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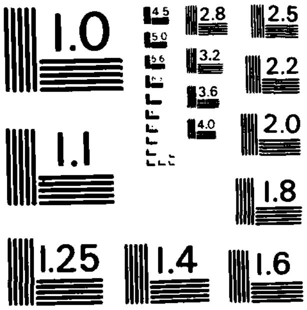

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4th SYMPOSIUM ON APPLIED SURFACE ANALYSIS

by

John T. Grant

Final Technical Report

For the Period 1 February 1982 - 1 February 1983

March 1983

AFOSR
~~AFOSR Grant No.~~ 82-0151

UNIVERSITY OF DAYTON
RESEARCH INSTITUTE
Dayton, Ohio 45469

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John T. Grant

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300 College Park
Dayton, OH 45469

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Coatings
Corrosion

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

The 4th Symposium on Applied Surface Analysis was held at the University of Dayton, 9-11 June 1982. This Symposium was held to meet a need, namely to show the transition between basic surface science research and applications of this research in areas of Air Force interest. Areas receiving special attention at this Symposium were ion implantation, corrosion, catalysis, thin films, and electronic materials. Other topics discussed included adsorption, oxidation, coatings, cathodes, and developments in instrumentation. Approximately 125 scientists active in the field of surface analysis participated in the Symposium. Three

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20. ABSTRACT (concluded)

scientists presented invited papers at the Symposium. There were 39 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.

NOTICE

Parts I & II
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Information Division

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FOREWORD

This report describes the 4th Symposium on Applied Surface Analysis held at the University of Dayton, 300 College Park Avenue, Dayton, Ohio 45469, on 9-11 June 1982, under AFOSR Grant No. 82-0151.

The Symposium was conducted by the University of Dayton with Dr. John T. Grant of that organization and Dr. T. W. Haas of the Materials Laboratory, Air Force Wright Aeronautical Laboratories as Co-Chairmen. This report was submitted in March 1983.

SECTION 1
INTRODUCTION

The 4th Symposium on Applied Surface Analysis was held at the University of Dayton, 9-11 June 1982. 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 Air Force interest. Areas receiving special attention at this Symposium were ion implantation, corrosion, catalysis, thin films, and electronic materials. Other topics discussed included adsorption, oxidation, coatings, cathodes, and developments in instrumentation. Approximately 125 scientists active in the field of surface analysis participated in the Symposium. Three scientists presented invited papers at the Symposium. There were 39 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 2
SYMPOSIUM GOALS

The 4th Symposium on Applied Surface Analysis was held at the University of Dayton, 9-11 June 1982. 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 Air Force 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. T. W. Haas of the Materials Laboratory, Air Force Wright Aeronautical Laboratories as Co-Chairmen. Three invited speakers were selected to cover specific areas of interest. These speakers and their topics were:

1. T. L. Barr
   Universal Oil Products Inc.
   "Industrial Applications of ESCA: A Study of Si As It Occurs in Absorbents, Catalysts and Thin Films"

2. G. K. Hubler
   Naval Research Laboratory
   "Ion Implantation in Corrosion Research and Corrosion Protection"

3. J. W. Mayer
   Cornell University
   "Applications of Rutherford Backscattering"

Contributed papers on applied surface analysis were also solicited from the research community, resulting in 39 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. Twenty five 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 early in the Spring of 1983.

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 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 vendors' 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 3

PARTICIPANTS

Approximately 125 scientists active in the field of surface analysis attended the Symposium.

The list of participants follows.

LIST OF PREREGISTERED ATTENDEES

4th SYMPOSIUM ON APPLIED SURFACE ANALYSIS

9 - 11 June 1982
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SECTION 4
PROGRAM

The technical program was divided into five sessions. Three 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.

SECTION 5
SYMPOSIUM CONCLUSIONS

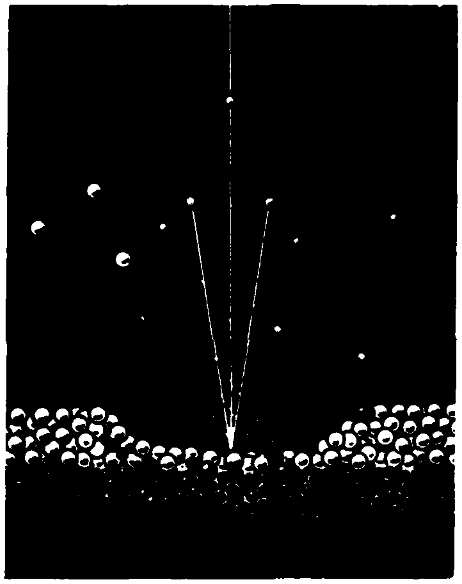
The purpose for holding this Symposium was to bring together basic research, applied research and problem solving efforts all involving some type of surface analysis of Air Force interest into one meeting to try to improve communications and understanding of persons in these various endeavors. A need for such a meeting exists based on the many favorable comments received and from the growing interest in this Symposium.

The Air Force Office of Scientific Research is to be commended for supporting this Symposium.

AFOSR-TR- 83-0216 - Part 2

4th SYMPOSIUM ON

APPLIED SURFACE ANALYSIS



FINAL PROGRAM

Supported by
AIR FORCE OFFICE OF SCIENTIFIC RESEARCH

9-11 JUNE 1982

UNIVERSITY OF DAYTON
Dayton, Ohio

83 04 14 101

4TH SYMPOSIUM ON APPLIED SURFACE ANALYSIS

9 - 11 JUNE 1982
UNIVERSITY OF DAYTON

GENERAL INFORMATION

The 4th Symposium on Applied Surface Analysis is being supported by the Air Force Office of Scientific 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

The proceedings will be published. The 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, 9 June through lunch on Thursday, 10 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 can pick up their key in Apartment 2A, 363 East Stewart Street, upon arrival. Check out time is before noon.

SYMPOSIUM ADMINISTRATION

* * *

SYMPOSIUM SUPPORT

Air Force Office of Scientific Research
Bolling Air Force Base, DC

SYMPOSIUM CHAIRMEN

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AGENDA

WEDNESDAY MORNING, 9 JUNE 1982

SESSION A

Chairman: Professor C. G. Pantano
The Pennsylvania State University
University Park, PA

8:00 a.m. Registration, Lobby of Miriam Hall

9:00 a.m. Introductory Remarks

A-1 9:20 a.m. INDUSTRIAL APPLICATIONS OF ESCA: A STUDY OF Si AS IT OCCURS IN ABSORBENTS, CATALYSTS AND THIN FILMS
T. L. Barr (*Invited*)
Universal Oil Products Inc.
Des Plaines, IL

A-2 10:10 a.m. CHARACTERIZATION OF MOLYBDENUM-PLATINUM CATALYSTS SUPPORTED ON γ-ALUMINA BY X-RAY PHOTOELECTRON SPECTROSCOPY
J. E. deVries*, H. L. Yao, R. J. Baird, and H. S. Gandhi
Ford Motor Company
Dearborn, MI

10:30 a.m. *COFFEE BREAK*

A-3 10:50 a.m. FUNDAMENTAL STUDIES OF SILICA/SILYLATED SURFACES
Dwight E. Williams* and J. P. Cannady
Dow Corning Corporation
Midland, MI

A-4 11:10 a.m. APPLICATIONS OF FOURIER TRANSFORM INFRARED PHOTOACOUSTIC SPECTROSCOPY (FT-IR/PAS) TO SURFACE AND CORROSION PHENOMENA
Joseph A. Gardella, Jr.*, William P. McKenna, Da-Zhen Jiang, and Edward M. Eyring
University of Utah
Salt Lake City, UT

A-5 11:30 a.m. OXIDATION OF AIRCRAFT FUEL DEPOSITS ON METAL SURFACES
James L. Lauer*, Leonhard E. Keller, and Simon Fung
Rensselaer Polytechnic Institute
Troy, NY

A-6 11:50 a.m. CHARACTERIZATION OF FAILURE SURFACES OCCURRING ALONG WEAK BOUNDARY LAYERS
W. L. Baun
Air Force Wright Aeronautical Laboratories
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*Denotes presenting author.

12:10 p.m. *LUNCH*
VENDORS EXHIBITS OPEN

WEDNESDAY AFTERNOON, 9 JUNE 1982

SESSION B

Chairman: Dr. R. W. Springer
Los Alamos National Laboratory
Los Alamos, NM

B-1 1:30 p.m. ION IMPLANTATION IN CORROSION RESEARCH AND CORROSION PROTECTION
G. K. HUBLER (*Invited*)
Naval Research Laboratory
Washington, DC

B-2 2:20 p.m. ROOM TEMPERATURE OXIDATION OF CHROMIUM AND NICKEL IMPLANTED IRON, AND BULK ALLOYS
P. J. Osborne, P. J. K. Paterson*,
Royal Melbourne Institute of Technology
Melbourne, AUSTRALIA, and
O. Spillecke
Fritz-Haber Institut Der M.P.G.
Berlin, GERMANY

B-3 2:40 p.m. SURFACE ANALYSIS OF COMMERCIALLY MADE MIRRORS
T. M. Thomas*, J. R. Pitts, and A. W. Czanderna
Solar Energy Research Institute
Golden, CO

3:00 p.m. *COFFEE BREAK - (EXTRA TIME ALLOWED FOR VENDORS EXHIBITS)*

B-4 4:00 p.m. THE APPLICATION OF TAPER-SECTIONING TECHNIQUES FOR DEPTH PROFILING USING AUGER ELECTRON SPECTROSCOPY
J. M. Walls*, I. K. Brown, and D. D. Hall
Loughborough University of Technology
Loughborough, Leicestershire, ENGLAND

B-5 4:20 p.m. THE IMPACT OF IMPROVED SPATIAL RESOLUTION IN ESCA
Robert Chaney, Michael Kelly, and Charles Bryson*
Surface Science Laboratories Inc.
Mountain View, CA

B-6 4:40 p.m. COMBINED XPS AND ISS CHARACTERIZATION OF Bi - Te - S COMPOUNDS
Paul A. Lindfors*, Thomas W. Rusch
Perkin-Elmer
Eden Prairie, MN, and
William A. Schwalm
University of North Dakota
Grand Forks, ND

5:00 p.m. *INFORMAL RECEPTION*

THURSDAY MORNING, 10 JUNE 1982

SESSION C

Chairman: Dr. T. L. Barr
Universal Oil Products
Des Plaines, IL

8:00 a.m. Registration, Lobby of Miriam Hall

C-1 8:30 a.m. SURFACE AND GRAIN BOUNDARY SEGREGATION RELATED TO THE TEMPER EMBRITTLEMENT OF A 2 1/4Cr-1 Mo STEEL
P. Ho*, D. F. Mitchell, and M. J. Graham
National Research Council of Canada
Ottawa, CANADA

