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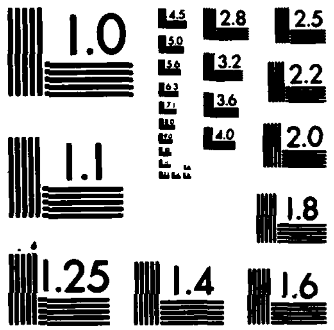

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5th SYMPOSIUM ON APPLIED SURFACE ANALYSIS

by

JOHN T. GRANT

Final Technical Report

April 1984

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DAYTON, OHIO 45469

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John T. Grant

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University of Dayton Research Institute
300 College Park Avenue
Dayton, Ohio 45469

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Adhesion	Electronic devices
Wear	Coatings
Thin films	Corrosion

20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

The 5th Symposium on Applied Surface Analysis was held at the University of Dayton, 8-10 June 1983. This Symposium was held to meet a need, namely to show the continuing transition between basic surface science research and applications of this research to areas of Department of Defense interest. Areas receiving special attention at this Symposium were chemical bonding and reactions at metal-semiconductor interfaces, surface analysis and the tribological processes of ion implanted materials, microbeam analysis and laser

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20. Abstract (Continued)

ionization of sputtered neutrals. Other topics discussed included adsorption, adhesion, corrosion, wear, and thin films. Approximately 110 scientists active in the field of surface analysis participated in the Symposium. Four scientists presented invited papers at the Symposium. There were 29 contributed presentations. The proceedings of the Symposium are being published in a special issue of the journal "Applications of Surface Science" by North-Holland Publishing Company.

FOREWORD

This report describes the 5th Symposium on Applied Surface Analysis held at the University of Dayton, 300 College Park Avenue, Dayton, Ohio 45469, on 8-10 June 1983, under ONR Grant N00014-83-G-0068.

The Symposium was conducted by the University of Dayton with Dr. John T. Grant of that organization and Dr. J. S. Murday of the Naval Research Laboratory, as Co-Chairmen. This report was submitted in April 1984.

SECTION I
INTRODUCTION

The 5th Symposium on Applied Surface Analysis was held at the University of Dayton, 8-10 June 1983. This Symposium was held to meet a need, namely to show the transition between basic surface science research and applications of this research to areas of Department of Defense interest. Areas receiving special attention at this Symposium were chemical bonding and reactions at metal-semiconductor interfaces, surface analysis and the tribological processes of ion implanted materials, microbeam analysis and laser ionization of sputtered neutrals. Other topics discussed included adsorption, adhesion, corrosion, wear, and thin films. Approximately 110 scientists active in the field of surface analysis participated in the Symposium. Four scientists presented invited papers at the Symposium. There were 29 contributed presentations. The proceedings of the Symposium are being published in a special issue of the journal "Applications of Surface Science" by North-Holland Publishing Company.

SECTION II
SYMPOSIUM GOALS

The 5th Symposium on Applied Surface Analysis was held at the University of Dayton, 8-10 June 1983. This Symposium was organized to meet a need, namely, to show the continuing transition between basic research and applications of this research to areas of Department of Defense interest. Workers engaged in basic research and those engaged in applications of surface science research were brought together at the Symposium in an environment suitable for promoting the maximum possible interaction between such workers.

The Symposium was planned and organized by the University of Dayton with Dr. John T. Grant of the University of Dayton and Dr. J. S. Murday of the Naval Research Laboratory as Co-Chairmen. Four invited speakers were selected to cover specific areas of interest. These speakers and their topics were:

1. P. S. Ho
   IBM Thomas J. Watson Research Center

   "Chemical Bonding and Reactions at Transition Metal - Silicon Interfaces"

2. I. L. Singer
   Naval Research Laboratory

   "Surface Analysis and the Tribological Processes of Ion Implanted Materials"

3. J. H. Thomas III
   RCA Laboratories

   "Microbeam Analysis Procedures Using Auger Electron Spectroscopy"

4. N. Winograd
   The Pennsylvania State University

   "Laser Ionization of Sputtered Neutrals: A Novel Approach to Materials Characterization"

Contributed papers on applied surface analysis were also solicited from the research community, resulting in 29 such papers being presented at the Symposium.

It was decided to publish as many of the papers as possible in an appropriate journal, thereby providing a permanent record of the work presented at the Symposium. The journal "Applications of Surface Science" was selected, and the journal editor was pleased to be able to publish such a proceedings. Thirteen manuscripts were submitted for peer review and eventual publication in the proceedings. It is hoped that the proceedings will be published and distributed to all attendees in the Spring of 1984.

The Symposium was advertised through appropriate scientific societies, equipment manufacturers, scientific journals, and at other meetings. It was decided to hold the Symposium on the University of Dayton campus and to encourage attendees to stay in the University apartments in order to provide an informal atmosphere and to optimize the interaction between attendees.

Two events were organized by the University and held in conjunction with the Symposium, namely a two-day short course and a vendor's exhibit. The short course topic was "Techniques for Surface Analysis," and was held immediately before the Symposium to provide an opportunity for attendees to learn the fundamentals of several surface analysis techniques, so that they could gain as much as possible from the Symposium presentations and in their own research. The vendors' exhibit provided an opportunity for attendees to learn about the commercial surface instrumentation that is currently available and what is planned for the future.

SECTION III
PARTICIPANTS

Approximately 110 scientists active in the field of surface analysis attended the Symposium.

The list of participants follows.

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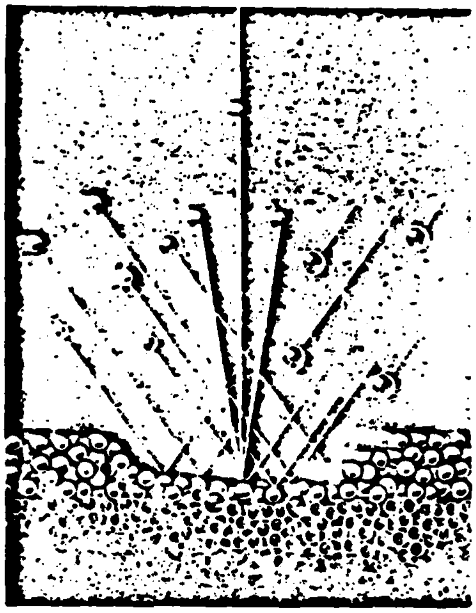
SECTION IV
PROGRAM

The technical program was divided into five sessions. Four of the sessions started with a presentation by one of the invited speakers.

The program organization and the abstracts of the papers presented at the Symposium follow.

5th SYMPOSIUM ON

APPLIED SURFACE ANALYSIS



FINAL PROGRAM

Supported by
OFFICE OF NAVAL RESEARCH
WASHINGTON, DC 20376

8-10 JUNE 1983
UNIVERSITY OF DAYTON
Dayton, Ohio

5th SYMPOSIUM ON APPLIED SURFACE ANALYSIS

8 - 10 JUNE 1983
UNIVERSITY OF DAYTON

GENERAL INFORMATION

The 5th Symposium on Applied Surface Analysis is being supported by the Office of Naval Research. The Symposium will provide an opportunity for workers in all areas of surface characterization to meet and discuss applications of surface analysis. Topics include corrosion and oxidation, adhesion, electronic materials, energy conversion, thin films, thermionic emission, catalysis, and measurement techniques. The aim of this Symposium is to attract participants from universities, government, nonprofit, and industrial laboratories to promote the maximum interaction between workers in different areas.

HEADQUARTERS

The Symposium headquarters will be adjacent to the O'Leary Auditorium located in Miriam Hall on the University of Dayton Campus. Emergency telephone messages can be taken at (513) 229-3437 during the Symposium. All sessions will be held in O'Leary Auditorium.

REGISTRATION

Symposium registration will be held daily in the lobby of Miriam Hall beginning at 8:00 a.m. All attendees *must* be registered.

PROCEEDINGS

If you plan to publish in the proceedings your manuscript (*original,* plus three copies) should be delivered to the Registration Desk during the Symposium.

VENDORS EXHIBITS

A vendor's exhibit will be displayed from noon Wednesday, 8 June through the afternoon coffee break on Thursday, 9 June. Representatives from the major surface instrumentation and ultrahigh vacuum equipment manufacturers will be available to discuss your instrumentation needs. An extended coffee break is scheduled in this area from 3:00-4:00 p.m. on Wednesday afternoon and an informal reception will follow the technical session at approximately 5:00 p.m.

PHYSICAL ACTIVITIES CENTER

The Physical Activities Center on the University of Dayton campus will be available for use by the symposium attendees at a cost of $1 per visit. Various activities at the facility include swimming, jogging, basketball, racquet ball, and handball.

