

On W tip a symmetrical protrusion grows on the (110) plane, in the range T = 1200 - 1400K and at the coverage e \( \approx 4 \) /1,2/.

On Mo tip, under the almost same conditions as for the W, it shows a BU of (211) plane, and no BU of (110) plane similar to that of Si/W appears.

At the same Si coverage and tip temperature as above mentioned, a more sharp protrusion occurs on the (320) plane of W tip when a higher field is applied. On the contrary a protrusion grows on the (311) plane of Mo tip. In the temperature range beyond 1400K, both the BU of W and of Mo tip covered by Si reveal similar features.

The BU of (110) plane of W tip has been interpreted as due to the growth of Si or Si-W complex on the plane rearranged. In the BU of Si/Mo, there was no evidence of the rearrangement on the (110).

Besides the BU phenomena, the adsorption and crystal growth of Si on W or on Mo shows some differences respectively, especially for the shape of the nucleus of crystal and for the zone where the nucleation occurs /3/.

On Mo, when the Si is deposited at room temperature on one side of the tip and diffused over the tip surface by heating at the temperature below 1000K, some stable Si layers cover the last layer which has been diffused previously, regardless of the plane index. No such multi-layers are observed on the W tip. The interaction between Si and Si on the substrate, therefore, seems to be different on the W opposed to the Mo.

/1/ The coverage e of Si was defined from the characteristics of work function versus Si deposition.


Crystal Shape Changes by Adsorption

by

M. Drechsler and A. Müller

Centre de Recherche des Mécanismes de la Croissance Cristalline,
CNRS, Université d'Aix-Marseille, Saint-Jérome, France

+Fritz-Haber-Institut der Max-Planck-Gesellschaft
Faradayweg 4-6, 1000 Berlin 33, West Germany

The heating of a metal (without electric field) changes the crystal shape by adsorption influenced diffusion (faceting) as known from many microscopic observations including FEM and FIM. A better study of this phenomenon requires better informations on (1) the crystallography of the appearing faces (2) the adsorbed substances, (3) the coverage degrees (4) the adsorption structures and (5) the surface energy changes. We have studied this phenomenon in particular for carbon on tungsten. Equilibrium states were produced on tungsten crystals depending on temperature \((1200 < T < 2600 \text{ K})\) and coverage \((0 < \Theta < 10^{15} \text{ atoms/cm}^2)\). Absolute coverages were determined by the method of Piquet et al \(/1/\). Changes of the geometry of the tip crystal were measured using FEM and FIM, to determine the surface free energies of different faces by Wulff's construction respectively by an analogous equation. The results allow an energy classification of different C on W adsorption sites or states.

A first attempt is described for a new method to determine adsorption structures by a FEM-FIM technique, i.e. using data on the coverages and the crystallography of the reconstructed faces. A few structure models are presented. The carbon atoms adsorb preferentially along surface steps respectively on surface sites where the adatoms can enter as deep as possible into the surface. The old idea that carbon forms a two dimensional graphite structure on \(\{334\}\) has probably to be revised. According to a new hypothesis, the \(\{334\}\) faces are formed by a less dense carbon adsorption structure. Sometimes terrace structures appear whose step height exceeds the normal atomic height. Such structures can be explained as part of special equilibrium shapes of minimum potential energy. One of the conclusions is: The study of chemisorption at temperatures where surface self-diffusion occurs is incomplete if this does not include investigations of the reconstructed adsorption structures with its changes of the size and energy of different faces.

\(/1/\) A. Piquet, G. Pralong, H. Roux, R. Uzan, M. Drechsler
Le Vide 185 Suppl., 1977, p. 376
notes
EFFECTS OF GLASS ELECTROLYSIS ON
ELECTRICAL BREAKDOWN IN HIGH VACUUM

Karl G. Hernqvist
RCA Laboratories
Princeton, New Jersey 08540

Glass is a commonly used high voltage insulator in vacuum. Under the influence of high electric fields significant ionic conduction (electrolysis) in the glass takes place at moderately high temperatures (~100°C), particularly in high-lead and alkali glasses. The ions may drift to the surface, where they affect high voltage insulation.

Measurement of the ionic conductivity in 0137 glass will be described. It is shown that the conductivity is appreciable at 100°C and increases rapidly with temperature. Extended electrolysis tests show that alkali metals and lead accumulate on the glass surface at the negative electrode at the rate of about 1/3 monolayer per hour at 100°C and a field of 10 kV/cm. Both solid contacts and free electron flow to the glass were used to provide "negative contact" to the glass.

The pre-breakdown phenomenon of field emitted electrons from the insulator-cathode junction providing contact to the glass surface for ionic conduction in the glass is discussed. Alkali metals on the surface of the glass quickly diffuse over the surface yielding a high surface conductivity which may lead to desorption flashover. Experimental verification for these processes will be presented.

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Regenerative Processes Leading to Surface Flashover in Vacuum*

R. A. Anderson and J. P. Brainard
Sandia Laboratories, Albuquerque, New Mexico 87185

Charging of the surface of an electrically stressed insulator in vacuum by an avalanche of secondary emission electrons is well established,\textsuperscript{1} and there is experimental evidence that the avalanche is involved in establishing the flashover path.\textsuperscript{2,3} However, the transition from the low-current prebreakdown secondary emission avalanche to a highly conductive plasma channel has not been satisfactorily explained. A model based on electron-stimulated desorption of gas from the insulator surface is presented which accounts for the transition to breakdown in fast-pulsed surface flashover. The time delay preceding breakdown in this model is largely determined by the rate of accumulation of a layer of positive charge, composed of desorbed ions and desorbed neutrals ionized while drifting through the layer of electrons in the avalanche. Eventually the positive charge enhances the electric field near the cathode end of the insulator and a regenerative growth of desorption and ionization occurs. As the electric field rises other regenerative processes become possible which rapidly lead to breakdown. Time delays according to this model are in good agreement with our experimental results for polymethyl methacrylate and alumina ceramic insulators which were of the order of 10 mm long and broke down in 4 ns at $10^7$ V/m. In addition, this model accounts for the approximate proportionality between the time delay and the inverse square of applied voltage for time delays between 1 and 20 ns, as well as the more rapid increase in time delay with decreasing voltage beyond 20 ns.