C-2 8:50 a.m. FRACTURE SURFACE ANALYSIS OF EMBRITTLED STEELS BY AUGER ELECTRON SPECTROSCOPY
W. R. Broughton, P. J. K. Paterson*,
Royal Melbourne Institute of Technology
Melbourne, AUSTRALIA, and
W. J. Pollock
Aeronautical Research Laboratories
Melbourne, AUSTRALIA

C-3 9:10 a.m. AES STUDIES OF PHOSPHOROUS IMPURITY EFFECTS ON HYDROGEN EMBRITTLEMENT OF Ni_2Cr
J. A. Kargol, R. Asfahani, T. M. Parrill, and M. V. Zeller*
University of Notre Dame
Notre Dame, IN

C-4 9:30 a.m. AUGER ELECTRON SPECTROSCOPIC STUDY ON GUN TUBE EROSION AND CORROSION
Sin-Shong Lin
Army Materials and Mechanics Research Center
Watertown, MA

C-5 9:50 a.m. APPLICATIONS OF SURFACE ANALYSIS IN THE WIRE INDUSTRY
David A. Stout
National Standard Company
Niles, MI

10:10 a.m. *COFFEE BREAK*
VENDORS EXHIBITS REOPEN

C-6 10:40 a.m. COMPOSITIONAL, AND MORPHOLOGICAL ANALYSIS OF THE SURFACE AND NEAR-SURFACE REGION OF VAPOR-DEPOSITED AgCl FILMS
W.-A. Luo*, M. G. Lagally
University of Wisconsin-Madison,
Madison, WI, and
A. Belkind and E. Ezell
Airco CRD
Murray Hill, NJ

C-7 11:00 a.m. OPTICAL PROPERTIES OF THIN In-Sn OXIDE FILMS
K. D. Christian and S. R. Shatynski
Rensselaer Polytechnic Institute
Troy, NY

C-8 11:20 a.m. SURFACE ANALYSIS OF TESTED SOLAR COATINGS
R. W. Springer*
Los Alamos National Laboratory
Los Alamos, NM, and
R. A. Osiecki
Lockheed Research Laboratory
Palo Alto, CA

C-9 11:40 a.m. QUANTITATIVE ANALYSIS OF LIGHT ELEMENTS IN FILMS USED IN SOLAR ENERGY CONVERSION
David D. Allred
University of Arizona
Tucson, AZ

12:00 p.m. *LUNCH*

THURSDAY AFTERNOON, 10 JUNE 1982

SESSION D

Chairman: Dr. G. K. Hubler
Naval Research Laboratory
Washington, DC

1:30 p.m. *VENDORS EXHIBITS CLOSE*

D-1 1:30 p.m. APPLICATIONS OF RUTHERFORD BACKSCATTERING
James W. Mayer (*Invited*)
Cornell University
Ithaca, NY

D-2 2:20 p.m. RUTHERFORD BACKSCATTERING ANALYSIS OF CATALYSTS
K. R. Padmanabhan* and Y. F. Hsieh
Wayne State University
Detroit, MI

D-3 2:40 p.m. ION IMPLANTED n TYPE SURFACE LAYERS IN GaAs-
SUBSTITUTIONALITY AND DOPANT PRECIPITATION
P. P. Pronko*, A. K. Rai, and R. S. Bhattacharya
Universal Energy Systems Inc
Dayton, Ohio

3:00 p.m. *COFFEE BREAK*

D-4 3:30 p.m. COMPOSITIONAL AND GROWTH PROPERTIES OF THIN Cr-Si FILMS
S.-H. Lin, V. Conger*, D. Stevenson, and M. G. Lagally
University of Wisconsin-Madison
Madison, WI, and
P. C. Karulkar
Rockwell International
Anaheim, CA

D-5 3:50 p.m. THE INFLUENCE OF LOW ENERGY ION BOMBARDMENT ON THE CHEMISTRY
OF THE SILICON DIOXIDE SURFACE
J. H. Thomas III*
RCA Laboratories
Princeton, NJ, and
S. HOFMANN
Max-Planck-Institut for Metallforschung
Stuttgart, WEST GERMANY

D-6 4:10 p.m. XPS STUDIES OF ION-INDUCED DAMAGE ON SEMICONDUCTOR AND
INSULATOR SURFACES
A. B. Christie, J. Lee, I. Sutherland, and J. M. Walls*
Loughborough University of Technology
Loughborough, Leicestershire, ENGLAND

D-7 4:30 p.m. USE OF SURFACE BEHAVIOR DIAGRAMS TO COMPARE ANODIC AND
PHOTOCHEMICAL OXIDES OF $Hg_{0.8}Cd_{0.2}Te$
G. D. Davis*, S. P. Buchner, W. A. Beck, and N. E. Byer
Martin Marietta Laboratories
Baltimore, MD

D-8 4:50 p.m. QUANTITATIVE ANALYSIS IN CHEMICALLY DISSIMILAR MATRICES
BY SECONDARY ION MASS SPECTROMETRY
W. Katz, G. Smith*, and C-Y. Wei
General Electric Research and Development Center
Schenectady, NY

FRIDAY MORNING, 11 JUNE 1982

SESSION E

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

8:00 a.m. Registration, Lobby of Miriam Hall

E-1 8:30 a.m. THERMAL DESORPTION OF CO, O_2 and CH_4 MOLECULES FROM Ni(111) SINGLE CRYSTALS
Gordon A. Sargent, J. Chao, and G. B. Freeman*
University of Kentucky
Lexington, KY

E-2 8:50 a.m. ASSESSMENT OF DIFFERENT MODELS FOR QUANTITATIVE AUGER ANALYSIS IN APPLIED SURFACE STUDIES
E. Minni* and E. Suoninen
University of Turku
Turku, FINLAND

E-3 9:10 a.m. THE AUGER PARAMETER APPLIED TO SOLID AND GASEOUS COMPOUNDS
C. D. Wagner*
Surfex Company
Oakland, CA, and
J. Ashley Taylor
Perkin-Elmer Corporation
Eden Prairie, MN

E-4 9:30 a.m. LOCALIZED MEASUREMENT OF D_2O CONCENTRATIONS USING NUCLEAR REACTION ANALYSIS WITH APPLICATION TO MOISTURE DIFFUSION IN COMPOSITES
R. L. Schulte*, R. J. DeIasi and J. R. Whiteside
Grumman Aerospace Corporation
Bethpage, NY

E-5 9:50 a.m. AUGER ELECTRON SPECTROSCOPY STUDIES IN Y-Fe-O and La-O SYSTEM
Seong Rae Lee, A. E. Miller, and M. V. Zeller*
University of Notre Dame
Notre Dame, IN

E-6 10:10 a.m. QUANTITATIVE AES ANALYSIS OF Fe-Cr and Fe-Ni-Cr ALLOYS
W. Matthews, P. J. K. Paterson* and H. K. Wagenfeld
Royal Melbourne Institute of Technology
Melbourne, AUSTRALIA

10:30 a.m. *COFFEE BREAK*

E-7 10:50 a.m. DURABILITY OF SOLID FLUOROPOLYMER THIN FILMS ON METAL
SURFACES FOR SILICONE OIL CREEP
C. A. Haque
Bell Laboratories
Columbus, OH

E-8 11:10 a.m. SURFACE TECHNIQUES APPLIED TO THE ANALYSIS OF DEFECTS ON
EXTRUDED ALUMINUM ALLOYS
K. J. Holub*
Westinghouse Electric Corporation
Baltimore, MD, and
L. J. Matienzo
Martin Marietta Laboratories
Baltimore, MD

E-9 11:30 a.m. STUDY OF CHROMIC-ACID-ANODIZED TITANIUM OXIDE USING AUGER
LINE SHAPES
G. D. Davis, M. Natan, and K. A. Anderson*
Martin Marietta Laboratories
Baltimore, MD

E-10 11:50 a.m. AN RBS STUDY OF INHIBITOR REACTIONS WITH THE SURFACE OF
ALUMINUM IN AQUEOUS ELECTROLYTES
R. C. McCune
Ford Motor Company
Dearborn, MI, and
R. L. Shilts
General Ionex Corporation
Newburyport, MA

E-11 12:10 p.m. APPLICATION OF LOW DAMAGE SIMS/AES/XPS OF THE Ba-O-W
SYSTEM TO CATHODE CHARACTERIZATION
B. C. Lamartine, J. v.Czarnecki*, and T. W. Haas
Air Force Wright Aeronautical Laboratories
Wright-Patterson Air Force Base, OH

E-12 12:30 p.m. THE USE OF THE VAPOR COLLECT METHOD AND LINE OF SIGHT
RESIDUAL GAS ANALYSIS TO DETERMINE EVAPORATION RATES OF
Ba FROM IMPREGNATED TUNGSTEN DISPENSER CATHODES
G. L. Jones* and J. T. Grant
University of Dayton
Dayton, OH

E-13 12:50 p.m. THE ANALYSIS OF PROFILE STRATA FOR SURFACE TEXTURE
SPECIFICATION
Richard B. Zipin
The Bendix Corporation
Dayton, OH

1:10 p.m. *SYMPOSIUM ADJOURNMENT*

Industrial Applications of ESCA:
A Study of Si as It Occurs in
Adsorbents, Catalysts and Thin Films

Tery L. Barr
Corporate Research Center
UOP Inc.
10 UOP Plaza
Des Plaines, IL 60016

This cyclical talk is focused upon ESCA studies of silicon in a number of diverse environments. First, the ESCA analysis of Si in its "clean" zero valent form is reported. Second the changes in these ESCA spectra are described, when the element is oxidized to SiO_2. Third, the changes experienced by silica are documented when metal aluminates are injected to create such common adsorbents as clays and zeolites. As a fourth example, unique ESCA analyses are reported of the changes in silica when it is employed as a support for a metal oxide catalyst during on-line use situations. Finally, the cycle is completed by describing thin films of ($\sim 500\mathring{A}$) SiO_2 on Si^o. Contrary to previous contentions, it is shown that a detailed description of the SiO_2 film, the resulting interface, and the Si^o underlayer may be achieved employing common ion-sputter etching and ESCA analysis.

Wednesday Morning - 9:20 a.m.