ACCOMMODATIONS

Attendees staying in the University apartments at 361 East Stewart Street can pick up their key upon arrival in Apartment 2A, 363 East Stewart Street. Check out time is before noon on Friday, 10 June 1983.

LIMOUSINE SERVICE

Special arrangements have been made and limousine service is available from Dayton International Airport to the apartment complex at 361 East Stewart Street. The cost is $6/person or $11/person round trip. This service departs the airport every 40 minutes beginning at 5:45 a.m. and the last scheduled run is 11:25 p.m. This service is also available for your return to the Airport. We are asking that you inform the Symposium Secretary of your return schedule a day in advance of your departure.

PARKING

Parking is available on the University of Dayton campus. To obtain a valid visitors pass, kindly let the symposium secretary know when you preregister or by calling (513) 229-3016.

SYMPOSIUM ADMINISTRATION

* * *

SYMPOSIUM SUPPORT

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AGENDA

WEDNESDAY MORNING, 8 JUNE 1983

8:00 a.m.	REGISTRATION, LOBBY OF MIRIAM HALL
9:00 a.m.	INTRODUCTORY REMARKS

SESSION A

Chairman: J. H. Thomas III
RCA Laboratories
Princeton, NJ

A-1	9:20 a.m.	CHEMICAL BONDING AND MATERIAL REACTIONS AT TRANSITION METAL-SILICON INTERFACES P. S. Ho (Invited) IBM Thomas J. Watson Research Center Yorktown Heights, NY
	10:10 a.m.	COFFEE BREAK
A-2	10:50 a.m.	EARLY STAGES OF SILICIDE FORMATION ON W, Ni and Pt SURFACES, THE ATOMIC STRUCTURES AND COMPOSITIONS T. T. Tsong, S. C. Wang, F. H. Liu, H. Cheng, and M. Ahmad The Pennsylvania State University University Park, PA
A-3	11:10 a.m.	CHARACTERIZATION BY AUGER ELECTRON SPECTROSCOPY OF ION IMPLANTS IN SILICON P. A. Lindfors Perkin-Elmer Corporation Eden Prairie, MN and C. F. Hiatt and B. T. McClure Honeywell Bloomington, MN
A-4	11:30 a.m.	APPLICATION OF PIXE IN SPECIFIC SUBLATTICE SITE DETERMINATION OF IMPLANTS IN THE NEAR SURFACE REGION OF GaAs R. S. Bhattacharya* and P. P. Pronko Universal Energy Systems Dayton, OH
	11:50 a.m.	LUNCH VENDORS EXHIBITS OPEN

*Denotes presenting author.

WEDNESDAY AFTERNOON, 8 JUNE 1983

SESSION B

Chairman: P. S. Ho
IBM Thomas J. Watson Research Center
Yorktown Heights, NY

B-1	1:30 p.m.	MICROBEAM ANALYSIS PROCEDURES USING AUGER ELECTRON SPECTROSCOPY J. H. Thomas III (Invited) RCA Laboratories Princeton, NJ
B-2	2:20 p.m.	STRUCTURE OF CARBON FILMS FORMED BY THE PLASMA DECOMPOSITION OF HYDROCARBONS M. P. Nadler, T. M. Donovan, and A. K. Green Naval Weapons Center China Lake, CA
	2:40 p.m.	COFFEE BREAK - EXTRA TIME ALLOWED FOR VENDORS EXHIBITS
B-3	3:40 p.m.	A NOVEL CMA ALIGNMENT TECHNIQUE FOR AUGER SPECTROSCOPY J. D. Geller JEOL Peabody, MA
B-4	4:00 p.m.	TEMPERATURE DEPENDENCE OF THE SURFACE PROPERTIES OF IMPREGNATED DISPENSER CATHODE B. C. Lamartine*, W. V. Lampert, and T. W. Haas Air Force Wright Aeronautical Laboratories Wright-Patterson Air Force Base, OH
B-5	4:20 p.m.	AUGER SURFACE COMPOSITION AND ADHESION CHARACTERIZATION OF GALVANIZED STEEL B. M. Joshi and R. F. Kiesel Ford Motor Company Dearborn, MI
	4:40 p.m.	INFORMAL RECEPTION

THURSDAY MORNING, 9 JUNE 1983

	8:00 a.m.	REGISTRATION, LOBBY OF MIRIAM HALL

SESSION C

Chairman: N. Winograd
The Pennsylvania State University
University Park, PA

C-1	8:30 a.m.	SURFACE ANALYSIS, ION IMPLANTATION AND TRIBOLOGICAL PROCESSES AFFECTING STEELS I. L. Singer (Invited) Naval Research Laboratory Washington, DC
C-2	9:20 a.m.	CHARACTERIZATION OF TITANIUM, MOLYBDENUM AND YTTRIUM ION IMPLANTED METAL SURFACES S.-S. Lin Army Materials and Mechanics Research Center Watertown, MA
C-3	9:40 a.m.	SURFACE ANALYSIS STUDIES ON THE CORROSION RESISTANCE OF Mo IMPLANTED Al ALLOYS M. V. Zeller* and J. A. Kargol University of Notre Dame Notre Dame, IN
	10:00 a.m.	COFFEE BREAK
C-4	10:40 a.m.	AES AND RBS ANALYSIS OF LASER-MIXED Cr AND Ni MULTILAYER FILMS ON Cu ALLOYS I. Sawchyn* and C. W. Draper Western Electric Princeton, NJ
C-5	11:00 a.m.	THE ADSORPTION AND THERMAL DECOMPOSITION OF TRICRESYL-PHOSPHATE (TCP) ON IRON AND GOLD D. R. Wheeler* NASA Lewis Research Center Cleveland, OH and O. D. Faut Wilkes College Wilkes-Barre, PA
C-6	11:20 a.m.	THE EFFECT OF WEAR ON SURFACE TEXTURE R. B. Zipin The Bendix Corporation Dayton, OH
C-7	11:40 a.m.	FRACTAL, SELF-SIMILAR SURFACES P. Pfeifer, D. Farin, and D. Avnir* The Hebrew University of Jerusalem Jerusalem, Israel
	12:00 p.m.	LUNCH

THURSDAY AFTERNOON, 9 JUNE 1983

SESSION D

Chairman: I. L. Singer
Naval Research Laboratory
Washington, DC

D-1 1:30 p.m. LASER IONIZATION OF SPUTTERED NEUTRALS: A NOVEL APPROACH TO MATERIALS CHARACTERIZATION
N. Winograd (Invited)
The Pennsylvania State University
University Park, PA

D-2 2:20 p.m. QUANTITATIVE ANALYSIS OF SEGREGATION AT THE SiO_2/Si-INTERFACE BY SIMS AND RBS
P. Eichinger, E. Frenzel, and H. Ryssel
Fraunhofer Institüt für Festkörper Technologie
West Germany
and
H. Frenzel*
Atomika Technische Physik GMBH
Munich, West Germany

2:40 p.m. COFFEE BREAK, VENDORS EXHIBITS CLOSE

D-3 3:20 p.m. CHEMISTRY OF GLASS-CERAMIC TO METAL BONDS IN HEADERS FOR USE IN INITIATORS AND ACTUATORS IN DETONATOR SYSTEMS
W. E. Moddeman*, S. M. Craven, and D. P. Kramer
Monsanto Research Corporation
Miamisburg, OH

D-4 3:40 p.m. AUGER AND DEPTH PROFILE ANALYSIS OF SYNTHETIC CRYSTALS FOR DISPERSION OF SOFT X-RAYS
K. D. Rachocki*, P. N. Arendt, D. R. Brown, and R. W. Springer
Los Alamos National Laboratory
Los Alamos, NM

D-5 4:00 p.m. MOLECULAR ORIENTATION AND STRUCTURE OF COPPER-BENZIMIDAZOLE FILMS
H. G. Tompkins
Bell Telephone Laboratories
Columbus, OH
and
D. L. Allara and G. A. Pasteur
Bell Telephone Laboratories
Murray Hill, NJ

D-6 4:20 p.m. EMISSION FTIR ANALYSES OF THIN MICROSCOPIC PATCHES OF JET FUEL RESIDUES DEPOSITED ON HEATED METAL SURFACES
J. L. Lauer and P. Vogel
Rensselaer Polytechnic Institute
Troy, NY

4:40 p.m. CLOSE

FRIDAY MORNING, 10 JUNE 1983

8:00 a.m. REGISTRATION, LOBBY OF MIRIAM HALL

SESSION E

Chairman: B. C. Lamartine
Air Force Wright Aeronautical Laboratories
Wright-Patterson Air Force Base, OH