*Work supported by U.S. Department of Energy.
notes
Field Ion Microscopy of Silicon

T. Sakurai
Department of Physics
The Pennsylvania State University
University Park, PA 16802

and

A. J. Melmed
Surface Science Division
National Bureau of Standards
Washington, DC 20234

ABSTRACT

We have been investigating <111> oriented silicon whiskers using field ion microscopy (FIM), atom-probe FIM, and field desorption microscopy (FDM), and have found that silicon behaves quite differently from a metal in the presence of a high field. For instance, Si field evaporates as random clusters of Si ions, when evaporated at or below room temperature in vacuum, instead of evaporating orderly from the surface kink sites. This anomaly in field evaporation is believed to be due to a combination of field penetration in the near-surface bulk and the unique bonding geometry of the tetrahedral diamond structure. We also have observed a strong effect of light illumination on the field ion image intensity and quality, unique to semiconductor emitters. This mysterious effect, known for some years, can be summarized as follows:

(1) The intensity of a FI image using H₂, He, Ar and Kr imaging gases increases markedly (a factor of up to 10) by infrared illumination when a Si emitter surface is not fully developed.

(2) As field evaporation continues, the illumination effect decays out gradually.

(3) When the Si surface is completely developed to show a fully ordered pattern, there is practically no illumination effect.

We show that this effect results from an increase in surface potential, which causes an increased surface field due to the sharp resistance drop across the surface oxide (SiO₂) layer, all due to the well-known photoconductivity effect.

* Partially supported by NSF.
Handling damage of FIM specimens caused by field corrosion.

H-O Andrén, A Henjered and H Nordén
Department of Physics, Chalmers University of Technology
S-402 20 Göteborg, Sweden

Many polymers acquire substantial surface charges when making sliding contact with metals (1). The time for the subsequent discharge may be very long, but if the material is not kept under vacuum, the surface charge gets neutralized after some time by a layer of ions from the surrounding atmosphere. This layer is loosely bound and it can easily be removed by friction (2).

Consequently, if an unshielded field-ion specimen is passed through a polymeric gasket (e.g. a Viton O-ring or a PTFE sliding seal), the tip may be exposed to a considerable field strength, and specimen damage from field corrosion is very likely to occur. The shape of a damaged specimen is approximately that of a truncated cone. The edge of the cone is usually sufficiently sharp to give a ring-shaped image in the field-ion microscope, and if the damage is severe, it is not possible to obtain a fully developed image.

These findings have important consequences for the design of specimen changers for field-ion and atom-probe instruments. Examples of dangerous and safe designs will be shown.

A STUDY OF SPINODAL DECOMPOSITION IN Cu-Ti ALLOYS BY MEANS
OF THE FIM-ATOM-PROBE AND FDM

R. Wagner
Institute for Metal Physics
University of Göttingen
Göttingen, West Germany

In literature there exists a controversy about the precipitation
mechanism in Cu-Ti above 330°C. Some authors propose a classical
nucleation and selective growth mechanism,\textsuperscript{1} others propose a spinodal
mechanism.\textsuperscript{2} From a preliminary study of the FIM-atom-probe in
variously heat-treated Cu-Ti alloys, we concluded that the decom­
position reaction is the spinodal one. The mechanical hardening of
these alloys supports this conclusion, and one should be able to
quantify the hardening mechanism in modulated structures by measuring
the modulation amplitude and wavelength with the FIM atom-probe.

1. Tsujimoto, Trans. JIM 1, 445 (1975)
FIM Study of Mo-Re Alloys

K. Teramoto, H. Morikawa, Y. Yashiro, N. Igata* and A. Kohyama*

Department of Coordinated Science
Nagoya Institute of Technology
Gokiso-cho, Showa-ku, Nagoya 466, Japan

*Department of Materials Science
Faculty of Engineering
University of Tokyo
7-3-1 Hongo, Bunkyo-ku, Tokyo 113, Japan

FIM observations were made of molybdenum alloys (Mo-0.03 at% Re-0.1 at% C, Mo-0.3 at% Re-0.2 at% C, Mo-3 at% Re-0.1 at% C). Many additional bright spots scattered on the usual molybdenum image were characteristic of the alloy images. The concentration of bright spots obtained on (110) and {121} planes was about 10 times larger in the Mo-3 at% Re alloy than in the Mo-0.3 at% Re alloy, and also in Mo-0.3 at% Re alloy than in Mo-0.03 at% Re alloy, suggesting that these characteristic bright spots came from the rhenium atoms. However, the concentration of bright spots in the Mo-0.3 at% Re alloy images was about 0.5%, somewhat higher than that of the rhenium atoms themselves. This difference may imply the partial contribution of carbon atoms to the bright spots.

The distribution of bright spots was also studied in a circular area on a (110) plane. A series of images obtained by sequential field evaporation gave a spacial distribution.

Similar observations were carried out on specimens annealed at 1800°C for 1 hr. There was no noticeable difference in concentration and distribution of the bright spots between these as-rolled and annealed specimens.
notes
Field-Ion Microscopic Study on Precipitation Processes in Fe-Cr and Al-Cu Alloys

Tetsuo Abe and Ken-i-chi Hirano
Department of Metallurgy, Materials Science and Metal Processing,
Faculty of Engineering, Tohoku University, Aoba, Aramaki, Sendai,
980 JAPAN

The field-ion microscopy can provide us with more direct informations on atomic processes of phase transformations, such as the spinodal decompositions and formation of the G.P. zones, than the transmission electron microscopy.