CHARACTERIZATION OF MOLYBDENUM-PLATINUM CATALYSTS SUPPORTED ON γ-ALUMINA BY X-RAY PHOTOELECTRON SPECTROSCOPY

J. E. deVries, H. L. Yao, R. J. Baird, and H. S. Gandhi

Engineering and Research Staff
Ford Motor Company
Dearborn, MI 48121

ABSTRACT

X-ray photoelectron spectroscopy (XPS) has been used to investigate MoO_3 and $MoO_3 + PtO_2$ supported on γ-Al_2O_3. After H_2 reduction of MoO_3/Al_2O_3 at 500°C Mo(VI), (V), and (IV) coexist on the surface of the catalyst. Hydrogen reduction of dispersed $MoO_3 + PtO_2$ on γ-Al_2O_3 shows a partial reduction of Mo(VI) to Mo(V) and Mo(IV) occuring at significently lower temperatures than is the case with the supported MoO_3 alone. Higher loadings of PtO_2 correlate directly with relatively more reduction of the Mo(VI) to Mo(V) and Mo(IV).

Wednesday Morning - 10:10 a.m.

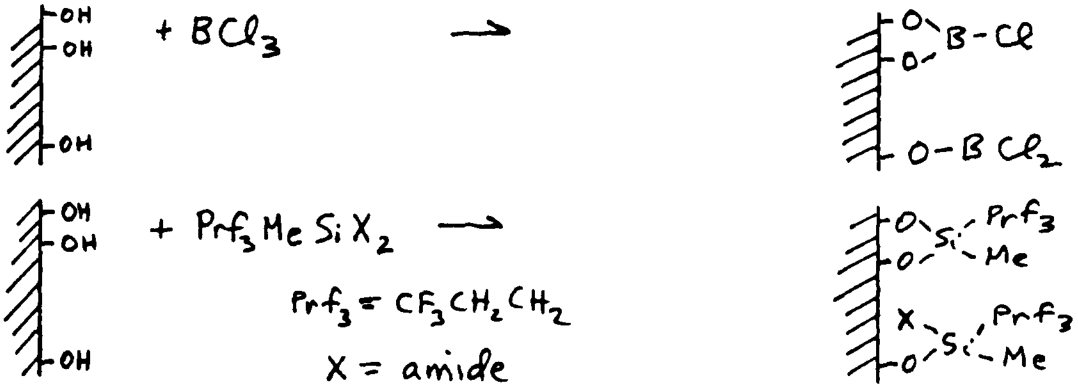
FUNDAMENTAL STUDIES OF SILICA/SILYLATED SURFACES

Dwight E. Williams and J. P. Cannady
Dow Corning Corporation
Midland, Michigan 48640

A variety of hyroxyl-bearing oxide surfaces have been transformed in various ways and examined by ESCA. The studies have focused on understanding and controlling the distribution of hydroxyl groups on organosilicon (silylated) surfaces and on the inorganic substrate such as vitreous or high surface area silica, sapphire, titania, and zirconia. Novel derivatization reagents that bore various elemental tags were used to make ESCA more specific.

An ultra-high vacuum (UHV) furnace/reactor was designed and assembled which allows us to control the total amount of hyroxyl groups and vary the ratio of adjacent to isolated groups. This UHV apparatus provides the somewhat unusual conditions of up to 900°C and 10E-10 Torr. Subsequent gas/solid reactions are conducted without intervening atmospheric exposure.

Tests of various derivatization schemes to measure total hydroxyl and to distinguish between adjacent vs. isolalated hydroxyl will be discussed, such as:



Mixed carbinol/silanol surfaces, and pure carbinol and pure silanol surfaces were studied to develop and compare schemes to distinguish between these groups. Three schemes were tested, as follows: I. Carbinol Derivative Hydrolyzed; II. Silanol Derivative Hydrolyzed (Converse of Scheme I); III. Kinetically Controlled Labelling.

In these schemes ESCA labelled silylating reagents, organic hydroxyl protecting reagents, and a reagent which shows a marked carbinol specificity (i.e. isocyanate) were used.

This research has been supported by NSF Grant CHE7823123 under the Industry/University Cooperative Program.

Wednesday Morning - 10:50 a.m.

Applications of Fourier Transform Infrared Photoacoustic Spectroscopy (FT-IR/PAS) to Surface and Corrosion Phenomena

Joseph A. Gardella, Jr., William P. McKenna, Da-Zhen Jiang and Edward M. Eyring, Department of Chemistry, University of Utah, Salt Lake City, Utah 84112

The development of FT-IR/PAS as a surface analytical technique is described by analytical studies presented in this paper. Studies have focused on the use of FT-IR/PAS alone and in comparison with other surface techniques to describe the analytical capabilities of the technique. Comparisons to transmission and reflection FT-IR and with X-Ray Photoelectron Spectroscopy (XPS or ESCA) have yielded a variety of information especially in the realm of polymer and catalyst surfaces and to the elucidation of species formed in an HCN/NH_3 corrosion reaction.

Spectral information available from FT-IR/PAS experiments includes an enhanced surface response and thermal properties convoluted with the normal infrared vibrational absorption function. By comparing transmission and reflection spectroscopy of polymers such as polystyrene, and various biopolymers to FT-IR/PAS we can define the limits of surface sensitivity and the thermal response which make FT-IR/PAS a more generalizable surface infrared technique.

Analysis of catalyst surfaces provides another example of the unique capabilities of FT-IR/PAS to provide surface infrared analysis of both gas phase and adsorbed reactants. Both supported metal oxide and transition metal complex systems have been examined. Comparison of ESCA and FT-IR/PAS results provide information on the molecular structure of adsorbants and catalytically active species, besides allowing reactions to be monitored in situ.

Finally, the utility of FT-IR/PAS for the analysis of corrosion species is illustrated by the analysis of an $Fe/NH_3/HCN$ system. FT-IR/PAS combined with ESCA allowed an unambiguous assignment of the structure of the resulting compounds which were water soluble, allowing for the observed pitting corrosion.

Wednesday Morning - 11:10 a.m.

Oxidation of Aircraft Fuel Deposits on Metal Surfaces

James L. Lauer, Leonhard E. Keller and Simon Fung
Department of Mechanical Engineering,
Aeronautical Engineering & Mechanics
Rensselaer Polytechnic Institute
Troy, New York 12181

Fuel deposits collected on stainless steel strips in the large test rig of NASA-Lewis Research Center were analyzed in situ by polarization infrared Fourier Micro-emission spectrophotometry, a procedure recently developed [1]. A deposit from a fuel containing a small concentration of a mercaptan was found to have infrared bands corresponding to methylene rocking and carbon/carbon stretching in the 630 - 1230 cm^{-1} region. However, there were spectral differences between fresh samples and year-old samples in this region, mainly band shifts of about 10 cm^{-1}. Since bands containing oxygen do not occur in this region, these differences had to be ascribed to changes of molecular weight and solid state structure. To confirm these effects, a fresh sample on a similar metal strip was subjected to fast oxidation by an hour's exposure to an air stream at 120°C. The infrared spectrum of this material was between that of the fresh and the year-old sample. Heating in nitrogen had a similar but less pronounced effect.

These results were interpreted in terms of changes in the polymeric structure of these deposits. They confirm the widely held view of the presence of very active free radicals in fresh deposits and point to the need of care in handling and storing fuel deposits for analysis.

Preliminary measurements of deposit thickness and contour with a phase-locked optical interference microscope are consistent with these results.

This work was funded in part by Grant No. NSG-3205 from the National Aeronautical and Space Administration and by Grant No. AFOSR-81-0005 from the Air Force Office of Scientific Research.

[1] Lauer, J.L., and Keller, L.E., "Analysis of Aircraft Fuel Line Deposits by Polarization Infrared Fourier Micro-Emission Spectrophotometry," *Application of Surface Science*, 8, (1981).

Wednesday Morning - 11:30 a.m.

CHARACTERIZATION OF FAILURE SURFACES OCCURRING ALONG WEAK BOUNDARY LAYERS

W. L. Baun

Nonmetallic Materials Division
Materials Laboratory
Air Force Wright Aeronautical Laboratories, AFWAL/MLBM
Wright-Patterson Air Force Base, Ohio 45433

Failure surfaces at an interface where two dissimilar materials were in direct contact such as evaporated films or adhesive bonds seldom show a clean 100% "adhesive" mode of failure. Even when the locus of failure visually appears to be interfacial, closer examination by sensitive analytical surface methods show mixed mode failure. When interfacial failures do occur, they normally result from the formation of a weak boundary layer (WBL), (assuming that initial adhesion was achieved). Examination of WBL failure surfaces with ISS, SIMS, AES and XPS suggest that WBL's are formed by several different processes including diffusion at elevated temperatures, chemical conversion of an oxide species, and transport phenomena resulting in an elemental concentration at an interface. The latter type of WBL is illustrated by the sharp concentration of fluorine at the metal/oxide interface of most valve metals anodized in electrolytes containing fluorine in solution.

Wednesday Morning - 11:50 a.m.

Ion Implantation in Corrosion Research and Corrosion Protection

G. K. Hubler
Naval Research Laboratory
Washington, DC 20375

Ion implantation is being investigated as a technique for the beneficial modification of surface-sensitive, life-limiting properties of metals including resistance to corrosion, wear, and fatigue. Ion implantation is attractive because it features the modification of surface properties without affecting the bulk metal and because there are no changes in dimension so that the process can be applied to otherwise finished parts. Unique metastable surface alloys can be produced which have practical applications but also provide the materials researcher with a wide variety of new and interesting research problems.

Typical implantation depths are about 100nm and so are well suited for study by means of surface analysis techniques. Examples of corrosion research of ion-implanted metals will be given which demonstrate the utility of combining ion implantation with the surface sensitive analysis techniques for the elucidation of corrosion mechanisms. Applications of ion implantation for corrosion protection within the Navy will be presented.

Wednesday Afternoon - 1:30 p.m.

ROOM TEMPERATURE OXIDATION OF CHROMIUM AND NICKEL IMPLANTED IRON, AND BULK ALLOYS

P.J. Osborne*, P.J.K. Paterson* and O. Spillecke**

* Applied Physics Department, Royal Melbourne Institute
of Technology, Melbourne, Australia.

** Fritz-Haber Institut Der M.P.G., D-1000, Berlin 33.

This investigation sets out to directly compare the room temperature oxidation of Cr and Ni implanted Fe with bulk alloys in the range of commercial stainless steels. The samples investigated include pure single crystal (100) Fe and polycrystalline Fe foils implanted with Ni and Cr-ions at 40 keV, with doses to match the commercial stainless steel compositions. Analyses of the surfaces were carried out by Auger Electron Spectroscopy (AES) and Ar-ion sputter profiling. The samples were oxidized for two weeks at atmospheric pressure and room temperature.