E-1 8:30 a.m. AN XPS STUDY OF THE ADSORPTION OF OXYGEN AND WATER VAPOR ON CLEAN LITHIUM FILMS
J. R. Hoenigman*
University of Dayton Research Institute
Dayton, OH
and
R. G. Keil
University of Dayton
Dayton, OH

E-2 8:50 a.m. APPLICATIONS OF BREMSSTRAHLUNG-INDUCED AUGER LINES FOR THIN FILM ANALYSIS
D. S. Becker* and R. G. Hayes
University of Notre Dame
Notre Dame, IN

E-3 9:10 a.m. SYNCHROTRON RADIATION STUDIES OF PASSIVE IRON FILMS - EXAFS
J. J. Rusek*, J. I. Eldridge, J. A. Mann, Jr., and R. W. Hoffman
Case Western Reserve University
Cleveland, OH

E-4 9:30 a.m. NEUTRON DEPTH PROFILING FOR MATERIALS CHARACTERIZATION
R. F. Fleming*, R. G. Downing, and D. H. Vincent
National Bureau of Standards
Washington, DC

E-5 9:50 a.m. X-RAY DIFFRACTION TECHNIQUE FOR MEASURING STRESSES IN THIN OXIDE LAYERS ON METALS
N. Jayaraman
University of Cincinnati
Cincinnati, OH

10:10 a.m. COFFEE BREAK

E-6 10:30 a.m. QUANTIZATION OF THE SURFACE COVERAGE OF Ba AND O ON W SUBSTRATES USING AUGER ELECTRON SPECTROSCOPY
K. Eyink*, B. C. Lamartine, W. V. Lampert, and T. W. Haas
Air Force Wright Aeronautical Laboratories
Wright-Patterson Air Force Base, OH

E-7	10:50 a.m.	MEASUREMENT OF AUGER PEAKS BY FACTOR ANALYSIS H. S. Wildman IBM Corporation Hopewell Junction, NY
E-8	11:10 a.m.	RESOLUTION OF AUGER AND X-RAY PHOTOELECTRON SPECTRA OF MIXTURES BY SPECTRAL RATIOING S. W. Gaarenstroom* and R. A. Waldo General Motors Research Laboratories Warren, MI
E-9	11:30 a.m.	DETERMINATION OF OXIDE FILM THICKNESS AND COMPOSITION OF INDIUM AND CHROMIUM BY DECOMPOSITION OF AUGER ELECTRON SPECTRA D. J. Siconolfi* and R. P. Frankenthal Bell Laboratories Murray Hill, NJ
E-10	11:50 a.m.	APPLICATIONS OF DECONVOLUTION TO X-RAY PHOTOELECTRON SPECTROSCOPY M. F. Koenig* and J. T. Grant University of Dayton Research Institute Dayton, OH
E-11	12:10 p.m.	APPLICATION OF BACKGROUND SUBTRACTION TECHNIQUES TO AUGER SPECTRA FROM A COMMERCIAL CMA R. E. Chase Ford Motor Company Dearborn, MI
	12:30 p.m.	SYMPOSIUM CLOSE

1:30 p.m.- 2:30 p.m.	ASTM Subcommittee Meeting on Auger Electron Spectroscopy. The Subcommittee will discuss recent ballots of documents.

CHEMICAL BONDING AND MATERIAL REACTIONS AT TRANSITION METAL-SILICON INTERFACES

Paul S. Ho
IBM Thomas J. Watson Research Center
Yorktown heights, New York 10598

The transition metal-silicon interface is highly reactive, some forming silicide compounds spontaneously upon metal deposition at room temperature on clean silicon surfaces. The material reaction alters the stoichiometry, atomic structure and chemical bonding at the interface. To understand the electrical characteristics, e.g. the formation of Schottky barriers, of this type of metal-semiconductor interfaces, it is essential to study the reaction and chemistry at the interface. In the past 3-4 years, these interface characteristics have been investigated using surface electron spectroscopies, high- resolution transmission electron microscopy and ion-channeling techniques. This paper reviews the present knowledge of these interface properties, particularly regarding the extent of atomistic details that can be extracted from existing results. First it discusses the chemical bonding in bulk silicides and the trend of its variation with the number of d electrons in the metal. This is followed by examining the electronic structure observed during in-situ formation of the interface which is compared to the corresponding bulk characteristics. The existence of true interface states is indicated and interpreted to reflect specific metal-silicon bonds at the interface. The role of material reaction in establishing intrinsic and extrinsic interfaces is described and its implications on interfacial properties are discussed.

Wednesday Morning - 9:20 a.m.

A-1

Early Stages of Silicide Formation on W, Ni and Pt Surfaces, the Atomic Structures and Compositions

T. T. Tsong, S. C. Wang, F. H. Liu, H. Cheng and M. Ahmad
Physics Department, The Pennsylvania State University
University Park, Pennsylvania 16802

The atomic structures and composition of silicide layers grown on W, Ni and Pt surfaces have been studied in the atom-probe and the field ion microscope. Four stages of silicide growth on W surfaces have been identified. On the W (112) plane, the atomic image structures of the top surface layer in the first stage, and the first three atomic layers in the second stage of silicide formation do not correlate well with the atomic image structures of WSi_2. The W (110) does not react with Si atoms during the first two stages of silicide formation. In the third stage, the growth of thin silicide films proceeds nearly epitaxially from the W (001) planes to cover the entire W substrate, forming miss matches of silicide lattices along the [110] zone line of the W substrate. In the final stage, thick layers of polycrystalline silicide are formed. Their orientations are no longer closely correlated to the W substrate. Atom-probe analyses show the stoichiometry of these atomically perfect crystallines to be exactly WSi_2.

Identifications of crystal planes of WSi_2 can be best done by comparing the image structures with the atomic structures of W atoms in the silicide surfaces since majority of Si atoms are not imaged in the field ion microscope. We have been able to analyze and identify some of the fundamental and superlattice planes of tungsten silicide. In the field ion microscope, a pure Si atomic layer on top of the WSi_2 (112) plane gives a faint image of only about 1 % of the regular image intensity of a W layer. Field ion microscope images show the phase boundary between W and WSi_2 to be very sharp. We also find thick tungsten silicide layers to be very resistive to oxidation.

Thick layers of silicides grown on Pt and Ni surfaces have also been analyzed in the atom-probe. The Pt silicide layers near the Pt interface have the stoichiometry of Pt_2Si. Beyond the relatively sharp interface, a few Si atoms can still be found tens of atomic layers inside the platinum matrix. Near the interface, platinum silicide seems to grow nearly expitaxially from the (111) planes. The composition of nickel silicide varies from one section to another. No sharp boundaries seem to exist between different silicide phases.

To ascertain the conclusions drawn from our atom-probe analyses, we have also made a computer simulation of composition analysis by the atom-probe. These and other new developments in the atom-probe thin film analysis will be presented and discussed.

Wednesday Morning - 10:50 a.m.

A-2

"Characterization by Auger Electron Spectroscopy of Ion Implants in Silicon"

Paul A. Lindfors
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6509 Flying Cloud Drive
Eden Prairie, MN 55344
612/828-6411

C. Fred Hiatt and B. Thompson McClure
Honeywell - Corporate Technology Center
10707 Lyndale Ave. So.
Bloomington, MN 55420

Ion implants of As, P and B in Si wafers were characterized via Auger electron spectroscopy (AES). Shallow, high density implants are becoming of more interest as geometries are reduced in integrated circuits and device speeds are increased. Implant energies used were in the range of 35-100 keV and dosages $\sim 1 \times 10^{16}$ ions/ cm^2.

AES depth profiles were obtained using both the conventional method of sweeping through an energy region for an element and a method that just detected counts at an energy defining a peak and at either one or two energies defining the background. The latter method resulted in considerable savings of time.

The implant profiles for P and As were found to reflect expected distributions. The implant profiles for B, implanted using BF_2, showed effects of dissociation of the BF_2 and multiple peak distributions.

Other aspects of implant distributions were also investigated.

Wednesday Morning - 11:10 a.m.