In Fe-Cr alloy systems, the spinodal decomposition can not be fully studied by the transmission electron microscopy, because it can not distinguish the Fe-rich zones from the Cr-rich zones, because of the similarity of Fe and Cr atoms in the atomic radii and the scattering amplitudes for electron. However, the field-ion microscopy will be a powerful tool, because it can provide us with images of individual atoms in which Fe atoms can be distinguished from Cr atoms.

In some Al alloys, the formation processes and the structure of the G.P. zones have been proposed based on the X-ray analysis, but the atomic structure of the G.P. zones cannot be displayed clearly by the transmission electron microscopy, because of the effect of the strain field around the G.P. zones. Here again, the field-ion microscopy may be useful.

We have succeeded in observation of the modulated atomic arrangement in Fe-45 at.% Cr alloy formed by the spinodal decomposition and the G.P. zones (mono-layer discs of Cu atoms) in Al-1.5 at.% Cu alloy.
Composition of carbide particles in stainless steels

H-O Andrén, A Henjered and H Nordén
Department of Physics, Chalmers University of Technology
S-402 20 Göteborg, Sweden

The creep strength of stainless steels containing MC-type particles is at present being investigated. In particular, the effect of adding two carbide forming elements to the steel is studied. As a part of this study, atom-probe analyses of the carbide particles are being made.

MC-type particles are non-stoichiometric in that some carbon is missing (the stability range of TiC is 32 to 49% C to mention one example). Some carbides form ordered structures at certain compositions (e.g. VC$_{0.84}$), and it is believed that ordering contributes to stability against particle coarsening during high temperature service.

Analysing these particles in the atom-probe, we have found Ti, V and Nb to evaporate from particles doubly and triply charged (Nb also as Nb$^{4+}$), but no metal-carbon complex ions of any kind have been detected.

The analyses made so far show, that small particles contain considerable amounts of chromium (but little iron). The particles were analysed using a large acceptance angle which gives a greater yield and thus a more accurate measure of the composition. In this way, estimates could also be made of the variation in composition within the particle and at the particle – matrix interface.
ATOM PROBE STUDIES ON SURFACE SEGREGATION IN THE BINARY ALLOY TUNGSTEN - MOLYBDENUM

M. Leisch

Institut für Angewandte Physik, Technical University Graz, Austria

In the past evidence has been obtained indicating that the surface composition of a binary alloy can differ from the bulk composition. Experimental work mainly with Auger spectroscopy\(^1\) and recently atom probe studies\(^2\) have been done on this subject. In addition there is an abundance of theoretical work\(^3\).

In this case an imaging atom probe has been used to analyze the top surface layers. A probe diameter of about 50 \(\mu\)m and evaporation rates ranging from 1-5 events per pulse have been used.

A quantitative analysis of the TOF-spectra of unannealed specimen give a surface composition close to the bulk composition. After heat treatment in situ remarkable changes in the abundance of tungsten and molybdenum ions occurs. The obtained surface composition depends on annealing temperature and the duration of the heat treatment. The preliminary results will be discussed with respect to the cited works.

1) W.M.H.Sachtler, Le Vide 163-165 (1973) 19
2) T.T.Tsong, Yee S.Ng, S.V.Krishnaswami Appl.Phys.Let. 32 (1978) 778
notes
Zones in Aluminium Alloys

E.D. Boyes

University of Cambridge and University of Oxford

The techniques\textsuperscript{1,2} for imaging and analysing aluminium-based materials in the FIM/Atom-probe will be reviewed briefly and their applications to the study of specific materials described.

The initial low temperature homogeneous precipitation produces zones which may continue to contain some aluminium, in, for example, Al-Cu, Al-Zn-Mg and Al-Mg-Si but not Al-Si alloys, and it has been suggested that both the structure and morphology of the zones may be a sensitive function of the alloy composition.

The advantages of the FIM are:
(a) the sensitivity and relatively simple interpretation of image contrast features a few nm in size
(b) the resolution of size measurements
(c) simplified micro-analysis, particularly for matrix elements in those precipitates which are more refractory than the matrix.

The fine-scale distribution ($10^{17} - 10^{19}$ per cc) is convenient for FIM/Atom-probe.

References


An "Imaging Atom-Probe"
For The Analysis of Electroplatings

Michael Martinka and S. Brooks McLane
The Pennsylvania State University, Physics Department
University Park, Pennsylvania 16802

This paper describes the design of an "Imaging Atom-Probe"[1,2,3] which will display the spatial distribution of a selected mass constituent of an electrodeposit. Within the mass spectrometer's flight path, field evaporated ions are retarded and then post accelerated to increase flight time and reduce angular timing errors (flat 75mm chevron detector), respectively.

Particular attention is given to the waveshapes of the evaporation pulse and the detector gating pulse. The gate pulse is applied to a stainless steel support ring at the perimeter of the channel plate, and propagates through a conductive coating toward the center of the detector surface. B. Nahrath, M. Shakhatre and G. Decker[4,5] have simulated these transmission properties by a 40 element distributed RC system, and they obtained a delay time of 8 nanoseconds (ns), between the perimeter and a 1 inch diameter circle about the center. This delay together with the attenuation and broadening (Fig. 2, Ref. 4) would limit the extent to which the distribution of a selected mass species can be imaged simultaneously. We have directly measured (1 Meg. ohm, 1.5pf Probe) the transmission properties of a 75mm diameter Inconel coated, with a resistance of 10 ohms from perimeter to center, channel plate. Both 7 and 14ns wide pulses with rise times of .5ns, terminated into 50 ohms, were applied to the channel plate, and waveforms were observed at the terminator with the plate not connected, the channel plate perimeter and at the center of the detector. The pulse at the perimeter is attenuated and somewhat broadened. This pulse shape is maintained as the pulse propagates to the center of the detector. Expanded time sweeps, 5ns/cm, show less than a 1ns delay time between the perimeter and the center. This was simulated by a capacitor, 130-150pf, in series with a small resistor, 15-20 ohms, with an RC constant of 2-3ns. Note, to obtain a rise and fall time of 3ns at this channel plate the length of transmission line (cap. to ground-30pf/ft) from the pulser to the terminator-channel plate system must be kept short, less than one foot.