Results revealed that both measured and calculated Ni-implant distributions in Fe were gaussian, with the measured distribution showing a greater degree of skewness. The measured Cr-implant distribution in Fe is significantly different from that for the calculated distribution. The measured Cr^{+++} distribution peaked sharply at 6nm, (coinciding with the calculated peak in the vacancy distribution produced by implantation), then again with a broader peak at approximately 30nm. These results were representative of the Ni^{++} and Cr^{+++} distributions in both sets of single crystals and polycrystalline foils.

The bulk alloys possessed a greater surface enrichment of Cr than the implanted crystals and foils. The room temperature oxidation of the samples revealed that the oxygen concentration variation with depth for the bulk alloys was lower compared to the implanted samples. The oxide thickness was inversely related to the surface Cr concentration.

Wednesday Afternoon - 2:20 p.m.
B-2

SURFACE ANALYSIS OF COMMERCIALLY MADE MIRRORS*

T. M. Thomas, J. R. Pitts, and A. W. Czanderna

Solar Energy Research Institute[+]
1617 Cole Boulevard
Golden, Colorado 80401

Most commercially made mirrors, consisting of a multilayer stack of glass/Ag/Cu/paint, are made using a wet electroless process. Two of the failure modes of atmospherically weathered mirrors are delamination at the Ag/glass interface and electrochemical corrosion of the copper and silver. AES, XPS, SIMS, and ISS have been used for surface analysis during profiles to reach the glass/Ag, Ag/Cu, and Cu/paint interfaces as well as to analyze for impurities present in each layer of the mirror. Surface analysis results will be presented from studies of complete mirror stacks and of partially made mirrors prepared in our laboratory. Emphasis in the discussion will be to elucidate the chemistry of the Ag/glass interface and the corrosion induced by impurities trapped in the mirror during fabrication. A model for the mechanism of bonding silver to glass during mirror preparation will be discussed.

*Work performed under U.S. DOE Contract No. EG-77-C-01-4042.

[+] A U.S. DOE facility.

Wednesday Afternoon - 2:40 p.m.

THE APPLICATION OF TAPER-SECTIONING TECHNIQUES FOR DEPTH PROFILING USING AUGER ELECTRON SPECTROSCOPY

by

J.M. Walls, I.K. Brown and D.D. Hall
Department of Physics
Loughborough University of Technology
Loughborough, Leicestershire LE11 3TU
England

ABSTRACT

The provision of accurate composition-depth profiles to depths $>1 \mu m$ is important in the characterisation and failure diagnosis of thick films, surface coatings, surface treatments and many other important applications. Profiles to these depths are best achieved by mechanically polishing the surface by angle lapping or ball cratering. The latter technique has been shown to be a convenient and accurate method and employs a rotating steel ball, coated with fine diamond paste, to abrade a well-defined spherical crater in the sample, the depth and width of which can be controlled accurately. With all taper-sectioning techniques it is necessary to first clean the specimen in situ, using a scanning ion beam; the profile is then obtained by point-by-point Auger analysis down the side of the taper-section.

The depth resolution of depth profiles obtained by taper-sectioning techniques depends on the diameter of the probing beam and the extent of the mechanical roughness produced by the wear process. In this paper a detailed study of the wear process will be presented, the interpretation of which enables the abrasion conditions for different materials to be defined for optimum surface finish, and depth resolution. Finally, composition-depth profiles through commercially important nitrocarburizing surface treatments and protective surface coatings are presented to illustrate the usefulness of the techniques.

Wednesday Afternoon - 4:00 p.m.

The Impact of Improved Spatial Resolution in ESCA

Robert Chaney, Michael Kelly, and Charles Bryson

Surface Science Laboratories, Inc.
1206 Charleston Road
Mountain View, CA 94043
(415)962-8767

An improvement in the spatial resolution available with ESCA (Electron Spectroscopy for Chemical Analysis) provides an increase in the range of material problems addressable by surface sensitive techniques. Many characterization problems are best approached using ESCA because of the need for chemical bonding information, the need to minimize the damage induced by the measurement, and the need to minimize matrix effects affecting quantitization of the results. Prior to recent advances, the applications of ESCA have been limited by the large area analyzed, i.e. ~ 1-5 mm^2. We will describe a small spot ESCA spectrometer able to analyze areas down to .05 mm^2, and will present problems solved with this instrument. In addition, we will discuss the impact this improvement will have in the types of problems addressable with ESCA.

Wednesday Afternoon - 4:20 p.m.

Combined XPS and ISS Characterization of Bi - Te - S Compounds

Paul A. Lindfors, Thomas W. Rusch
Perkin-Elmer/Physical Electronics Division
6509 Flying Cloud Drive
Eden Prairie, Minnesota 55344
(612) 828-6411

and

William A. Schwalm
Department of Physics
University of North Dakota
Grand Forks, North Dakota 58201

Xray photoelectron spectroscopy (XPS or ESCA), including angularly resolved measurements, and ion scattering spectroscopy (ISS) were performed on compounds containing Bi, Te and S. For bismuth tellurium sulfide single crystal specimens the angularly resolved ESCA measurements indicate surface enrichment of Te, some surface depletion of S, plus oxygen and carbon as surface contaminants. For the same specimen the ISS measurements also indicate the surface enrichment of Te but interpretation of the distribution of S is more difficult. Correlation of the XPS and ISS data is discussed.

Wednesday Afternoon - 4:40 p.m.

Surface and Grain Boundary Segregation Related to the Temper Embrittlement of a $2\frac{1}{4}$Cr-1 Mo Steel

P. Ho*, D.F. Mitchell and M.J. Graham
Division of Chemistry
National Research Council of Canada
Ottawa, Canada K1A 0R9

Abstract

Temper embrittlement of a $2\frac{1}{4}$Cr-1 Mo steel is considered to be promoted by the segregation of impurities, such as P, to grain boundaries. Analysis by scanning Auger microscopy and XPS of in-situ fractured surfaces has quantified the extent of grain boundary segregation for differently heat treated steels. XPS has also been used to give an indication of the chemical state of the P and the interaction between P and Mo.

The grain boundary segregation data have been complemented by studies of surface segregation of P using reflection high-energy electron diffraction (RHEED) and X-ray emission analysis (XRE). P diffusion coefficients and activation energies have been derived.

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* NRC Guest Worker / Centre for the Study of Materials, University of Toronto, Toronto, Canada M5S 1A4.

Thursday Morning - 8:30 a.m.

FRACTURE SURFACE ANALYSIS OF EMBRITTLED STEELS BY AUGER ELECTRON SPECTROSCOPY

By

*W.R.Broughton, *P.J.K. Paterson and **W.J. Pollock

*Materials Group, Applied Physics Department, Royal Melbourne Institute of Technology, G.P.O. Box 2476V, Melbourne. Australia.

** Aeronautical Research Laboratories, Defence Science & Technology Organisation, Department of Defence, G.P.O. Box 4331, Melbourne. Aust.

Auger Electron Spectroscopy has proved to be a successful surface analysis technique in the study of impurity and solute atom segregation at prior austenite grain boundaries in temper embrittled steels. The technique enables embrittled specimens to be fractured intergranularly in the Auger spectrometer by impact loading at temperatures below the ductile-brittle transition temperature at ultra high vacuum pressures $\approx 10^{-7}$Pa. However, to investigate low alloy steels susceptible to hydrogen assisted cracking (hydrogen embrittlement) it was necessary to modify an Auger Spectrometer enabling intergranular subcritical crack growth, final fracture and subsequent Auger analysis of hydrogen precharged specimens.

The paper presents the results obtained in the investigation of impurity and solute atom segregation in temper embrittled Fe-Ni-Cr-C-P steels; correlating tempering temperature, ageing time, hardness and grain boundary concentration of segregants. This paper, also, describes the modifications of the Auger spectrometer to ensure relatively contaminant free intergranular fracture surfaces of hydrogen precharged D6AC and EN26 Steel specimens. The results relating to D6AC from a preliminary investigation, correlate crackgrowth rate, mechanical properties and grain boundary concentration of segregants.

It is therefore expected that Auger Electron Spectroscopy with the developed modifications will serve a multi-purpose role in extending the knowledge in the areas of microstructural control and environmental fracture.

Thursday Morning - 8:50 a.m.

C-2

AES Studies of Phosphorus Impurity Effects on Hydrogen Embrittlement of Ni_2Cr

J. A. Kargol, R. Asfahani, T. M. Parrill and M. V. Zeller

College of Engineering
University of Notre Dame
Notre Dame, IN 46556

High strength Ni-based stainless alloys are excellent materials for use in the nuclear and petroleum industries in which resistance to corrosion and environment assisted cracking in aggresive aqueous environments is mandatory. In current applications of those alloys, thermal-mechanical treatments are often used to increase alloy strength; however, that processing may decrease resistance to corrosion and embrittlement. In particular, the processing may cause an increase in hydrogen embrittlement (HE).

Suggested explanations for the increase in HE for the Ni alloys are alloy ordering and impurity segregation. The roles of ordering and segregation on HE were studied for Ni_2Cr, the basic ordering composition in the Ni alloy series, containing three different phosphorus (P) impurity concentrations. Time-to-failure tests were conducted to obtain a relationship between aging conditions and HE resistance. Hardness as a function of aging was determined for the Ni_2Cr alloys. Ordering effects were also examined and correlated to the cracking resistance.

Auger Electron Spectroscopic (AES) analysis was made for in situ fracture surfaces of the variously thermal aged plus cathodically H precharged Ni_2Cr alloys. The Ni_2Cr alloys which were thermally aged for extended times of 100h or longer underwent intergranular fracture; the AES analysis indicated that considerable P segregation occurred to grain boundaries in the aged Ni_2Cr. The Ni_2Cr alloys aged for shorter periods exhibited both intergranular and ductile fracture for the in situ tests.

Thursday Morning - 9:10 a.m.

Auger Electron Spectroscopic Study on Gun Tube Erosion and Corrosion

by

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The gun tube erosion is a highly complex phenomenon in which a number of severe thermal, chemical and mechanical interactions with the bore surface takes place in a short time. These reactions occur at the same time and vary in intensity along the length of the barrel. The extent of erosion depends on specified ammunition and firing schedules. In the chemical erosion process, the reaction of gun surface with hot and reactive gases of propellant produces thermally incompatible substances to form cracks, chips and scales on the bore surface. These loosened materials are then removed by frictions of the gas and the projectile.