A-3

Application of PIXE in Specific Sublattice Site Determination of Implants in the Near Surface Region of GaAs

R.S. Bhattacharya and P.P. Pronko
Universal Energy Systems, Inc.
4401 Dayton-Xenia Road
Dayton, Ohio 45432

Ion implantation is a convenient technique used in making active layers in GaAs and has found applications in integrated circuits and microwave device technology. High electrical activation efficiency for high doping levels has not been achieved in GaAs. This limits its application to only certain devices. The problem may lie, as one among others, in the proper substitutionality of the implanted atoms after annealing the implanted GaAs at high temperature. In the last symposium,[1] we have shown that Rutherford backscattering (RBS) and Proton induced X-ray excitation (PIXE) in combination with the channeling technique can be used to measure the substitutionality of S and Si in GaAs. However, in a binary compound semiconductor it is not enough to merely have information about the substitutionality. It is necessary to have more complete knowledge about the specific site occupancy of a particular dopant. For instance, S would have to occupy As lattice sites in GaAs in order to become n type active whereas Si should occupy Ga sites for similar electrical properties. In this paper, we shall demonstrate the use of PIXE in combination with channeling for the determination of specific lattice site occupation of S and Si implanted in GaAs. The measurement makes use of the fact that the <110> axis in a zinc blende structure is made up of atomic strings containing only one of the two types of matrix atoms, for example Ga or As. When an ion beam is incident parallel to the {110} plane but at an angle $+\theta$ with respect to the <110> axis, it has a higher probability of initially striking one of the specific atomic types such as a Ga row rather than an As row. The opposite will happen if the beam is incident at an angle $-\theta$ to the <110> axis. Thus if a complete angular scan is taken, an asymmetric channeling dip will result. When a substitutional impurity shows an asymmetry under these circumstances it can be concluded that the impurity predominantly occupies one sublattice site. From a comparison of the associated asymmetry of the host atoms, the specific site can be identified.

Reference

1. P.P. Pronko, A.K. Rai and R.S. Bhattacharya, 4th Symposium on Applied Surface Analysis, Dayton, 1982.

Wednesday Morning - 11:30 a.m.

Microbeam Analysis Procedures using Auger Electron Spectroscopy

by

J. H. Thomas III
RCA Laboratories
Princeton NJ 08540

Auger microbeam analysis employing elemental mapping has been shown to be extremely useful in solving a large number of problems in diverse technologies. Elemental mapping with a submicron electron beam can be time consuming due to the large time required to acquire statistically significant data. For example, at 10 kV, a 1000Å diameter beam is produced by a beam current of 1 to 10 nA. Although the total secondary current from the sample is large, the Auger signal component is small. Times required to produce a single element Auger map of a matrix element can exceed an hour. When the map is complete, topographic features on the sample may tend to obscure the desired data. The most reasonable way to resolve these situations is to use complimentary imaging modes.

Most microbeam Auger instruments are equipped with secondary electron detection systems. This provides a means of rapidly investigating the sample topography and locating points of interest. Other methods such as energy dispersive x-ray detection permits rapid elemental evaluation and has been shown to strongly supplement surface analysis of difficult surfaces.

The largest component of the signal current leaving an electron bombarded sample surface is the backscattered electron current. The amplitude of the backscattered electron current is sensitive to the average atomic number of the scattering centers and is routinely used in commercial scanning electron microscopes to produce rapid qualitative elemental maps. This method can be surface sensitive. In this presentation, the use of backscattered electron imaging as a complimentary technique with small beam Auger analysis will be discussed and a few interesting applications to metallurgical grade silicon will be presented.

Wednesday Afternoon - 1:30 p.m.

B-1

STRUCTURE OF CARBON FILMS FORMED BY THE PLASMA DECOMPOSITION OF HYDROCARBONS

M. P. Nadler, T. M. Donovan, and A. K. Green

Michelson Laboratory, Naval Weapons Center, China Lake, California 93555 U.S.A.

Much attention has been given to carbon films formed by ion beam processes. The films have a hardness approaching that of diamond and are transparent, but the structure is mostly amorphous.[1] This raises the interesting question of the relationship of the film structure or carbon bonding configuration to the unique optical and mechanical properties.

Infrared spectroscopy has shown the films contain hydrogen (H) and that the H is bonded to carbon atoms which are tetrahedrally coordinated (SP^3). However, depending on starting materials and/or deposition conditions, SP^2 and SP^1 coordination have also been observed.[2]

To explore the role of H in determining optical and mechanical properties, we have heated the films in vacuum and observed, by IR spectroscopy, changes in C-H bonding and H content. On heating in the 500°C range, the SP^3 carbons lose H, forming SP^2 bonds. Remaining H is lost and the films lose transparency upon further heating to 700°C or more, suggesting a critical nature of H in the films. However, since only 1-30% of carbon is originally bonded to H, further experiments such as NMR spectroscopy on C^{13} are planned to clarify the carbon coordination.

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1. H. Vora and T. J. Moravec, "Structural Investigation of Thin Films of Diamond-Like Carbon," J. Appl. Phys. 52, 6151 (Oct. 1981).
2. B. Dischler, A. Bubenzer, and P. Koidl, "Hard Carbon Coatings with Low Optical Absorption," Appl. Phys. Lett. (Apr. 1983).

Wednesday Afternoon - 2:20 p.m.

B-2

A Novel CMA Alignment Technique for Auger Spectroscopy

by

Joseph D. Geller
JEOL (USA), Inc.
11 Dearborn Road
Peabody, Ma. 01960

Quantitative analysis using Auger electron spectroscopy has not yet achieved the degree of accuracy and precision that other spectroscopies (i.e. electron microprobe) have developed. One reason is the basic data collection procedures used in terms of sample positioning, and the other the lack of quantitative correction procedures relating the non-linearities observed between the signal intensity and the concentration.

This talk will deal with a new technique used to calibrate the intensity and energy axis of the (CMA) cylindrical mirror analyzer thereby improving accuracy and precision. The alignment is based upon reproducing the standard and unknown sample position mechanically using an externally adjustable tilt axis stage. Energy calibration is accomplished by referencing a known Auger transition. The current practice is to maximize the intensity and energy on a elastically scattered peak of a known energy. As one changes sample position from the standard to unknown it is imperative the electron beam strike the sample at the focal point of the CMA. The reference peak energy must remain stable within 1 eV at a voltage of 2,000 eV to accomplish this.

Wednesday Afternoon - 3:40 p.m.

Temperature Dependence of the Surface Properties of Impregnated Dispenser Cathode

B. C. Lamartine, W. V. Lampert, and T. W. Haas
AFWAL/MLBM
WPAFB, OH 45433

Measurements have been made of the temperature dependence of various properties of impregnated tungsten dispenser cathodes after in-situ activation. Work function changes were measured over the range of 800 to 1200°C using a novel method which does not depend on thermionic emission models. Surface composition changes were determined using Auger electron spectroscopy and seondary ion mass spectroscopy. The changes observed are interpreted in terms recent measurements of the dependence of work function on surface coverage of oxygen and barium on polycrystalline tungsten substrates[1]. Increases or decreases of work functions and surface chemistry can be interpreted successfully in these terms. Implications for long-lived cathode behavior in travelling waves tubes will also be discussed.

1. B. C. Lamartine, J. v. Czarnecki, and T. W. Haas, to be published.

Wednesday Afternoon - 4:00 p.m.

B-4

Auger Surface Composition and Adhesion Characterization of Galvanized Steel

Bhanuprasad M. Joshi and Rodney F. Kiesel

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Scientific Research Laboratory
Dearborn, Michigan 48121

ABSTRACT

Variable adhesive strengths were observed upon bonding different galvanized steel with the same adhesive. To find the cause of this variation, the surface composition of the galvanized steel strips was determined using Auger Electron Spectroscopy.

We observed a direct relationship between adhesive strength and surface zinc composition for three different adhesives. Most test strips with lower zinc concentrations showed lower adhesive strengths. However when they were washed in a sodium phosphate-based cleaning solvent, alkaline elements, such as calcium, magnesium and aluminum were removed from the surface and the adhesive strength was significantly improved.

Different surface compositions and different adhesive strengths were found to be associated with galvanizing bath formulations as well as galvanizing application procedures.

Although the galvanizing coatings contain over 99.5% zinc, the surface consisted of 35-80% zinc as zinc oxide. Magnesium and/or aluminum, which constitute less than 0.5% of the bulk coating, were observed on the surface at up to 60% concentration as oxides.

Wednesday Afternoon - 4:20 p.m.

B-5

SURFACE ANALYSIS, ION IMPLANTATION AND TRIBOLOGICAL PROCESSES AFFECTING STEELS

Irwin L. Singer
Surface Chemistry Branch, 6170
Naval Research Laboratory
Washington, D.C. 20375

Ion implantation can modify the chemistry and microstructure of surfaces, so it is not surprising that friction and wear behavior can also be affected. In order to discover the mechanisms by which ion implantation produces superior tribological surfaces, it is necessary to examine the mechanical behavior, chemistry and microstructure of layers thinner than 200 nm. This talk will deal specifically with analytical and mechanical techniques used to probe implanted surfaces and to discover effects of ion implantation on the tribological (e.g. friction and wear) behavior of steels.