PERFORMANCE AND APPLICATIONS OF AN IMAGING ATOM PROBE

M.K. Miller*, T.J. Godfrey, P.A. Beaven, P.R. Williams, K.M. Delargy and G.D.W. Smith

University of Oxford, Department of Metallurgy and Science of Materials, Parks Road, Oxford OX1 3PH, England.

An Imaging Atom Probe has been developed for metallurgical research. The design features (1) a 76.2mm double curved channel plate assembly with a flight path of 118mm, (2) a continuously variable nanosecond gate and delay circuit of particularly simple design, and (3) facilities for the direct display and recording of mass spectra.

Experimental work has mainly been directed towards gaining a better understanding of factors controlling the quantitative accuracy of Atom Probe results. Systematic studies have been carried out of the effects of a number of variables on the mass spectra produced from alloys. The main variables studied were evaporation pulse fraction and pulse repetition rate. Both of these factors require careful control if reproducible results are to be obtained. The effects of residual gases and image gases on the mass spectra have also been investigated. The necessity of carrying out analyses under the best possible vacuum conditions is emphasised.

Initial metallurgical applications of the instrument will also be described. The distribution of alloy elements in steels and nickel superalloys is currently being investigated.
Quantification of Atom-Probe FIM Data
T. T. Tsong and Yee S. Ng
Physics Department
The Pennsylvania State University
University Park, Pennsylvania 16802

It is shown that the atom-probe signals of a sample do not give directly the true composition of the sample. A statistical method has been devised to convert the apparent composition to the true composition.\(^1\) The analysis is valid when (a) the field evaporation rate is low enough that each H.V. pulse removes only a small fraction of a surface layer, and (b) the pulse-dc voltage ratio is high enough that field evaporation occurs only during the pulses. Under such conditions, the average number of signals of the \(i\)th species detected per each H.V. pulse is given by

\[
\bar{n}_i = \sum_{n=0}^{\infty} \sum_{n_i=0}^{n} \left( 1 - (1 - e_t e_d)^n \right)^{n_i} \frac{n!}{n_i! (n-n_i)!} \left( 1 - (1 - e_t e_d)^n \right)^{n_i} P_{n_i} (n) P_n (\bar{n}) , \quad \text{or}
\]

\[
\bar{n}_i = \sum_{n_i=0}^{\infty} P_{n_i} (f_i \bar{n}) \left( 1 - (1 - e_t e_d)^n \right)^{n_i} = 1 - \exp(-e_t e_d f_i \bar{n}) .
\]

In the equation \(e_t\) represents the transmission coefficient, \(e_d\) the detector efficiency, \(f_i\) the true fraction of the \(i\)th species, and \(\bar{n}\) the average number of atoms field evaporated per H.V. pulse covered by the probe hole, and

\[
P_{n_i} (n) = \frac{n!}{n_i! (n-n_i)!} f_i^{n_i} (1-f_i)^{n-n_i}
\]

\[
P_{n} (\bar{n}) = \left( \frac{\bar{n}}{n!} \right) \exp(-\bar{n}) .
\]

The apparent fractional abundances \(F\)'s are thus related to the true fractional abundances \(f\)'s by

\[
F_j = \frac{1 - \exp(-e_t e_d f_j \bar{n})}{\sum_{j=1}^{N} 1 - \exp(-e_t e_d f_i \bar{n})}
\]

with \(j = 1, 2, \ldots N\) for an \(N\) - components system.

The details of how to use this statistical analysis to practical problems will be discussed. Using the analysis, the true composition depth profiles of dilute alloys are obtained with a single atomic layer resolution in a surface segregation study.\(^1\) We will demonstrate how miss interpretation of data can result without a proper statistical analysis.

\(^*\)This work has been supported by NSF Grant No. DMR-76-11418.

\(^{1}\)T. T. Tsong, Yee S. Ng and S. V. Krishnaswamy, Appl. Phys. Letters 32, 778 (1978).
notes
The surface segregation in dilute alloys can be investigated with the ToF atom-probe FIM with a single atomic layer resolution. The technique has now been successfully applied to study the segregation of Cr atoms to the surface of stainless steel 410. After a surface was developed by field evaporation, it was annealed at 500 ± 50°C for three to five minutes in the absence of an applied field to equilibrate the surface composition. The tip was then quenched down to 80°K. A thermal end form developed after each annealing. Atom-probe signals were subsequently collected by slow field evaporation of the top surface layer and the underlying layers. By using the statistical analysis reported, the apparent composition obtained directly from the atom-probe data was then converted into the true composition.

Cr atoms were found to segregate to the surface of stainless steel 410 at that temperature. The first layer concentration of Cr in the \{110\} plane was found to be 38.5 ± 12.5% and 63.4 ± 10.2% respectively for samples with average near surface layers Cr concentrations of 6.3 ± 2.1% and 11.9 ± 2.5%. The heat of segregation of Cr to the \{110\} plane of stainless steel 410 was derived to be 3.43 kcal/mole and 3.92 kcal/mole respectively from the two sets of data when an ideal solution model of surface segregation is adapted. However, our data fit better with a regular solution model.

We have also detected segregation of Cr atoms to the \{012\} plane of stainless steel 410. Since this plane was not well developed after each annealing, we could not discriminate those signals coming from the top surface layer and those coming from the near surface layers.