The eroded surfaces of three large caliber guns have been examined with the surface analytical technique using a PHI model 548 ESCA/AES instrument. The depth profile of the eroded surface is accomplished by mechanically exposing the interiors of the bore surface. In general there are two or more physically distinguishable layers; the top layer of cracked and chipped reaction or molten materials, sometimes a thin second layer of white colored materials, and the last layer of unreacted metal phases.

The chemical compositions of the top layer vary to a large extent with the ammunition used in firing. The elements such as Cr, Fe, Sn, Cu, Mn, Zn, Na, Ca, Si, O, S, and C are frequently observed. The formation of metal oxides, carbides and nitrides, cause the reacted bore surface to be thermally incompatible after repeated firing-cooling cycles. Cracks, chips, and scales are found to dominate the physical appearence of the top layer. The white second layer is found to consist of mostly iron and carbon. The third layer is the original martensite phase, and the composition is not yet affected by chemical reaction of propollant gases.

Thursday Morning - 9:30 a.m.

APPLICATIONS OF SURFACE ANALYSIS IN THE WIRE INDUSTRY

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The quality of wire is judged not only by its physical properties such as tensile strength and fatigue resistance but also by its surface finish. The surface roughness, oxide formation, cleanliness, and plating homogeneity and porosity are just a few of the surface properties that can influence the performance of a wire product. Coupled to this is the large amount of surface area generated in drawing wire. For example, a ten pound spool holds nine miles of 0.006" diameter Stainless Steel wire. For these reasons surface analysis has become important both to the manufacture and consumer of wire products.

When surface analysis equipment such as AES, ESCA, and SIMS was first becoming commercially available in the late sixties and early seventies, the wire industry was beginning to enter a phase of technological development for many of its products. Wire manufacturers and users began using surface analysis to investigate such diverse topics as adhesion of brass plated automobile tire cord to rubber, fracture studies of tungsten wire, diffusion of layered deposits, and thermocompression bonding. Examples of some of these topics as well as illustrations of the usefulness of surface analysis in solving production problems will be presented.

Thursday Morning - 9:50 a.m.

COMPOSITIONAL AND MORPHOLOGICAL ANALYSIS OF THE SURFACE AND NEAR-SURFACE REGION OF VAPOR-DEPOSITED AgCl FILMS

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AgCl films have been deposited on glass substrates by evaporation from a thermal AgCl source. The surface and near-surface composition and surface morphology of the resulting films were analyzed as a function of film thickness (1000Å to 10μm), of deposition conditions (including different substrate temperatures and deposition in a high background of Ar), and of subsequent treatment of the films (including ageing in air, heating, and UV irradiation). The elemental and chemical compositions of the films as a function of depth were determined using XPS and AES. The effect of ion and electron beams on the compositional analysis was investigated. A depth-averaged compositional analysis of the films and their near-surface region was made using EDAX at two angles of incidence and two energies. The surface morphology of the films was determined by SEM. The surface compositions of films freshly prepared at various deposition conditions, films aged in air for a month, UV-irradiated films, and a bulk AgCl sample were compared. Cl enrichment is found at some conditions. Electron and ion beam effects lead to Cl depletion, the amount depending on beam parameters. XPS valence band, core-level chemical shift, and Auger parameter measurements are presented. Excess Cl at the surface is observable as a chemically shifted Cl 2p doublet. Cl depletion is observable in the Ag Auger line shape as a AgCl doublet and a Ag doublet shifted ≈1eV from it. The shape of the depth profile indicates that Cl is constantly replenished from deeper in the film by rapid diffusion, providing a dynamic surface composition between the true surface composition and that which would be achieved in the absence of diffusion. The surface behavior of these AgCl films is discussed in terms of the evaporation parameters and of mechanisms for AgCl decomposition.

Thursday Morning - 10:40 a.m.

OPTICAL PROPERTIES OF THIN IN-SN OXIDE FILMS

by

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Thin In-Sn oxide (ITO) has been shown to be an excellent semiconductor transparent to short wave radiation but opaque to long-wave energy. Such coatings on glass have been proposed as passive solar windows. Thin ITO films were prepared on glass using simple evaporation and in-situ oxidation. The films were evaporated using resistance heating to a carefully controlled thickness. After plating the alloys were oxidized at 100°C. The extent of oxidation was monitored and the optical properties examined periodically. In addition, a variety of In-Sn alloys were prepared and oxidized while their transmittance was measured over the wavelength range of 375 to 2500 millimicrons. A sharp shoulder was found at a wavelength of 492 millimicrons. Nearly 90% transmittance was found in the visible range whereas approximately 50% transmittance was observed in the infra-red region for very thin films. The extent of oxidation, film thickness and alloy composition not only modified the percentage of transmittance but also changed the plateau for the onset of transmittance. These sharp transitions are related to the electronic state of the solid oxide surface. The analysis of such states is dependent on the In to Sn ratio and thus the band gap. Initial indications are that Sn doping of In provide the narrowest surface band gap for the oxide and hence the most desireable window composition.

Thursday Morning - 11:00 a.m.

Surface Analysis of Tested Solar Coatings

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ABSTRACT

A variety of currently popular electroplated coatings have been tested and analyzed by surface analytical techniques. The types of coatings examined fall into four groups. Group 1 consists of black chrome coatings deposited on nickel substrates. Group 2 consists of black copper on a copper substrate. Group 3 consists of a black nickel on a nickel substrate. Group 4 consists of a thickness sensitive paint. Under the test conditions of various time exposures at 90C dry and 90C - 95%RH, only samples from group 1 and 2 were found to exhibit an optical change.

Auger Electron Spectroscopy depth profiles of the chrome and oxygen in group 1 revealed only a slight change in Cr/O ratio after integrating the depth profiles. Optical results show no difference in the high humidity samples, but a small change after dry exposure. X-ray Photo Electron analysis of the chrome 2p lines indicate a mixture of Cr^{+6}, Cr^{+3} and Cr in the unaffected samples. The samples that did exhibit a change in spectral response were found to contain only Cr^{+3} and Cr. This change in valence state due to a reduction oxidation reaction is believed to be responsible for the small observed change in morphology and spectral response. A simple model relating the changes in density with valence state will explain the observed changes.

The group 2 coating shows a simple increase in oxidation as revealed from integrated copper oxygen ratios obtained from AES depth profiles.

The other coatings are believed to be stable due to the nature of the oxides and protective binder.

The results of this study will be presented and discussed and the significance of these current findings will be related to early changes observed in solar coating.

Thursday Morning - 11:20 a.m.

QUANTITATIVE ANALYSIS OF LIGHT ELEMENTS IN FILMS USED IN SOLAR ENERGY CONVERSION David D. Allred, *Optical Sciences Center, University of Arizona, Tucson, Arizona 85721

The search for materials which will efficiently convert solar radiation to heat has created a need for the quantitative chemical analysis of thin films. The quantitative determination of the light element content of the films is particularly important.

We report on the use of an electron microprobe equipped with wavelength dispersive detection for the routine determination of the composition of thin films used in solar energy conversion for all elements having atomic numbers greater than that of Be. A number of the films which we studied by microprobe were also characterized by RBS and Auger. While the micoprobe lacks depth resolution, its sensitivity to trace elements and good lateral resolution recommend its use.

The microprobe is convenient, quantitative, nondestructive and sensitive to lateral differences in composition. There are, however, several obstacles which must be handled to employ it fully in thin film analysis. If the layer is thin, in our case .1 to 1.0μm, or the energy of the exciting beam high, the electron beam can completely penetrate the film, thereby exciting atoms in the underlying substrate. In addition, the quantitative determination of the light element fraction in the sample is complicated by high backgrouds, overlapping and unsuspected diffraction peaks, chemically induced changes in diffraction peaks, matrix effects and the unsuitability of conductive overcoats on nonconductive samples.

We have overcome these difficulties by a variety of strategies including decreasing the electron beam energy to 5 KeV, defocusing the beam to avoid depositing carbon and fabricating special calibration standards whose composition and chemical bonding are representative of the samples to be analyzed. Whenever possible we have deposited nonconducting films on conducting surfaces such as thin film molybdenum or silicon wafers. By this method, we have avoided the use of conductive overcoats which can change the apparent composition of the sample.

Special precautions were also taken in collecting data. The background counting rate is very high, the peaks are broad and high order diffraction lines from other elements in the samples are also detected on the lead stearate crystal used to detect the light elements. As a result, the location on either side of the peak where background was taken was carefully checked to avoid lines from other elements in the sample. The counting time for each of the background points was half as long as for the desired x-ray peak maximum and the total counting time was sometimes as long as 1000 sec to decrease statistical uncertainties.

By these means as little as .025 wt % boron was detected in a doped amorphous silicon film, and the quantitative analysis of oxygen and carbon in molybdenum thin films was found to agree with results of other techniques including Auger, Rutherford Backscattering and traditional chemical analysis. Unfortunately, due to the coincidence of x-ray lines some elements, in certain matrices, such as N in a-Ge:N, can not be analyzed.

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Thursday Morning - 11:40 a.m.

C-9

Applications of Rutherford Backscattering

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Rutherford backscattering and channeling measurements with MeV ^{4}He ions have found wide acceptance for the evaluation of composition depth profiles of thin-film, layered or ion-implanted structures. The driving forces for the application of backscattering techniques grew out of silicon integrated circuit technology. The cruicial region is the outer micron which contains ion-implanted emitters and sources, oxide layers and metal layers for contacts and interconnects. Backscattering provides a fast, non-destructive method of analysis without the requirement for standards.

This review covers the basic concepts of channeling and backscattering analysis by means of examples drawn from silicon technology. The formation of silicide layers - growth kinetics, stability and epitaxial relationships - and the application of pulsed laser annealing to ion implanted layers will be highlighted.

References

1. Backscattering Spectrometry, W. K. Chu, J. W. Mayer and M.-A. Nicolet, Academic Press, New York, 1978.

2. Materials Analysis by Ion Channeling, L. C. Feldman, J. W. Mayer and S. T. Picraux, Academic Press, New York, 1982.

3. Thin Films - Interdiffusion and Reactions, edited by J. M. Poate, K. N. Tu and J. W. Mayer, Wiley-Interscience, New York, 1978.

Thursday Afternoon - 1:30 p.m.

Rutherford Backscattering Analysis of Catalysts

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This paper describes the application of Rutherford backscattering in the analysis of Copper Chromite and Bismuth Molybdate Catalysts. The performance of Copper Chromite Catalyst in the production fatty alchohols was found to be influenced by the presence of BaO in the catalyst as analysed by RBS, PIXE and $O^{16}(d,p)O^{17}$ nuclear reaction.