The ions responsible for improved tribological performance in steels can be divided into two classes: light interstitial ions, such as N^+ and C^+; and carbide-forming transition metal ions, such as Ti^+, Ta^+... For both classes implantation must be carried out at relatively high fluences, typically 2×10^{17} ions/cm^2 or greater to produce peak concentrations of 10-20 at.%. Auger electron spectroscopy is especially valuable for analyzing such implants. At these concentrations, it is sensitive to most all elements regardless of atomic weight, it provides chemical bond (compound) information and when used with ion milling, it provides composition vs. depth profiles with good depth resolution. Optical interferometry of ion milled craters can give accurate depth scales for implant profiles, while metallographical analysis of the same (ion etched) center can reveal microstructural changes in ion implanted surfaces. Other surface spectroscopies such as secondary ion mass spectroscopy (SIMS), Nuclear Backscattering Spectrometry (NBS), energy dispersive X-ray analysis (EDX) and microscopies such as scanning electron (SEM), transmission electron (TEM) and Optical have also been used to analyze implanted steel surfaces.

Two very simple mechanical devices have been exploited to investigate the response of ion implanted surfaces to the two most common wear modes: adhesive and abrasive wear. Adhesive wear of metals during sliding contact is studied by rubbing ball sliders against implanted surfaces at low speeds and repetitively over the same track. Wear scars on slide tracks and sliders are examined optically, by SEM and EDX, then by scanning auger microscopy (SAM). Abrasive wear, or perhaps more relevant, the response of implanted surfaces to abrasive particles, is studied by a polishing implanted and reference disks then carefully measuring relative weight losses. The technique is capable of measuring wear vs. depth of implanted layers with a depth resolution of 20 nm. The abrasion resistance of their layers has been correlated with hardness, and ''hardness'' vs. depth profiles for implanted steel surfaces have been obtained. These polished surfaces are particularly well suited for both chemical (Auger, NBS) and microstructural (TEM) analyses before, during and after wear.

Tribology studies of two steels, N-implanted stainless steel and Ti-implanted 52100 bearing steel, will be presented, and analytical investigations which have led to models for their improved wear behavior will be discussed. A process by which implanted metal ions can react with residual gases in the vacuum chamber to ''carburize'' steel and produce superior tribological surfaces will also be discussed.

Thursday Morning - 8:30 a.m.

Characterization of Titanium, Molybdenum and Yttrium Ion Implanted Metal Surfaces

by

Sin-Shong Lin

Army Materials and Mechanics Research Center
Watertown, Massachusetts 02172

ABSTRACT

The surfaces of the titanium and molybdenum ion implanted steels as well as the yttrium ion implanted nickel base alloy have been characterized by Auger electron spectroscopic(AES) analyses. The AES technique was used together with argon ion milling to study the compositions of the surface and the surface sublayers. The topological features of these surfaces were also examined by the scanning electron microscope(SEM).

The titanium ion implanted AISI M50 steel was obtained at 185 keV and 4.6×10^{17} ions/cm^2 *for wear resistance.* It was found that during the ion implantation, not only titanium but also carbon and oxygen were implanted into the surface. The depth profiles of these elements in subsurface layers were studied and analyzed. The formation of hard TiC roughly equal to the concentration of the host lattice in the amorphous layers is believed to be a major cause for the enhanced wear resistance.

The Molybdenum ion implanted AISI 52100 steel at 100 keV and 3.5×10^{16} ions/cm^2 was examined, and it was found that the dosage and the energy of the implantation were not sufficient to cause substantial changes to the uppermost surface structure. The implanted Mo was found to aggregate into clusters. Other features associated with the molybdenum implantation are discussed.

The yttrium ion was implanted on unpolished surfaces of the nickel base alloy at 150 keV and 1 or 5×10^{16} ions/cm^2 for high temperature oxidation resistance. Since the yttrium concentration of the implanted surface was too small for the present AES analysis, the depth profile of yttrium was obtained by the secondary ion mass spectrometric(SIMS) technique. The yttrium profile showed that the implanted atom had a rather high depth penetration into the subsurfaces and that there was no significant alteration of the surface composition due to the small dosage of the implantation.

NOTE: The ion implantation of these materials was performed at Naval Research Laboratory, Washington, D.C. Some of the implanted samples were provided by Dr. G. K. Hubler, NRL.

Thursday Morning - 9:20 a.m.

C-2

Surface Analysis Studies on the Corrosion Resistance of Mo Implanted Al Alloys

by

M. V. Zeller and J. A. Kargol
College of Engineering
University of Notre Dame
Notre Dame, IN 46556

The effect on corrosion resistance by Mo additions into Al alloys is examined as a function of corrosion treatment. Two different doses of Mo are incorporated into alloy surfaces by ion implantation. The unimplanted and implanted alloys are subjected to general and localized corrosion testing using sulfate and sulfate plus chloride solutions. Electrochemical properties obtained from potentiostatic polarization curves are correlated to the Mo content and the surface chemistry as measured by Auger Electron Spectroscopy. The results show that Mo implantation forms a complex oxide within the top 1000A° of the Al surface and inhibits pitting attack of these alloys.

Thursday Morning - 9:40 a.m.

C-3

AES AND RBS ANALYSIS OF LASER-MIXED CR AND NI MULTILAYER FILMS ON CU ALLOYS

I. Sawchyn and C. W. Draper
Western Electric, Engineering Research Center
P.O. Box 900, Princeton, N.J. 08540

A new and growing area of research is the use of directed energy, such as laser, electron and ion beams, to produce surface alloys with chemical compositions that are vastly different from their bulk substrates. The strategic nature or cost of the alloying species makes surface rather than bulk alloying very attractive. In addition, directed energy methods can be used to produce surface alloys that have no bulk counterparts by circumventing equilibrium phase diagram restrictions.

One of the challenges in this new area of surface metallurgy is the characterization of the affected regions. Chemical information is required not only from the atomic surface but also as a function of depth. This paper will describe the analysis of Cr and Ni multilayer films that have been laser-mixed with Cu substrates and analyzed with Auger electron (AES) and Rutherford backscattering spectroscopies (RBS). For multilayer, transition metal-on-metal systems, such as Cu/Cr/Ni, the complementary use of both AES and RBS results in a more detailed understanding of the laser-mixed compositional profiles than would result from the application of either technique alone. The relative strengths and weaknesses of both analytical methods are pointed out.

Thursday Morning - 10:40 a.m.

C-4

Proposed Paper for Presentation at the
Fifth Symposium on Applied Surface Analysis
to be held at the University of Dayton,
Dayton, Ohio, June 8-10, 1983

The Adsorption and Thermal Decomposition of Tricresyl-Phosphate (TCP) on Iron and Gold

by Donald R. Wheeler
NASA Lewis Research Center
Cleveland, Ohio 44135

and

Owen D. Faut
Wilkes College
Wilkes-Barre, Pennsylvania

Adsorption and Thermal Decomposition of Tricresyl-Phosphate (TCP) on Iron and Gold by Donald R. Wheeler, NASA Lewis Research Center, Tribology Branch, Cleveland, Ohio, and Owen D. Faut, Wilkes College, Department of Chemistry, Wilkes-Barre, Pennsylvania.

Because tricresyl-phosphate (TCP) is a common anti-wear additive in lubricants, there is great interest in its interactions with metal substrates. In this work, TCP was allowed to adsorb on polycrystalline iron and gold at room temperature. X-ray photoelctron spectroscopy (XPS) was used to analyze the adsorbed species. The substrate was then heated in steps to 300C, and the changes in the adsorbate were analyzed after each step.

On both substrates saturation adsorption occured at about one monolayer, but the sticking coefficient was less on gold than on iron. Comparison of the XPS spectra of TCP on each substrate with the spectrum from condensed TCP indicated non-dissociative adsorption on gold, possibly by a dipole-induced dipole interaction. On iron, there was apparently additional interaction between the substrate and the tolyl groups on the TCP molecule.

Upon heating, TCP desorbed molecularly from gold beginning just above room temperature. The desorption was complete by 200C. However, when the iron substrate was heated TCP did not desorb, but decomposed between 150C and 250C. A phosphate formed during the decomposition and one of the tolyl groups was lost. The temperature range in which this decomposition occured corresponds to previously observed transitions in the lubricating properties of TCP.

Thursday Morning - 11:00 a.m.

THE EFFECT OF WEAR ON SURFACE TEXTURE

By

Richard B. Zipin, Senior Staff Engineer
The Bendix Corporation, Automation & Measurement Division
Dayton, Ohio 45401

Wear is an important phenomena to consider in the design and application of engineering surfaces. Surface metrologists generally study the effects of wear by means of the average height sensitive roughness parameters (1) which consider surface profile data as a whole. These parameters do not take account of the separate contributions to the overall surface texture of the remnant of the original unworn surface which remains after wear occurs and the new surface component created by the wear process.