In addition, preliminary results from Ni Cu and other dilute alloys will be presented. Application of various theoretical models to the analysis of our data will also be discussed.

*This work also supported by NSF Grant DMR-76-11418.
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In close cooperation with Dr. A. J. Melmed of NBS, we have made some progress in studying semiconductors and insulators. We focus on the following three subjects:

1) Atom-probe mass spectroscopy: To achieve nano-second pulse field evaporation of semiconductors, we used GaAs emitters heavily doped with Zn\((2 \times 10^{18} \text{ cm}^{-3})\). Although the pulse field evaporation could only be marginally seen by eyes, we obtained ToF atom-probe mass spectra with relatively good yield. Molecular ions such as As\(_2^+\), As\(_4^+\), and Ga\(_2^+\) have been observed. In hydrogen and oxygen, the number of arsenic hydrides and oxides ions detected greatly exceeded that of gallium hydrides and oxides, in consistent with the assumption that on GaAs surfaces, chemisorption of hydrogen and oxygen occurs only on As sites at low coverages. Other interesting features of this study will be presented.

2) Photoillumination effect: It has been found earlier that field ionization above and field evaporation of semiconductors and insulators are enhanced by photoillumination. We found that this effect is very pronounced for silicon oxide surfaces at low temperatures even at a relatively low light level. We now believe that photoconductivity is mainly responsible for this effect. The potential drop across a oxide layer depends on the layer thickness, the tip temperature, and the light intensity. It may amount to a few kV. This quantity can be derived quite accurately from I-V curves. In addition the electron-hole recombination rate, which again depends on both the tip temperature and the oxide layer thickness, and which may amount to several seconds, can be measured from the relaxation time of the FI enhancement. By changing the oxide layer thickness the effect of band bending on the electron-hole recombination rate can be investigated. It is important to note that the effect of band bending is exactly the same as that of field penetration, and can therefore be controlled by applied field. Our study should yield significant information on physics of semiconductors and insulator surfaces.

3) The effect of field penetration on semiconductor and insulator surfaces has been investigated in the past by many theorists. The theory will be extended and adapted to the case where the surface field amounts to a few V/\(\AA\). The effect of field penetration to both field evaporation and field ionization will be discussed.

* This work was supported by NSF Grant-DMR-76-11418.

\(^1\) T. T. Tsong, Yee S. Ng and A. J. Melmed, to be published in Surface Sci.
notes
PROGRESS WITH FIM AND ATOM PROBE INVESTIGATIONS OF NICKEL BASED SUPERALLOYS


University of Oxford, Department of Metallurgy and Science of Materials, Parks Road, Oxford OX1 3PH, England.

Further investigations of the microstructure and fine-scale composition of nickel based superalloys have been carried out using TEM, FIM, T.O.F. Atom Probe and Imaging Atom Probe techniques. These studies follow the initial work on the determination of \( \gamma \) and \( \gamma' \) phase composition in alloy IN939, reported last year (1).

New results to be presented concerning alloy IN939 include the following:

1) Determination of the composition of the ultra-fine secondary precipitates formed during the low temperature (700°C) final ageing treatment. The phase present has been identified as \( \gamma' \), but with a substantially lower cobalt content than in the primary \( \gamma' \) precipitates.

2) Investigation of the initial stages of decomposition of the solution-treated alloy. Computer methods have been devised to follow the localised composition fluctuations occurring during the early stages of \( \gamma' \) formation. Fluctuations in the concentrations of any number of elements can be followed simultaneously.

3) Determination of the composition of the primary MC carbide phase. All the major carbide forming elements (Ti, Zr, Ta, Nb, Cr and W) were found to be present in carbide particles of overall composition \( \text{MC}_{0.88} \). Nitrogen and boron were also present the latter being enriched at the carbide/matrix interface.

Work is also in progress to investigate the effects of prolonged exposure, under stress, at elevated temperatures on the microstructure and properties of superalloys. Highly anisotropic \( \gamma' \) particle shapes have been observed by FIM in crept specimens of NIMONIC 90. Similar work is being undertaken on IN939.

References

notes
FIM AND ATOM PROBE STUDIES OF DEFECTS IN DOPED TUNGSTEN LAMP WIRES

P.A. Beaven, K.M. Delargy, M.K. Miller, P.R. William and G.D.W. Smith

University of Oxford, Department of Metallurgy and Science of Materials, Parks Road, Oxford OX1 3PH, England.

Previous FIM observations of defects in aluminium-potassium-silicon ('AKS') doped tungsten lamp wire (1,2) have been substantially extended. The accumulated evidence shows that a large number of observations in the earlier FIM literature of tungsten concerning the "crossover effect", "dissociated dislocation loops", "horseshoe dislocations" etc., can be ascribed to the imaging of small defects introduced into the tungsten wire during the doping process. These defects fall into two main classes: small voids (believed stabilised by the presence of potassium) and small solid particles, of hitherto unknown composition.

Each of these classes of defect has been investigated using the T.O.F. Atom Probe. In the case of the small voids, attempts at chemical analysis have been unsuccessful, owing to ion-optical aberration effects. In some cases, no substrate ions at all were collected from the region of the void, despite the presence of a large image gas ion current at the detector, thus demonstrating a substantial trajectory difference between the image gas ions and the substrate ions in the region of the void.

Microanalysis of solid particles was more successful. Particles located both in grain boundary and matrix regions have been analysed. The overall compositions obtained were in the range W$_5$O to W$_3$O which correspond approximately to that expected for the $\beta$-tungsten phase. Traces of dopant elements were found in solid solution in these particles, supporting earlier suggestions that the $\beta$-tungsten phase may be of importance in retaining the trace elements within the microstructure during the wire manufacturing processes (3).