In the case of Bismuth molybdate, the thermal decomposition of the catalyst was studied by RBS and nuclear reaction for oxygen. Samples annealed in oxygen or vacuum at temperatures ≃400°C tend to decompose into a 2:1 compound which probably is more stable and responsible for the catalytic behaviour.

Thursday Afternoon - 2:20 p.m.
D-2

ION IMPLANTED n TYPE SURFACE LAYERS IN GaAs - SUBSTITUTIONALITY AND DOPANT PRECIPITATION

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It is well established that the electrical activation of ion implanted n type dopants in GaAs occurs with very low efficiency in the formation of n^+ and n^{++} layers using medium dose implantation performed at room temperature. Improvements are obtained by implanting at higher temperatures; however, from a device production point of view, heating substrates represent a processing difficulty that is rarely or never used. Thus, it is important to understand the details of the matrix incorporation of n type dopants implanted at room temperature and subsequently annealed at elevated temperatures. We have done experiments for the n type dopants S, Si, and Se and the acceptor Cr. Proton induced x-ray excitation (PIXE) in combination with Rutherford backscattering-channeling (RBS-C) has been used to measure the damage recovery and relative substitutionality of the implanted ions in GaAs. These results are supported by and correlated with transmission electron microscopy (TEM) through detailed analysis of fine scale precipitation of the implanted dopants. It is found that an inverse relation exists between relative substitutionality and dopant precipitation. For example, the precipitation in the case of Si and S is found to be below 10% whereas the substitutionality is in the range of 70-90%. However, comparison with known electrical activation efficiencies clearly indicates that even under circumstances where dopants are in solid solution with the matrix and occupying substitutional lattice sites the electrical activation efficiencies remain low. In those cases the channeling angular scans suggest that defect complexes are associated with the substitutional dopants.

This work was performed at the Avionics Laboartory, Wright Patterson AFB, Ohio, under contract F33615-80-C-11J6.

Thursday Afternoon - 2:40 p.m.
D-3

COMPOSITIONAL AND GROWTH PROPERTIES OF THIN Cr-Si FILMS*

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The chemical and structural properties of thin chromium silicide layers produced by evaporation of Cr onto cleaned Si wafers and subsequently annealed have been investigated using Auger electron spectroscopy, x-ray photoelectron spectroscopy, scanning electron microscopy, and x-ray diffraction. Depth concentration profiles and quantitative compositional analysis of the silicide layers are presented as a function of annealing temperature and time for initial conditions that correspond to a finite source of Cr in contact with an infinite source of Si. Additionally, the effect of Si substrate cleanliness on the kinetics of silicide formation and growth is investigated. The final silicide under the conditions of the experiment is $CrSi_2$. Results for partially reacted layers are also presented. The interfaces between the silicide and Si, as well as the silicide and Cr, are quite broad, suggesting interface roughness or the presence of silicides with different stoichiometries. XPS core level shift and Auger parameter measurements are made at various depths. Cr 2p core-level shift measurements clearly indicate the presence of the silicide, a result corroborated by x-ray diffraction measurements. An activation energy and reaction rate for $CrSi_2$ formation is extracted from these measurements.

The rate of silicide formation of Si wafers that had a thin oxide on them is much slower. Initial results of attempts to extract activation energies and rate constants for this condition are presented. Additionally, the effect of the structure of the Si in contact with the Cr is investigated by sputter depositing an amorphous layer of Si onto Cr evaporated on a clean Si wafer. An initial comparison of the reaction kinetics at the two interfaces is presented. Finally, the compositional and chemical properties of cosputtered thin films of Cr and Si that are subsequently annealed are presented and compared to the above results.

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*Research supported by DOE.

Thursday Afternoon - 3:30 p.m.

D-4

The influence of low energy ion bombardment on the chemistry of the silicon dioxide surface, J. H. THOMAS III, RCA Laboratories, and S. HOFMANN, Max-Planck-Institut for Metallforschung, Stuttgart, West Germany - To determine the effects of sputtering on the surface chemistry of silicon dioxide, thermally grown silicon dioxide layers on silicon were bombarded with helium, argon and xenon ions at energies between 0.5 and 2.0 keV. X-ray photoelectron spectroscopy was used to probe the chemical changes induced by ion sputtering. Chemical shifts of the Si 2p and O 1s photoelectron peaks were not observed after ion bombardment. Therefore, the silicon dioxide surface is not chemically reduced or decomposed by ion bombardment. Small changes in surface chemistry are indicated by increases in the Si 2p and O 1s photoelectron peak width with increasing ion dose and ion energy. A steady state peak width was obtained at each ion energy for ion doses above $\sim 2 \times 10^{17}$ ions/cm^2. The steady state peak width depends on the ion species and ion energy and is adequately described by a simple model based on a uniform damaged region of the oxide roughly equal in depth to the ion range. The peak broadening is phenomonologically explained by Grunthaner's model[1] and is consistent with an increase in Si-O bond angle disorder within the ion penetration depth.

[1] F. J. Grunthaner, P. J. Grunthaner, R. P. Vasquez, B. F. Lewis, J. Maserjian and A. Madhukar, Phys. Rev. Lett. 43, 1683 (1979).

Thursday Afternoon - 3:50 p.m.
D-5

XPS STUDIES OF ION-INDUCED DAMAGE ON SEMICONDUCTOR AND INSULATOR SURFACES

by

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ABSTRACT

Inert-gas ion bombardment and plasma etching are important processes in the fabrication and micromachining of semiconductor devices. Sequential ion-etching and surface analysis is also widely employed as a means of device characterisation and investigation. In both applications, the incident ions produce topographical and compositional changes in the surface region of the material.

Changes in stoichiometry and oxidation state following ion-bombardment of a variety of insulator surfaces and passivation layers on semiconductors have been studied by x-ray photoelectron spectroscopy (XPS), as a function of ion dose, ion energy and ion angle of incidence. In most cases, stoichiometry changes caused by ion-induced dissociation of surface molecules are observed, generally resulting in a preferential loss of the anionic species. Such behaviour is observed for the carbonates, sulphates and nitrates of group I, II and IV metals. The extent of cation reduction following ion bombardment is found to be critically dependent on the composition and nature of the original surface. For example, Pb^{2+} appears stable to ion bombardment in most compounds, but is reduced to Pb^{o} in the monoxide. The extent of this reduction is markedly enhanced when the oxide is incorporated in a silica matrix. Ion bombardment of TeO_2 and anodically grown oxide films on telluride surfaces results in the reduction of Te^{4+} to Te^{o}, whereas $CdTeO_3$ forms a characteristic telluride species (Te^{2-}) following ion bombardment.

The results are discussed in relation to the characterisation and analysis of technological surfaces, with particular reference to the identification of certain surface species via their ion-induced decomposition products.

Thursday Afternoon - 4:10 p.m.

D-6

Use of Surface Behavior Diagrams to Compare Anodic and Photochemical Oxides of $Hg_{0.8}Cd_{0.2}Te$

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ABSTRACT

Surface behavior diagrams have been used to plot the chemical composition of anodized and photochemically oxidized $Hg_{0.8}Cd_{0.2}Te$ as a function of depth. The data were obtained by X-ray photoelectron spectroscopy in combination with ion sputtering. These diagrams are similar to phase diagrams except that the equilibrium condition has been relaxed to allow the surface composition to be represented as a function of chemical reactions or depth into the sample. The analysis indicates that both oxides have a thin cation-rich surface layer (a Cd-rich layer for the anodic oxide and a Hg-rich layer for the photochemical oxide) on top of a bulk oxide. The approximate bulk oxide compositions indicated are: 61% $(Cd_{0.73}Hg_{0.27})TeO_3$ and 39% $(Cd_{0.73}Hg_{0.27})Te_2O_5$ for the anodic oxide and 100% $(Cd_{0.79}Hg_{0.21})Te_2O_5$ for the photochemical oxide. The composition of the photochemically oxidized sample in the interfacial region is a linear combination of the composition of the bulk oxide and the bulk semiconductor, indicating a stoichiometric interface. For anodized sample, however, the Hg concentration in the interfacial region is lower than expected. This deviation is interpreted as a Hg depletion in the interfacial region of the semiconductor caused by a field-assisted dissolution during the anodization process. We use these examples to show that the surface behavior diagram is a powerful analytical tool: it allows small changes in the surface composition to be readily detected and permits evolution of the composition to be traced as a function of depth, growth conditions, or other parameters and compared with that expected from model systems.

Thursday Afternoon - 4:30 p.m.
D-7

QUANTITATIVE ANALYSIS IN CHEMICALLY DISSIMILAR MATRICES BY SECONDARY ION MASS SPECTROMETRY

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ABSTRACT

The ability of secondary ion mass spectrometry (SIMS) to achieve detection limits of a ppm or less for many of the elements throughout the periodic table makes SIMS one of the most sensitive characterization techniques available to the materials analyst. However, due to the wide variability of secondary ion yields, the ability to perform quantitative analysis has long been hampered by the requirement of a standard for the matrix being examined. We report here for the first time the ability to obtain quantitative measurements for the B concentration in a series of thermally grown PtSi layers on Si without the use of a separate standard for each matrix. This technique should be generally applicable to any material containing two dissimilar, coupled matrices. This study describes in detail the method used for quantization, its applications and limitations.

Thursday Afternoon - 4:50 p.m.

THERMAL DESORPTION OF CO, O_2 AND CH_4 MOLECULES FROM Ni(111) SINGLE CRYSTALS

By

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ABSTRACT

The thermal desorption of CO, O_2 and CH_4 molecules from a Ni(111) single crystal surface has been studied by low energy electron diffraction (LEED), Auger Electron Spectroscopy (AES) and Mass Spectroscopy. The crystal surface was exposed to the above molecules at pressures from 10^{-9} to 10^{-5} torr. Surface structures and bonding were studied by the above techniques. The absolute coverage of the molecules on the surface was measured and activation energies for thermal desorption were measured for each case.

Friday Morning - 8:30 a.m.
E-1

ASSESSMENT OF DIFFERENT MODELS FOR QUANTITATIVE AUGER ANALYSIS IN APPLIED SURFACE STUDIES

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The most common quantitative and semiquantitative models of Auger analytic spectroscopy in applied surface studies are considered [1,2]. The influence of the approximations made in each model on the achievable accuracy is discussed.