For the limited depths of wear which are allowed in most engineering applications before one decides that "failure" has occurred, the "worn" surface retains some of the features of the original surface in addition to having new features which were generated by the wear process. The former were found in the bottom of the original surface profile and remain at the bottom of the new surface profile, while the latter are confined to the top of the new profile. The surface resulting from a wear process thus exhibits a two-stratum transitional topography of type RS (2), and the surface texture contributions of the individual strata are readily analyzed by the use of probit scaled plots of the cumulative height distribution of the surface profile.

These ideas are illustrated by analysis of a simple deterministic profile model and a probabilistic model. They have been verified by the results of a series of controlled wear experiments.

1) R. B. Zipin, The Height Sensitive Surface Roughness Parameters, Appl. Surface Sci. 9, 266 (1981).

2) R. B. Zipin, The Analysis of Profile Strata for Surface Texture Specification, Appl. Surface Sci., in press.

Thursday Morning - 11:20 a.m.

FRACTAL,SELF-SIMILAR SURFACES

P. Pfeifer,(a) D. Farin(b) and D. Avnir(b), Fakultät für Chemie, Universität Bielefeld, D-4800 Bielefeld,FRG(a); and Department of Organic Chemistry, The Hebrew University of Jerusalem, Jerusalem 91904, Israel(b)

We have extended and adapted [1-3] Mandelbrot's fractal theory [4] to problems of surface geometrical irregularity (roughness). In doing so we have found a wealth of correlations, in many cases for the first time, between various parameters of surface science, e.g., $A \propto \sigma^{(2-D/2)}$; $n \propto R_g{}^{-D}$; $V_m \propto \sigma^{-D/2}$; $n \propto d^{D-3}$; $M \propto l^D$, where D: fractal dimension of the surface; A: apparent surface area; σ: effective cross-sectional area of the adsorbate; n: number of mole(cule)s at monolayer coverage of e.g., 1 gr absorbent; R_g: radius (of gyration) of adsorbed polymer or microsphere; Vm: monolayer coverage from the BET equation; d: average adsorbent-particle diameter; M: number of adsorption sites; l: distance from a fixed site. All these equations are obeyed only for scaling self-similar surfaces, i.e., those which may be characterized intensively by a fractal (effective, noninteger) dimension, so that $2 \leqslant D < 3$. Remarkably, we find fractal-surfaces to be the rule, and non-fractal surfaces the exception. This discovery offers D as a useful tool in dealing with surface roughness. Following is a partial list of fractal surfaces we found [1-3,5]. Values close to 3.0 indicate extreme wiggliness of the surface, whereas low values of D indicate regularity or smoothness. Alumina: 2.79; silica gel: 2.94; precipitated silica: 2.62; fumed silica: 2.01, quartz: 2.15; ferric oxihydrate: 2.61; crushed lead glass: 2.35; cellulose: 2.25; graphite (Vulcan 3G): 2.07; graphite (Graphon): 2.04; coal dust: 2.52; various charcoals: 2.04, 2.30, 2.54, 2.67; various limestones: 2.16, 2.58, 2.63, 2.91, 2.97.

1. D. Avnir and P. Pfeifer, Nouv. J. Chim., 7, 71 (1983).
2. P. Pfeifer, D. Avnir and P. Pfeifer, Surface Science, 126, (1983).
3. P. Pfeifer and D. Avnir; and, D. Avnir, D. Farin and P. Pfeifer, papers submitted for publication, 1982.
4. B.B. Mandelbrot, "The fractal Geometry of Nature", Freeman, San-Francisco, 1982.
5. D. Avnir, P. Pfeifer and D. Farin, paper in preparation.

Thursday Morning - 11:40 a.m.

Laser Ionization of Sputtered Neutrals: A Novel Approach to Materials Characterization

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We have recently demonstrated that neutral atoms with kinetic energy between 0 and 100 eV can be ionized with nearly 100% efficiency using laser induced multiphoton resonance ionization (MPI) techniques. By applying this method to the detection of sputtered neutrals, it is possible to attain detection efficiencies several order of magnitude greater than other post-ionization methods. In this work, we compare MPRI to low dose secondary ion mass spectrometry (SIMS) as a tool for examining the chemisorption of small molecules on metal surfaces. The high sensitivity of MPRI indicates there should be a number of advantages of this technique over conventional SIMS measurements for surface characterization studies. Other limitations and advantages of this approach will be elucidated, including a discussion of an improved approach for trace analysis of solids with less serious matrix effects than those exhibited by SIMS.

Thursday Afternoon - 1:30 p.m.

D-1

QUANTITATIVE ANALYSIS OF SEGREGATION AT THE SiO_2/Si-INTERFACE BY SIMS AND RBS

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and

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The segregation behaviour at the SiO_2/Si-interface is of major interest in silicon integrated circuit technology particularly for process development and modelling. The redistribution of the dopand species caused by thermal annealing in oxidizing atmosphere cannot be measured by electrical techniques, at least not in oxide and the region of high dopand concentration.

In this paper it will be shown that problems typically associated with SIMS depth profiling in the presence of a changing matrix composition may be overcome by appropriate bombarding and charge neutralization condition. Arsenic depth profiles of arsenic implanted silicon samples subjected to various annealing conditions in oxidizing atmosphere have been measured on the ion microprobe A-DIDA 3000-30. Comparison with Rutherford backscattering (RBS) data obtained from the same samples demonstrate that quantitative depth profiling of As across a SiO_2/Si-interface layer is such obtained.

Thursday Afternoon - 2:20 p.m.

ABSTRACT

Chemistry of Glass-Ceramic to Metal Bonds in Headers for Use in Initiators and Actuators in Detonator Systems

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The fabrication of glass-ceramic headers for use in initiators and actuators is a complex manufacturing process whose success depends on the quality of the glass-ceramic to metal seal. This is true because the seal must have properties (i.e. hermeticity, burst strength, etc.) sufficient to ensure the successful function of the header. Glass-ceramic headers usually consist of: a high tensile strength shell, one or several metal rings and/or pins which act as electrical feedthroughs, and a glass-ceramic functioning as the electrical insulator.

In the most recent headers manufactured at Mound, the glass-ceramic to metal seals have been fabricated using a lithia-alumina-silica glass and either Inconel 718 or Hastelloy C-276 as the metal piece parts. These metals were chosen for their high tensile strength and corrosion resistance.

SEM, EDX, AES, ESCA and in-depth sputter profiling were employed to study the change in the surface chemistry both on the metal piece parts and on the glass-ceramic caused by the different manufacturing processes necessary to prepare a header, and also were employed to examine the interfacial chemistry of the glass-ceramic to metal bond. Through these examinations a better understanding of each process treatment was obtained which lead to improvements, such as in the welding of Inconel 718 as well as improvements in burst strength.

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* Mound is operated by Monsanto Research Corporation for the U. S. Department of Energy under Contract No. DE-AC04-76-DP00053.

Thursday Afternoon - 3:20 p.m.

D-3

AUGER AND DEPTH PROFILE ANALYSIS OF SYNTHETIC CRYSTALS FOR DISPERSION OF SOFT X-RAYS

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Numerous samples have been facricated and analyzed as part of a program to produce soft X-ray dispersion elements for various laboratory applications. The majority of this work has centered around the carbon/tungsten system, although several other low Z/high Z pairs have been investigated. This report describes the development of certain vacuum deposition techniques for fabricating these dispersion elements, based upon results obtained from X-ray reflectivity measurements and Auger depth profile analysis. The composition of the films is chiefly tungsten carbide layered with carbon similar to other recent work (ref. 1). Excess carbon is introduced during the deposition of the tungsten to ensure that the carbide layer is fully stoichiometric. It can be shown that post-deposition annealing further sharpens the C/WC layers by excluding more of the free carbon from the carbide. The typical layer thickness is ~3.0 nm for both the carbide and carbon layers. The reflectivity measurements were made using Al K alpha at grazing incidence. Emphasis will be placed on the application of surface analysis results in suggesting modifications to the fabrication process and in evaluating the results such modifications have on the layer stoichiometry, continuity and periodicity of the dispersion elements so produced.

[1] T. W. Barbee, Jr., "Sputtered Layered Synthetic Microstructure (LSM) Dispersion Elements," Proceeding of the Topical Conference on Low Energy X-Ray Diagnostics (Monterey, Calif., June 81)

Thursday Afternoon - 3:40 p.m.

D-4

MOLECULAR ORIENTATION AND STRUCTURE OF COPPER-BENZIMIDAZOLE FILMS

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ABSTRACT

When a copper surface is dipped into an aqueous solution of benzimidazole, a film approximately 375Å thick is formed. Using X-ray Photoelectron Spectroscopy, the film is shown to be composed of an organo-metallic complex of the benzimidazole and the copper. The film is investigated using Reflection-Absorption Infrared Spectroscopy, and the film is shown to preferentially oriented with the plane of the benzimidazole ring parallel to the metal surface.