References


notes
Round-Robin Atom-Probe Experiment: Preliminary Results

A. J. Melmed and J. J. Carroll
Surface Science Division
National Bureau of Standards
Washington, D.C. 20234

and

S. S. Brenner
U.S. Steel Research Laboratory
Monroeville, Pennsylvania 15146

Following discussions at the 24th IFES in Oxford, we initiated a round-robin experiment intended to provide information about the relative and "absolute" accuracies of atom-probe instruments. A ternary Mo based alloy was chosen as the specimen material, and specimens were sent to all of the participating laboratories for analysis. Details of the specimen selection and preparation procedures will be described, experimental problems will be discussed, and a preliminary analysis of results will be presented.
Angle-Resolved Photo-Emission Studies of Surfaces using Synchrotron Radiation

E. W. Plummer
Physics Department
University of Pennsylvania
Philadelphia, Pennsylvania

Recent developments in angle-resolved photoelectron spectroscopy using synchrotron radiation will be discussed. Experiments which shed light on the nature of surfaces and surface reactions will be emphasized. The combination of prolonged light and angle-resolved collections allows the experimentalist to apply simple symmetry rules to determine the symmetry of each electronic state at the surface. This will be illustrated with examples of clean intrinsic surface states and adsorbate energy levels. In contrast, these same symmetry rules can be used with molecular states of known symmetry to determine the bonding configuration. The continuous range of photon energies available using synchrotron radiation allows one to look at resonances in the continuum, for defraction effects and to determine the photo-ionization cross section. How this information can be used to determine the spatial nature of the electronic states at the surface as well as the bonding configuration of adsorbates will be illustrated.
notes
TOTAL ENERGY DISTRIBUTIONS OF FIELD EMITTED ELECTRONS
AT HIGH CURRENT DENSITY

by
A. E. Bell
and
L. W. Swanson

Oregon Graduate Center
Beaverton, Oregon 97005

ABSTRACT

Measurements of the total energy distributions (TED) have been carried out at high-current density $J = 10^6$ to $10^8$ A/cm$^2$ for a high-work function (built-up (100)W, $\phi = 4.5$eV) and low-work function (Zr/100)W, $\phi \approx 2.5$eV) field emitter. At high values of $J$, both emitters give values of the full width at half maximum (FWHM) of the TED which exceed the values based on Fowler-Nordheim theory by a few eV. This deviation of the FWHM values increases with $J$, is independent of temperature and decreases with emitter radius for a specified beam acceptance angle. The study of these anomalous values of FWHM values extend from 84 to 1975 K.

The broadening of the energy distribution occurs on both sides of the Fermi peak and results in a FWHM of up to 3.5eV for the 2.5eV work function field emitter compared to an approximately 0.8eV calculated value. Similar FWHM values were observed for the 4.5eV work function emitter. The angular intensity of the field emission beam at the highest FWHM values observed was 0.9mA/Steradian in the case of the 4.5eV work function emitter and more than 1.4 mA/steradian in the case of the 2.5eV work function emitter. When the built-up emitter was blunted back so that the emitter field factor decreased from $2.4 \times 10^{-4}$ cm$^{-1}$, the FWHM decreased from approximately 3.1eV to 2.0eV when the emitter was operated at an angular intensity of $\sim 0.9$mA/stadian.

It is believed that coulomb interaction in the vacuum space a few emitter radii in front of the emitter can account for these results, although internal electron energy broadening mechanisms cannot be ruled out.
The thin-film, field-emission cathode [1] is a relatively low-voltage field emitter, and therefore can be expected to operate over long periods without significant damage due to sputtering. Tests in ultravacuum have now been continuing for over 29,000 hours with no measurable change in voltage-current characteristics, and the cathodes have operated successfully at pressures up to $10^{-5}$ torr.

We have recently tested the cathode's tolerance to high pressures of specific gases. The voltage-current relationship of a 5000-tip cathode array was established in a $10^{-9}$ torr vacuum, and the cathode was then exposed to pressures of $10^{-6}$ to $10^{-5}$ torr of argon, neon, helium, hydrogen, ammonia, methane, hydrogen sulfide, air, water, and oxygen. Exposure time varied from minutes to days for the various gases. In all cases, the emission returned to the initial value when the sample gas was pumped away. We will report the voltage-current characteristics under the influence of the sample gases, as well as the recovery characteristics when the the chamber was reevacuated to $\sim 10^{-9}$ torr.

A single, 5000-tip cathode was tested in all ten sample gases and in $10^{-9}$ torr for a total operating time of 1200 hours. At the end of that time, we found that 9% of the tips had failed. A proportionate change in emission current was not noted because the accuracy of the measurement was about ±5%.

notes
Magnetism at Surfaces and Interfaces by Spin Polarized Field Emission

E. Kisker, E. Kuhlmann, M. Campagna

Institut für Festkörperforschung der Kernforschungsanlage Jülich GmbH,
D-5170 Jülich

and

M. Landolt

Bell Telephone Labs, Murray Hill, New Jersey 07974, USA

Magnetic properties of surfaces and interfaces can be studied by complementing a field emission microscope with a probe hole and an electron spin polarization detector. In this talk we discuss experimental results obtained with single crystal Ni and Fe tips and with epitaxially grown Fe and EuS on W(112). In the case of the evaporated films two components of the polarization vector could be determined. We found $P_x = 9.7\% \pm 1.5\%$, $P_y = 36\% \pm 1.5\%$ for emission from Fe along (100). With nickel tips, a negative polarization ($-3\% \pm 1\%$) is observed from (100) planes, and with iron tips $25\% \pm 5\%$ for the 100 planes.

By admission of $H_2$ to full coverage the nickel spin polarization was found to vanish within experimental accuracy. Preliminary results for the epitaxially grown iron indicate on the contrary an increase of polarization.

A tentative explanation of these results based on band theory will be presented.