The suitability of the various models for different analysis problems is investigated experimentally. Elemental sensitivity factors are determined for seven elements included in the investigated samples using a VG ESCA3 MkII hemispherical analyzer. Analysis of several alloys in three binary systems (Cu-Al, Cr-Al and Ag-Cu) is presented. Multicomponent samples of AISI 304 stainless steels are also analyzed. Interpretation of the measurements is based on models including various stages of matrix corrections.

The obtained elemental sensitivity factors are found to agree well with corresponding factors for a cylindrical mirror analyzer [3]. Application of three different matrix corrections (mean free path, backscattering and atomic density) is found to give results in good agreement with the bulk analysis for samples fractured in the instrument vacuum, hence presumably representing the bulk composition. On the other hand, application of a mean free path correction only results in values possibly even worse than the uncorrected analysis. This is because of the opposite effects of the different corrections.

The large concentration changes often introduced by sputtering are probably the greatest problem in the quantitative analysis of alloy samples, particularly in depth profiling studies.

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[2] H.J. Mathieu and D. Landolt, Surf. Interface Anal. 3 (1981) 153

[3] L.E. Davis, N.C. MacDonald, P.W. Palmberg, G.E. Riach, and R.E. Weber, Handbook of Auger Electron Spectroscopy (Physical Electronics Industries, Inc., Minnesota, 1976)

Friday Morning - 8:50 a.m.
E-2

THE AUGER PARAMETER APPLIED TO SOLID AND GASEOUS COMPOUNDS

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The Auger Parameter is defined as the kinetic energy of the electrons in the sharpest Auger line minus the kinetic energy of the electrons in the most intense photoelectron line. Since it is a difference quantity, it is not subject to problems involving charge referencing or reference levels, and the quantity is a characteristic of a chemical and physical state.

The magnitude of the Auger Parameter is, to a first approximation, a measure of the polarizability of the atomic environment. Examination of photoelectron and Auger energies of many compounds of silicon, phosphorus, sulfur, chlorine, germanium, and bromine disclose that the Parameters for gaseous compounds never exceed those of any of the insulating solids, indicating that the solid state always provides greater screening of the final ions in the photoelectric and Auger transitions. Trends with structure are understandable on the basis of polarizability of attached atoms or groups of atoms.

Data now available on CS_2, SO_2, and SF_6 in both gas and solid phase demonstrate that neighboring molecules in the condensed state can contribute several electron volts of screening energy to the Auger electrons in the KLL transition.

Friday Morning - 9:10 a.m.

LOCALIZED MEASUREMENT OF D_2O CONCENTRATIONS USING NUCLEAR REACTION ANALYSIS WITH APPLICATION TO MOISTURE DIFFUSION IN COMPOSITES

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Moisture absorption by composite materials and epoxy adhesives has long been recognized as a cause of mechanical property degradation in these materials at elevated temperature. Until very recently the moisture absorption and transport properties of these materials were used to deduce moisture profiles from the measurement of bulk characteristics such as weight gain. These methods have limited applicability so we have pursued the development of a technique based on nuclear reaction analysis for measuring localized moisture content in the near surface region of resin based materials. The technique makes use of the $D(^3He,p)^4He$ reaction to measure the D_2O concentration profiles in specimens that have been conditioned in an environment containing heavy water vapor to simulate actual environmental exposure. A helium-3 ion beam from a Van de Graaff accelerator interacts with the deuterium within the first few microns of the surface and the interaction gives rise to prompt proton emission which is proportional to the D_2O concentration. Specimens are mounted on a micrometer driven device that translates it through the finely collimated ^3He ion beam to obtain the D_2O distribution along the specimen.

A full description of the methodology and the calibration of the system is presented. The applications to be discussed include the evaluation of moisture diffusivity of isothermally conditioned adhesive-bonded composites and moisture profile measurments to determine the effect on composites exposed to real-time environmental conditioning.

Friday Morning - 9:30 a.m.

Auger Electron Spectroscopy Studies in Y-Fe-O and La-O System

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A distinct spectral difference between Y in the metallic environment within an Y-Fe film and in the oxidized surface were found to be characteristically reflected in the low energy transition. The main peak intensity changes of the M2 M4 N2,3(3p3d4p), 122eV, transition and M5 N2,3 N4,5(3d4p4d), 126eV, transition of Y are due to the cross transitions (M5 L1 L2,3 and M2 M4 L1). The energy shift of the Y Auger spectra in the oxides arises from the normal chemical shift. The high energy transitions of Fe in the oxidized layer were not affected by oxidation, but the low energy M2 VV transition in the Y-Fe-O system showed a decreased intensity and a L1 L2 M1 transition subpeak developed. The amorphous Y-Fe films did not display a compositional influence on the Auger spectra over the range $YFe_{1.5}$-YFe_4.

In the La-O system, there are no significant chemical effects in the low energy transitions. However, in the high energy transitions a small subpeak at 636eV develops for La in the completely oxidized state.

Friday Morning - 9:50 a.m.

QUANTITATIVE AES ANALYSIS OF Fe-Cr AND Fe-Ni-Cr ALLOYS

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Auger Electron Spectroscopy (AES) is in general considered a semi-quantitative surface analysis technique, providing information on the surface elemental composition with an associated absolute error of up to 30%. This error may arise from the use of published rather than instrument-determined sensitivities, from matrix effects, from Ar^+ sputtering phenomena and more specific error sources such as peak overlap and peak-shape variations. This work aims at investigating the effects of these sources of error in the analyses of Fe-Cr and Fe-Ni-Cr alloys. Such alloys are widely used in industrial and commercial applications, mainly due to their corrosion resistance, and their surface elemental composition is a vital factor in adequate performance.

In general all Fe-Cr and Fe-Ni-Cr alloys have oxidised surfaces and the presence of oxygen provides several problems for quantitative analysis. The O peak in Auger spectra falls close to that for Cr, resulting in peak interference.

The conventional measure of elemental composition, the peak-to-peak height (PPH), becomes significantly into error for Cr as a result of this interference, and the alternative measurement of background to negative peak deflection (B-PH) is much less affected by O content, with quantitative analysis being possible for large O contents. A further problem concerning oxidized alloys is the change in peak shape which occurs for the major Fe peak (703eV).

Conclusions:

The use of instrument-determined sensitivities, and conventional AES derivative mode operation, provides quantitative analysis of surface elemental composition over a wide range of Fe-Cr and Fe-Ni-Cr alloys. Sputtering effects were pronounced in one Fe-Ni-Cr alloy but in general could be ignored. Peak interference between O and Cr was overcome by use of alternative measures of Cr peak height with accurate analysis being possible up to virtually full oxidation. Peak shape changes in Fe with oxidation were seen but use of the Fe650 peak appeared to significantly reduce these effects, and use of this peak is recommended.

Friday Morning - 10:10 a.m.

DURABILITY OF SOLID FLUOROPOLYMER THIN FILMS ON METAL SURFACES FOR SILICONE OIL CREEP

by

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ABSTRACT

The lifetimes of two fluoropolymer films intended to prevent silicone oil spread are determined using thermal aging as an accelerating mechanism and Auger spectroscopy to detect silicone oil.

Both the fluoropolymer barrier coating solutions contain the same solute, a 0.1 percent by weight concentration of the polymer poly-1H, 1H-pentadecafluorooctyl methacrylate (PFOMA). The solvents are different with one being Freon TF and the other a mixture of 10 percent Freon TF and 90 percent perfluoro-2-butyltetrahydrofuran. At this concentration, the surface films are about 150 A to 250 A thick and are solid polymer films, for the solvents being volatile, evaporate, leaving the polymer coating on the surface.

In the experiment, a 5 μl drop of 50 cs (viscosity) silicone oil is placed on a polished Cu, Pd-Ag 40 wt.% or Au coupon on one side of a 2 mm width strip of the fluoropolymer film. After thermal aging, the coupon is investigated for silicone oil on the other side of the fluoropolymer strip. Device design standards require a 40-year lifetime at 70°C and below. It was determined from an extrapolated Arrhenius relationship that PFOMA in Freon TF (PFOMA-1) has a 40-year life at temperatures up to 50°C while PFOMA in the other solvent (PFOMA-2) has a lifetime exceeding 40 years at 70°C.

Though the polymer in the two solutions is the same, the solvent in PFOMA-2 leaves a relatively more microscopically uniform spatial distribution of the polymer film compared to the solvent in PFOMA-1. This is considered to be the reason for the significant improvement in lifetime for PFOMA-2.

Friday Morning - 10:50 a.m.

ABSTRACT

SURFACE TECHNIQUES APPLIED TO THE ANALYSIS OF DEFECTS ON EXTRUDED ALUMINUM ALLOYS

by

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Various surface techniques are used to analyze "pickup" (a surface defect) on extruded 7275, 2024 and 2014 aluminum alloys. SEM revealed a typically rough, tear-drop shape morphology for all pickup areas with only a size difference between the alloys (confirming plant observations). Conventional AES was used to determine the surface composition of the extrusion samples, but due to the limitations of this technique, no useful analyses resulted. SAM was then utilized to resolve the compositional variation from the smooth to the defective areas of the specimens. SAM provides high resolution surface scans, depth profiles and elemental maps. Interpretation of our results indicates that:

- pickup is caused by the aluminum metal and oxides adhering to the die bearing surface rather than iron oxide from the die.
- a change in the relative amounts of magnesium to aluminum on the metal surface was seen when pickup formed.
- extruding under nitrogen influences the amount of oxides formed at the die/aluminum interface.
- pickup formation on the 2024 alloy was accompanied by an increase of Si on the extrusion surface.

Friday Morning - 11:10 a.m.

ABSTRACT

Study of Chromic-Acid-Anodized Titanium Oxide Using Auger Line Shapes

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The Ti $L_3M_{23}V$ Auger line shape has been compared for standards of TiO_2 (anatase and rutile), Ti_2O_3, and TiO and for an oxide layer on Ti-6Al-4V alloy coupons prepared by chromic acid anodization (CAA). Line shape and peak-to-peak height measurements of the standards indicate that line shapes are a sensitive indicator of the stoichiometry of the oxides. The CAA samples were examined as prepared, after heating in situ, and after immersion in hot water. Comparison of the line shapes for the standards and the fresh CAA oxide showed that the oxide was not strictly TiO_2. After heat treatments and water immersion the line shape indicated that the oxide had a stoichiometry which varied with depth in a manner that cannot be explained by sputtering-induced reduction. It is envisioned that the line shape can indicate the oxide stoichiometry when the peak-to-peak ratio method cannot be used.