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Thursday Afternoon - 4:00 p.m.

D-5

EMISSION FTIR ANALYSES OF THIN MICROSCOPIC PATCHES OF JET FUEL RESIDUES DEPOSITED ON HEATED METAL SURFACES

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ABSTRACT

The objective of the investigation was to relate fuel stability with fuel composition and to develop mechanisms for deposit formation, which can account for the large influence of small concentrations and non-hydrocarbons. Fuel deposits reduce heat transfer efficiency and increase resistance to fuel flow and are therefore highly detrimental to aircraft performance. Infrared emission Fourier transform spectroscopy was chosen as the primary method of analysis because it was sensitive enough to be used in situ on tiny patches (0.01 mm diameter and less) of monolayers or of only a few molecular layers of deposits which generally proved completely insoluble in any non-destructive solvents.

The deposits were produced by NASA on metal strips (shims) in a high pressure/high temperature fuel system simulator operated with aerated fuel at varying flow rates. The shims were mounted on a slightly heated (40°C) holder in such a way that the deposits faced an all-reflecting microscope objective of high numerical aperture and long working distance, which was coupled to an FTIR spectrophotometer. Temperature constancy of the test specimens proved to be extremely important to avoid reabsorption of emitted radiation. For maximum sensitivity polarization modulation was used.

Deposits of three base fuels were compared, dodecane, a highly aromatic jet fuel, and a synthetic jet fuel particularly rich in polynuclear aromatics. Every fuel in turn was provided with and without small additions of such additives as thiophene, furan, pyrrole and copper and iron naphthenates.

While the deposit densities were highest for the dodecane fuel compositions, the band intensities of C-C and C-H bands were highest for the most aromatic fuel compositions. In all cases furan and pyrrole increased deposit band intensities very strongly. Significantly, furan and, less so, thiophene and pyrrole, increased the intensity of the "amorphous" component of the 725/730 cm^{-1} methylene rocking mode of solid paraffinic structures.

Arguments are presented for a mechanism by which the additives are concentrated on the solid surfaces and interact with oxygenated hydrocarbon radicals to form amorphous polymers.

This work was funded by grants from the Air Force Office of Scientific Research, Grant No. AFOSR-81-0005, and from the National Aeronautics and Space Administration, Grant No. NAG-3-205. The United States Government is authorized to reproduce and distribute reprints for Governmental purposes notwithstanding any copyright notation hereon.

Thursday Afternoon - 4:20 p.m.

AN XPS STUDY OF THE ADSORPTION OF OXYGEN AND WATER VAPOR ON CLEAN LITHIUM FILMS*

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The need to develop high energy density power sources has produced a new generation of non-aqueous battery systems in which pure lithium is used as the primary anode material. In an effort to understand the complex chemical reactions which occur in these cells, it is necessary that the nature of the passivating film which forms on lithium before it is incorporated into a cell be examined in detail. In this study, x-ray photoelectron spectroscopy (XPS) was used to characterize low level exposures of clean polycrystalline lithium films to molecular oxygen and water vapor. High purity lithium was vapor deposited on an argon ion sputtered tantalum foil in a deposition chamber which was attached to a highly modified XPS system allowing specimens to be prepared and studied at or below 3×10^{-7} Pa pressure. Quantitative reaction rates for the two gases were determined. A two step reaction for the interaction of water vapor with lithium was observed and is a function of the total exposure. The results of these experiments differ in several aspects from those previously reported in the literature. These differences are discussed.

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*The work was supported by the Air Force Wright Aeronautical Laboratory/Aero Propulsion Laboratory.

Friday Morning - 8:30 a.m.

E-1

Applications of Bremsstrahlung-Induced Auger Lines for Thin Film Analysis

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If an electron that interacts with a target, as in an x-ray source, is of sufficient energy, two kinds of x-rays are produced: characteristic lines and continuum. In x-ray photoelectron spectroscopy (XPS) the characteristic lines are employed to generate photoelectrons from a sample material where the intensity of photoelectrons may be measured as a function of binding energy to gain chemical information on the sample. It has been demonstrated that for some elements the x-ray continuum, termed "bremsstrahlung radiation", is of sufficient energy to produce Auger electrons (1) of higher kinetic energy than obtainable by the characteristic X-ray lines. These Auger electrons appear as discrete lines that are characteristic of the material from which they originated. Sometimes these Auger electrons are more sensitive to oxidation state and chemical environment than are the photoelectrons, so the shift of the characteristic Auger transition peaks with oxidation state might be greater.

Using a normal XPS anode, such as Al, the difference in binding energy of the Si 2p photoelectrons from elemental Si and SiO_2 is about 4 eV. From the bremsstrahlung-induced Si KLL Auger lines the difference in kinetic energy of elemental Si and SiO_2 was reported to be approximately 7 eV. (2). The application of bremstrahlung-induced Auger lines for the analysis of ultra thin oxides on silicon semiconductors will be discussed as well as the more general aspects of the technique.

1. C. D. Wagner, J. Vac. Sci. Technol., 15(1978)518; P. M. Th van Attekum and J. M. Trooster, J. Phys. F, 8(1978)L169; J. E. Castle and R. H. West, J. Electon Spectrosc. Relat. Phenom., 16(1979)195.
2. C. D. Wagner and J. Ashley Taylor, J. Electron Spectrosc. Relat. Phenom., 20(1980)83.

Friday Morning - 8:50 a.m.

SYNCHROTRON RADIATION STUDIES OF PASSIVE IRON FILMS - EXAFS

J. J. Rusek, J. I. Eldridge, J. A. Mann, Jr. and R. W. Hoffman

The structure of electrochemically passivated layers has been an area of much debate in recent years. These surface layers have usually been analyzed in vacuum, hence they are not under potential control at the time of analysis. With the advent of synchrotron light sources, the use of Extended X-ray Absorption Fine Structure (EXAFS) spectroscopy appears promising as an in situ technique to study the local structure of these films. A discussion of the experiments performed at the Stanford Synchrotron Radiation Laboratory are presented. This technique is useful in determining the structure of thin film samples, examples are presented including air oxidized iron films, dried passivated iron films and iron evaporated on gold/plastic composites. Powder oxides as well as mineralogical oxides were analyzed as reference materials. For a given structure, the thickness of the passive layer can be obtained using iron colorimetry of the electrolyte after reduction of the passive film. Due to extreme instabilities in the light source the passive layer was crudely estimated to be comprised of predominantly an n-FeOOH species, most likely δ-FeOOH. Based on this structure the thickness of the passive layer was estimated at ca. 17A.

Friday Morning - 9:10 a.m.

NEUTRON DEPTH PROFILING FOR MATERIALS CHARACTERIZATION

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A Neutron Depth Profiling (NDP) facility has been developed for the non-destructive determination of concentration profiles in the near-surface region. The technique is specific for those target atoms which have large cross-sections for thermal neutron induced, charged particle emitting reactions--for example, ^{3}He, ^{6}Li, ^{10}B, ^{7}Be, and ^{22}Na. A beam of neutrons with intensity of 3×10^{5} n/s and energy ~10^{-2} eV uniformly illuminates the sample, producing initially mono-energetic charged particles such as H^{+} and He^{++}. These isotropically emitted particles lose energy in passing through the sample. The energy which they retain upon leaving the sample surface is a measure of the depth at which the reaction took place. The depth scale is monotonically related to the energy scale by the charged particle stopping power. Once the system has been calibrated with an appropriate elemental standard, the concentration scale is fixed independent of the sample composition. An analysis produces a profile several micrometers deep with a resolution of ~30 nm and a sensitivity of 10^{13} atoms/cm^{2} in favorable cases. A modified version of this technique can be used to profile bismuth atoms, using the alpha particle emitted by the ^{210}Po daughter.

The application of this profiling technique will be described in detail for the following cases:

- The distribution of ion-implanted helium in single-crystal nickel.
- The distribution of boron implanted in silicon and silicon dioxide.
- The boron distribution in thin borophosphosilicate glass films on silicon.
- The distribution of excess lithium in the near surface region of Li_7B_6 foils.
- The boron distribution in sputter-deposited iron-boron metallic glasses.

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Friday Morning - 9:30 a.m.

X-RAY DIFFRACTION TECHNIQUE FOR MEASURING STRESSES IN THIN OXIDE LAYERS ON METALS.