For EuS deposited on tungsten emitters, magnetic interface effects can be studied, and the "spin-filter"-action will be discussed.
notes
Many-Body and Spin Dependent Surface Potential Effects in Spin-Polarized Field Emission

D. Nagy, P. H. Cutler and E. T. Feuchtwang

Department of Physics
The Pennsylvania State University
University Park, Pennsylvania 16802

A spin dependent generalization of the Appelbaum-Brinkman transfer Hamiltonian method is developed and used to derive an expression for the spin-polarized current density in field emission from ferromagnets. For a given spin orientation, the current density depends on the spectral amplitudes for that spin orientation. The self-energy operators which determine the spectral amplitudes are approximated by local, energy independent, spin-diagonal potentials. Although spin-flip processes are thereby neglected, the detailed variation of the surface potential and an approximate account of many-body effects are included by means of an effective surface potential \( v_g(z) \). \( v_g(z) \) is the self-consistent surface potential of field-free, spin-polarized jellium in the local density approximation. The calculation of \( v_g(z) \) by means of the spin dependent generalization of the Kohn-Sham version of the Hohenberg-Kohn density functional formalism is described and numerical values are obtained for a bulk uniform background density \( \rho_0 \) and bulk spin polarization \( P \) appropriate for ferromagnetic Ni. With \( \rho_0 = 4.05 \times 10^{-2} \) electrons/(Bohr unit) and \( P = 0.20 \), a work function \( \phi = 4.7 \) eV is obtained, which is in good agreement with experimental value for Ni. It is also found that the surface potentials for the two spin orientations differ sufficiently to have a small but significant effect on the spin polarization of the emitted current.

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The Effects of Geometrical Asymmetry and Multiple Image Interactions of the Tunneling Properties of Point Contact Diodes

S. J. Shepherd, N. M. Miskovsky, T. E. Sullivan and P. H. Cutler
The Pennsylvania State University
University Park, Pennsylvania 16802

A. A. Lucas*
Facultes Universitaires N.D. de la Paix
B-5000 Namur, BELGIUM

Point contact diodes have been utilized as detectors, rectification, and frequency-mixing devices in the infrared (1,2). A fundamental application of these devices has been the most precise and accurate determination of the velocity of light (1). Nevertheless the intrinsic mechanism and the properties of point-contact diodes, although of basic interest, are not unambiguously understood. At present there are two models which attempt to explain the nonlinearity and rectification mechanism of the diodes: (1) Tunneling of electron through an intermediate oxide film from whisker to the metal base, i.e., the configuration is considered to be a metal-oxide-metal (MOM) tunneling junction. (2) The nonlinearity and rectification processes are the result of a thermally enhanced field emission (TFE) (2-6).

Recently a new explanation of the rectification mechanism was proposed which takes into account explicitly the geometrical shape of the two electrodes (2,6,7). In a model calculation of the tunneling asymmetry, the geometrical asymmetry was introduced by considering a configuration where the metal point electrode is assumed to be a hyperboloid of revolution positioned from a flat base of the same metal (7). It was found that for a tip radius equal to 100 Å and a tip-base distance of 20 Å the current ratio \(j_F/j_B\) may be as high as 5 suggesting that rectification properties of metal point-contact diodes can be explained without requiring material asymmetry.

In the work to be reported the analyses of the geometrical rectification mechanism has been extended to include, (i) effects of the electron image interaction; (ii) different geometrical configurations of the junction; (iii) the important multiple image interactions due to the close spacing of the metallic electrodes.

To include the effect of the image and multiple image interactions we have obtained, using a Green's function formalism, an exact solution of the potential field for hyperboloidal tip with a planar anode. This is used to construct the potential barrier in the calculation of the tunneling current as a function of bias voltage. Following Schneit's et al., these calculations use a free electron model and WKB transmission coefficient. Direct comparison with the triangular barrier model of Schneit's yields the corrections due to all image-force interactions.

We have also considered a diode configuration of two identical metallic spheres closely spaced (i.e., \(\leq 10\) Å). A complete solution has been obtained for the potential distribution, including the multiple-image interactions using Green's function formalism. The diode model with spherical electrodes has especial importance because experiments using this arrangement (i.e., spherical electrodes) have been conducted at NBS and I-V characteristics for spacings on the order of 10-20 Å have been obtained (8). Although some features of the measured curves exhibit qualitative agreement with planar MOM theory, there is an obvious need for I-V characteristics based on more realistic diode geometries, which the present analyses gives promise of providing.

REFERENCES


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notes
ABSTRACT

The Tokamak Fusion Test Reactor (TFTR) is presently under construction at the Princeton Plasma Physics Laboratory. The device is projected to reach conditions of energy breakeven by injection of greater than 20 MW of 120 keV neutral deuterium beams into a tritium target plasma. Before TFTR becomes operational in 1981, a number of crucial materials problems involving plasma-surface interactions require resolution. Of primary concern among these problems is an understanding of impurity generation mechanisms and the development of conditioning techniques and/or wall materials which might minimize impurity effects. A related problem concerns hydrogen isotope trapping and re-emission from surfaces exposed to plasma. The re-emission of hydrogen isotopes during a discharge strongly affects the evolution of the plasma density. An understanding of the trapping mechanism is particularly important to minimize the tritium wall loading. A variety of surface analytical techniques have been employed to diagnose these problems on existing tokamaks and recent measurements from the Princeton Large Torus (PLT) will be reviewed.
Notes
SURFACE ANALYSIS OF FIELD-EMITTER SAMPLES EXPOSED TO THE PLASMAS OF PLT AND ISX*

G. L. Kellogg and J. A. Panitz
Sandia Laboratories, Albuquerque, New Mexico 87185

ABSTRACT

In an attempt to help characterize the plasma-wall interactions in operating tokamak reactors, the Imaging Atom-Probe mass spectrometer (IAP), the Field-Ion Microscope (FIM), and the Transmission Electron Microscope (TEM) have been used to analyze the surface and near-surface region of field-emitter samples which were placed at the wall position and exposed to the plasmas of the Princeton Large Torus (PLT) and the Impurities Study Experiment (ISX) tokamak fusion reactors. Measurements of the extent of damage to the specimen surfaces, the composition and thickness of deposited surface films, and the depth distribution of low energy plasma species implanted into the near-surface region of the samples have been carried out.