Friday Morning - 11:30 a.m.
E-9

AN RBS STUDY OF INHIBITOR REACTIONS WITH THE SURFACE OF ALUMINUM IN AQUEOUS ELECTROLYTES

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Rutherford backscattering spectroscopy (RBS) has been utilized to determine qualitatively the extent of surface aluminum hydrate film growth for high purity aluminum in contact with water containing various inorganic inhibitors at 100°C. The anions nitrate, sulfate, phosphate, silicate, borate, molybdate and tungstate were studied at concentration levels typical of those used in automotive coolant formulations. The extent of film growth, and of inhibitor uptake by the surface was assessed immediately in each case from the RBS spectra. The inhibitors studied suggest three modes of behavior. In the first mode typical of silicate and borate, the film growth is virtually stifled by the interaction of the surface with the inhibitor. In a second mode, typified by the oxidizing inhibitors nitrate and sulfate, the growth of an identifiable hydrated oxide layer continues at lower levels than those observed with pure water. In phosphate, molybdate and tungstate based inhibitors, the surface film incorporates the inhibitor anion and continues to growth with level of exposure. Synergistic effects of mixed inhibitors, as well as apparent modes of inhibition in commercial formulations are readily observed using RBS.

Friday Morning - 11:50 a.m.
E-10

Application of Low Damage SIMS/AES/XPS of the Ba-O-W System to Cathode Characterization

by

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and

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The activation of an impregnated dispenser cathode was characterized by SIMS, AES, and XPS on the basis of multitechnique results from prepared thin film model standards of the Ba-O-W system. Proper SIMS characterization required low damage conditions (500eV Ar^+, ~5nA/cm^2) and ultrahigh vacuum (better than 2×10^{-10} Torr during analysis). Individual low damage SIMS yields from different model structures varied by several orders of magnitude; and consequently, the identification of the activated cathode structure from model compound yields was straightforward. While x-ray induced AES of cathodes requires curve stripping to identify the activation transition with temperature, the low damage SIMS yields showed obvious but continuous transitions through the activation temperatures. Some discussion of the pitfalls of the low damage SIMS technique will also be presented.

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Friday Morning - 12:10 p.m.

THE USE OF THE VAPOR COLLECT METHOD AND LINE OF SIGHT RESIDUAL GAS ANALYSIS TO DETERMINE EVAPORATION RATES OF Ba FROM IMPREGNATED TUNGSTEN DISPENSER CATHODES*

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Impregnated tungsten dispenser cathodes having molar ratios of BaO, CaO, and Al_2O_3 of 5:3:2 and 4:1:1 were studied by the vapor collect method and line-of-sight residual gas analysis. The evaporation rates, vapor pressures, and heats of evaporation were determined from the data collected by the vapor collect method and line-of-sight residual gas analysis. All measurements were performed in the ultra high vacuum system of a PHI model 545A scanning Auger microprobe. The vapor collect method was based on the work of Verhoeven and vanDoveren[1] except that the ratio of adsorbate to substrate Auger peak-to-peak heights was plotted versus time. Break points in these plots characterized the collection of one monolayer of adsorbate. Geometrical corrections then allowed the calculation of the evaporation rates. For line-of-sight residual gas analysis, an Extranuclear Quadrupole was used, and attempts made to calibrate the evaporation rate will be discussed. Rates were determined at cathode temperatures of 1050, 1100, and 1150°C.

[1] J. A. Verhoeven and H. vanDoveren, Microchimica Acta [Wein] 1, 331 (1979).

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

Friday Morning - 12:30 p.m.

THE ANALYSIS OF PROFILE STRATA FOR SURFACE TEXTURE SPECIFICATION

By

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Surfaces are created by a series of one or more processes and each process in the series leaves its signature on the surface. For many common manufacturing processes the height distribution of a surface profile is known to be essentially Gaussian, and if such a distribution is plotted on normal probability coordinates the resulting curve closely approximates a straight line whose slope is proportional to the rms average of the height distribution.

If the cumulative height distribution of a surface profile obtained from a surface created by a series of processes is plotted on normal probability coordinates, it can generally be represented by a series of straight lines each of which corresponds to one of the various processes involved in creating the surface. These straight lines can be used to define strata within the surface profile and the intersections among the lines are the heights at which the transitions between the strata occur. Within any of the strata, the surface texture is very different from adjacent strata, or else the existence of the strata would not be apparent from the plotted curve.

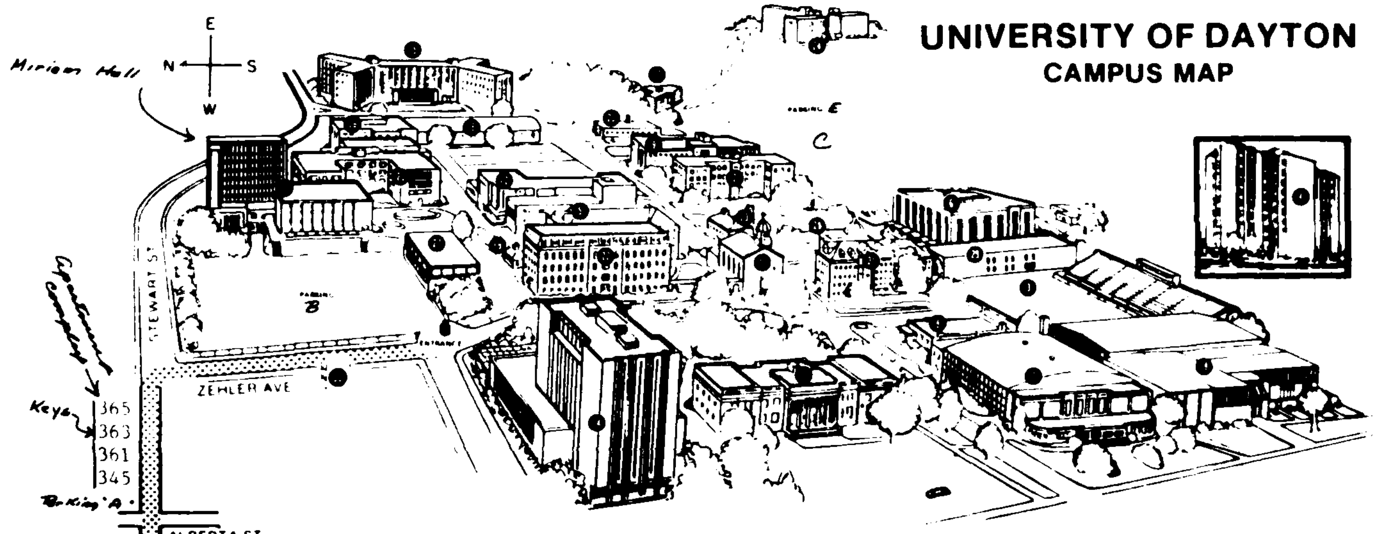
The analysis of the surface texture within individual strata of a profile provides a much clearer picture of the functionality of a surface than does the analysis of a complete profile. For any particular application of a surface it is the active stratum within the profile which determines the suitability of the surface for its intended function, and analysis of the complete profile thus has a much lower "signal-to-noise-ratio" than does analysis of the active stratum alone.

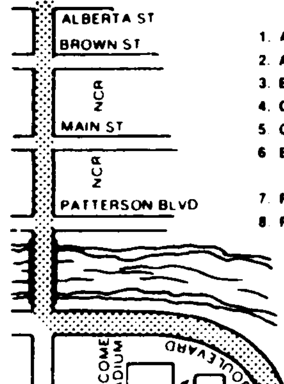
The usefulness of the cumulative height distribution function of a surface profile for the specification of engineering surfaces was recognized many years ago by Abbott and Firestone (1). Their bearing ratio curve is the height distribution plotted on linear scales. The advantages of using normal probability coordinates was first suggested by Williamson (2, 3), but it has only been recently with the advent of microprocessor based surface texture analysis equipment that the practical application of these ideas to the examination of surfaces has become feasible outside of the research laboratory. Since instruments capable of applying these ideas to production inspection on the factory floor are now available, it behooves us to reexamine the usefulness of these concepts since they can provide us with better means of specifying surface texture.

1) E.J.Abbott & F.A.Firestone, Specifying Surface Quality, Mech.Eng. 55, 569 (1933).
2) J.B.P.Williamson, Microtopography of Surfaces, Proc.Inst. Mech.Eng. 182 (3K), 21 (1967-68).
3) J.B.P.Williamson, J.Pullen, and R.T.Hunt, The Shape of Solid Surfaces, in F.F.Ling, Editor, Surface Mechanics, ASME, 1969, p. 24.

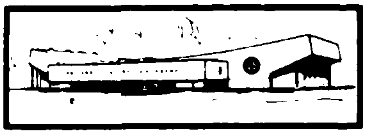
Friday Morning - 12:50 p.m.

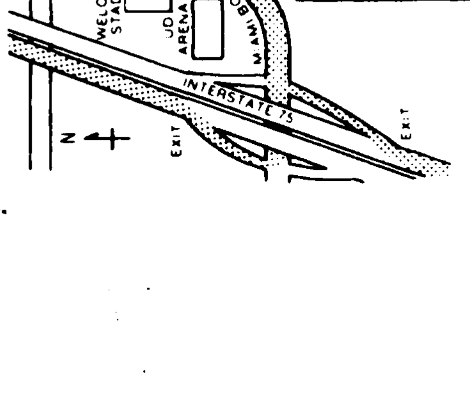
UNIVERSITY OF DAYTON
CAMPUS MAP





1. Albert Emanuel Hall (1928)
2. Alumni Hall (1924)
3. Baujan Field (1925)
4. Campus South (1969)
5. Chaminade Hall (1904)
6. Eugene F. Kettering Engineering & Research Laboratories (1970)
7. Fieldhouse (1950)
8. Reichard Hall (1962)
9. Founders Hall (1954)
10. Gosiger Health Center (1967)
11. Immaculate Conception Chapel (1869)
12. John F. Kennedy Memorial Union (1964)
13. Liberty Hall (1866)
14. Library (1971)
15. Marycrest Hall (1963-64)
16. Mechanical Engineering Bldg. (1948)
17. Miriam Hall (1966)
18. Physical Activities Center (1975)
19. Post Office (1903)
20. O'Reilly Hall (1952) R.O.T.C. Bldg.
21. St. Joseph's Hall (1884)
22. St. Mary's Hall (1870)
23. Sherman Hall of Science (1960)
24. Stuart Hall (1963-64)
25. U.D. Arena (1969)
26. Wohlleben Hall (1958)
27. Rike Center for Fine Arts (1978)
28. Zehler Hall (1865)
29. Music/Theatre Building (1978)
30. Law Clinic (1977)





DIRECTIONS TO THE UNIVERSITY OF DAYTON

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