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For the first time, the $\mathrm{Sin}^2\psi$ method (1) has been successfully used for the measurement of stresses in oxide layers formed on metal substrate. This method consists of finding the average strain in the oxide layer for different depth of penetration of x-rays. The average strain is determined by measuring the interplanar spacings of a particular set of planes. The depth of penetration of x-rays is altered by changing the orientation of the surface normal of the sample by an angle ψ with reference to the incident x-ray beam. The stress state in the oxide scale can be computed using the data on strain determined by the above technique. Recent research work by the author on the measurement of oxidation stress in NiO layer formed on annealed and electropolished polycrystalline Ni200 indicates that the stresses on the free oxide surface ($\sigma 11$ and $\sigma 22$) are compressive and the average normal stress through the thickness of the oxide scale ($\sigma 33$) is tensile. Polycrystalline Ni200 samples were oxidized in a dry oxygen atmosphere at 1400, 1600 and 1800 °F for this study. Surface stresses on the oxide formed at 1400 and 1600°F are found to be isotropic and both the surface stresses and the average normal stresses increase with increasing temperature of oxidation. For the oxide scale formed at 1800°F, the surface stresses are smaller than that formed at lower temperature. This relaxation in the surface stress is attributed to the observed deformation of oxide scale formed at this temperature.

The implications of the presence of growth stresses on the oxide scale integrity and on the performance of high temperature materials will be discussed. Advantages and shortcomings of the x-ray diffraction technique will be discussed in relation to possible application to various oxide/metal systems.

(1) Elements of X-ray Diffraction by B.D. Cullity, Addison-Wesley Publications 1978.

Friday Morning - 9:50 a.m.

E-5

Quantization of the Surface Coverage of Ba and O on W Substrates Using Auger Electron Spectroscopy

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Impregnated tungsten dispenser cathodes have been characterized in recent years by a wide variety of surface analytical techniques such as Auger electron spectroscopy, ion scattering spectroscopy, secondary ion mass spectrometry, and synchrotron photoelectron spectroscopy. Each of these techniques has contributed information on details of the surface layers of activated cathodes but on one technique has provided a complete quantitative relationship. It is well established that the surface of these cathodes consists of thin layers of Ba and O on a W substrate. The quantitative aspects of this adsorption system have been determined using Auger electron spectroscopy for varying coverages of Ba and O. Calibration of the Auger spectra for monolayer coverage of Ba was accomplished by these methods: The break point method, the attenuation of a W Auger feature, the relative attenuation of a high and low energy W Auger peak and also the Moore and Allison method.[1]

Agreement is good among the various methods and serves to calibrate quantities observed on actual activated, operating dispenser cathodes.

1. G. E. Moore and H. W. Allison, J. Chem. Physics, 23 (1954) 1609.

Friday Morning - 10:30 a.m.

Measurement of Auger Peaks by Factor Analysis

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The usual computer method of measuring Auger lines for depth profiling is inadequate for several reasons. It is prone to error because it incorporates signal noise in the determination of peak size and ignores background interference from other peaks. Also, it discards the chemical state information in the peak shapes. S.W. Gaarenstroom has demonstrated that factor analysis can be used to retain the chemical state information.[1] This paper describes how factor analysis can be used to improve sensitivity, remove interferences, and aid in measuring the Auger peaks for depth profiling. To illustrate this, analysis of SiO_2 on Al and of Si_3N_4 on Pt are used as examples.

Factor analysis consists of two stages: principal component analysis and factor interpretation.[2] In the first stage, principal component analysis selects, from the spectral data itself, the minimum number of abstract curves, i.e., profiles and spectra, that can best model the data. An important side benefit of this process is that the signal is separated from most of the noise in a frequency independent way. The abstract profiles are linear combinations of real profiles, and the abstract spectra are linear combinations of real spectra. A corollary of this is that a minimum number of abstract spectra contain all the spectral components in the data. An analyst can inspect a minimum number of spectra for the signature of different chemical compounds. Another corollary is that, if there is only one component or peak shape in an energy window, then the abstract spectrum is the real peak shape and the abstract profile is the real profile.

In the second stage, there are several choices. One can construct the elemental profile using any of the schemes for measuring the Auger peak size such as: from peak to peak, from negative peak to background, or from the area under the N(E) curve. Or, one can try to resolve the chemical state profiles and spectral components using least square curve fitting or maximizing techniques to find the proper linear combination of the corresponding abstract curves.

In one particular application, factor analysis of data from one sample (the reference) can be used to obtain a template for measuring a low concentration constituent in data from another sample that has the same components as the first. This template is a set of numbers that produces the size of a constituent peak when it is convolved with a spectrum.

References

1. S.W. Gaarenstroom, J. Vac. Sci. Technol., 20,458 (1982).

2. E.R. Malinowski and D.G. Howery, Factor Analysis in Chemistry (Wiley - Interscience, New York, 1980).

Friday Morning - 10:50 a.m.

E-7

RESOLUTION OF AUGER AND X-RAY PHOTOELECTRON SPECTRA OF MIXTURES BY SPECTRAL RATIOING

By

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ABSTRACT

A method is described for resolving spectra of pure components from Auger or X-ray photoelectron spectra of mixtures. The method begins by computing spectral ratios from two multicomponent spectra which contain the same unidentified components, but in different proportions. One requirement of the method is that a spectral region must exist for each component where only that component contributes to the signal. A second requirement is that the inelastic energy loss background must be suppressed. An appropriate background suppression method is presented for ESCA (Electron Spectroscopy for Chemical Analysis) survey spectra. Spectral ratioing is applied to Auger and ESCA spectra to identify surface segregates on heat-treated alloy steels.

Friday Morning - 11:10 a.m.

E-8

DETERMINATION OF OXIDE FILM THICKNESS AND COMPOSITION OF INDIUM AND CHROMIUM BY DECOMPOSITION OF AUGER ELECTRON SPECTRA

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ABSTRACT

The thickness and composition of thin oxide films on indium and on chromium have been obtained from their Auger electron spectra by a technique developed to decompose overlapping spectral peaks. In the first case, the spectral peak from an indium metal substrate is separated from the indium peak from an In_2O_2 surface film, so that the thickness of the oxide film can be ascertained. A similar separation in the second system shows that a gross error in the Cr content of the oxide is obtained if the spectrum is not decomposed before the Cr content is calculated. The only assumption in the decomposition technique is that the experimental spectrum can be described by a linear combination of the standard spectra of its components.

Friday Morning - 11:30 a.m.

E-9

Applications of Deconvolution to X-ray Photoelectron Spectroscopy*

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Deconvolution is a mathematical method of enhancing the resolution of spectra by reducing instrumental broadening effects. In x-ray photoelectron spectroscopy (XPS), deconvolution can be used to remove the satellite lines and broadening due to the x-rays used to obtain spectra. We have compared the results of deconvolution of data obtained with conventional Mg and Al K_α x-ray sources with spectra obtained using a monochromatic Al x-ray source from the same sample in the same instrument, a Kratos ES300 x-ray photoelectron spectrometer.

Deconvolution can also be used to remove broadening effects due to the finite resolution of the analyzer. This has been examined in the Kratos ES300 which utilizes a hemispherical analyzer with variable-width entrance and exit apertures. It has been verified that a practical limit of 30% reduction in the full width at half maximum of each peak can be reliably removed from the data.

The removal of background due to inelastic losses by deconvoluting with an appropriate function has also been examined. Successful background removal has been obtained over energy ranges as wide as 100 eV and encompassing several peaks.

Examples on the application of these aspects of deconvolution to XPS data will be presented. These examples will include spectra obtained from dispenser cathode surfaces and metal/metal oxide mixtures.

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* This work was sponsored by the Materials Laboratory, Air Force Wright Aeronautical Laboratories, Wright-Patterson Air Force Base, Ohio 45433

Friday Morning - 11:50 a.m.

E-10

APPLICATION OF BACKGROUND SUBTRACTION TECHNIQUES TO AUGER SPECTRA FROM A CONVENTIONAL CMA.

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The use of background subtraction techniques in Auger electron spectroscopy is finding increasing application. However, many conventional cylindrical mirror analyzers (CMA) provide data in only the so-called derivative mode. Because it is usually not possible to measure the spectrum in the vicinity of zero kinetic energy, straight-forward integration techniques cannot be applied to obtain N(E) from the derivative spectrum. A computer-based technique for circumventing this difficulty is described and its limitations are discussed.

Friday Morning - 12:10 p.m.

E-11

SECTION V

CONCLUSIONS

The purpose for holding this Symposium was to bring together basic research, applied research, and problem solving efforts all involving some type of surface analysis of Department of Defense interest into one meeting to try to improve communications and understanding of persons in these various endeavors. That a need for such a meeting exists seems, more and more, to be established from the many favorable comments received and from the continuing interest in this Symposium.

The Office of Naval Research is to be commended for supporting this Symposium.

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