Three sets of samples (two in PLT and one in ISX) have been investigated thus far. Although each set was subjected to different exposure conditions (e.g., type and number of plasma discharges, limiter material used, position of samples with respect to limiters), some consistent results from the analysis of the samples have been found. For example, all of the tips which had direct plasma exposure showed evidence for a change in surface morphology (i.e., lattice damage or deposited surface films), whereas those tips which were placed in the reactor at the same time but shielded from line-of-sight exposure to the plasma were apparently unchanged. Mass spectra obtained with the IAP showed that plasma and impurity species become trapped within the damaged regions and deposited films, but are not implanted into the substrate bulk.

The implications of these results (and others to be presented) as they relate to the plasma-wall interactions in operating tokamaks will be discussed.

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notes
Field Ion Microscopy of Tungsten Irradiated with He\(^+\), O\(^+\) and Ar\(^+\) Ion Particles

N.Igata, S.Sato, K.Shibata and A.Kohyama

Department of Materials Science University of Tokyo
7-3-1 Hongo Bunkyo-ku Tokyo 113, Japan

Hitherto we have investigated FIM observation of tungsten irradiated with C\(^+\) ions. In this study the purity of the used specimen was 99.95\% and annealed at 1800 °C for 2 hrs. For comparison of radiation damage with other charged particles, tungsten wires were irradiated with 80KeV He\(^+\), 300KeV O\(^+\) and 300KeV Ar\(^+\) ions. The irradiation temperature was 30 °C for He\(^+\) and 30 - 200 °C for O\(^+\) and Ar\(^+\). The size distribution of vacancy clusters were investigated after each irradiation. In the case of He\(^+\) irradiation the larger vacancy clusters between 10 - 15Å was not observed. This would be due to less primary knock on energy in He\(^+\) Irradiation compared with the case of O\(^+\) and Ar\(^+\) irradiation. The swelling \(\Delta V/V\) was plotted against the radiation damage. \(\Delta V/V\) showed saturation tendency in the earlier stages above 0.5dpa in the case of O\(^+\) and Ar\(^+\) irradiation, and the value was higher than the case of He\(^+\) irradiation. This would be also due to higher primary knock on energy and the interstitials almost escaped from surface in the earlier stage in the case of Ar\(^+\) and O\(^+\) irradiation.
A Discrete-charge model of a Field-ion Emitter Surface

Richard G. Forbes

University of Aston, Department of Physics, Gosta Green, Birmingham, UK

This paper explores some of the properties of a basic version of a new type of charged-surface model in which the surface atoms in a charged surface are represented in terms of an array of superimposed monopoles and dipoles, together with a distant array of monopoles of the opposite sign. (The distant array is necessary for electrostatic self-consistency.)

The model is a development of earlier work by the present author and by Tsong and Müller, and can be regarded as a significant step forwards from the normal description of charged surfaces provided by classical electrostatistics. The need for such a model seems an unrealised consequence of the Hellman-Feynman theorem.

Exploration of the model brings out a fundamental inadequacy in the concept of "applied field" as used in field-ion theory. The usual treatment is incompatible neither with classical continuous-charge-distribution models nor with discrete-charge models. This fact has significant consequences for theories of field adsorption and field evaporation.

The following tentative conclusions have been drawn from the analysis of an infinite square array:

1. The monopole contribution to field adsorption is less important than the dipole contribution, though it should not be neglected in detailed treatments.

2. Existing calculations of localised field-adsorption binding energies are not electrostatically self-consistent.

3. For a surface atom with a given external field above it, there may exist a theoretical maximum for its polarisation energy that is independent of the proper SI polarisability of the surface atom.

4. Non-linearity in the local field near the surface should in principle be taken into account in field-ion theory.

5. Significant negative field-induced work-function corrections may exist.

6. Estimates of "surface-atom polarisability" drawn from surface diffusion experiments in high fields may be unreliable.

7. Calculations of critical distances may need revision, and the whole question of "where is the emitter's electrical surface" needs re-examination.

The analysis also raises in the author's mind much more general doubts about the usefulness of flat-surface models of a charged surface. It seems much more likely that a good quantum-mechanical theory of charged surfaces would emerge from localised-orbital type models.
notes
Probability density of displacement of adatom on tungsten surface

Yuji Sadakane and Ken-ichi Hirano

Department of Materials Science, Faculty of Engineering, Tohoku University, Sendai, Japan.

By the FIM study of surface diffusion, it has been shown that the migration of the adatom at low temperature is restricted in the edge of the terrace. The edge of the terrace has two kind of role in the migration of the adatom; reflecting or adsorbing boundary. The role of the edge depends on temperature and time of diffusion treatment. When the distance between the edges of the terrace is a we define \( W_N(d,a) \), the probability that the adatom makes a displacement \( d \) after it makes \( N \) jumps by the following equation.

\[
W_N(d,a) = (1-k) W_{N}^{R}(d,a) + k W_{N}^{A}(d,a)
\]

where the symbol \( R \) denotes the case that the adatom is reflected at the edge of the terrace and \( A \) the case that the adatom is adsorbed by the edge of the terrace, and \( k \) is the adsorbing factor \( (0 \leq k \leq 1) \), which depends on temperature and time of diffusion treatment. The value of \( k \) can be estimated by comparing the experimental values of \( W_{N}^{R}(d,a) \) and \( W_{N}^{A}(d,a) \) with the theoretical values of them.

In the present work, the surface self-diffusion on the W (431) plane has been investigated theoretically and by FIM observation.