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WORKSHOP ON
"SURFACE SCIENCE AND CATALYSIS"
(4 - 8 May 1987)

SURFACE ANALYTICAL TECHNIQUES II

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Surface Analytical Techniques II

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Outline

1) Adsorption and desorption phenomena

   Thermal Desorption spectroscopy

   Molecular Beam Techniques

2) Examples of applications of XPS and Auger spectroscopy for studying simple surface reactions

   Synchrotron radiation: present and future applications in Surface Science

MECHANISMS OF THE CARBON MONOXIDE OXIDATION AND NITRIC OXIDE REDUCTION REACTIONS OVER SINGLE CRYSTAL AND SUPPORTED RHODIUM CATALYSTS: HIGH PRESSURE RATES EXPLAINED USING ULTRAHIGH VACUUM SURFACE SCIENCE

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The demonstration that surface parameters obtained in UHV experiments are applicable to high pressure catalytic reactions has long been a goal of surface science studies. We will attempt to show directly in these lectures that, for an important class of reactions, the strongly-bound species present under the conditions of UHV studies are the same surface species reacting at high pressures. We have chosen two test reactions, CO oxidation ($2CO + O_2 \rightarrow 2CO_2$) and NO reduction ($2CO + 2NO \rightarrow 2CO_2 + N_2$) over rhodium, which are important in automotive exhaust catalysis. We will also discuss the water formation reaction ($2H_2 + 2O_2 \rightarrow H_2O$) on Rh when appropriate. An outline of the content of the lectures is as follows:

I. Motivation

II. Approach
  A. Measure High Pressure Reaction Rates
  B. Measure appropriate surface properties in ultrahigh vacuum
  C. Develop kinetic model based on UHV results
  D. Compare high pressure results with predictions based on UHV work

III. Equipment and Methods
  A. High Pressure Reaction Measurements
  B. Ultrahigh Vacuum Measurements
    a. Temperature Programmed Desorption (TPD)
    b. High resolution ($\Delta E \sim 40\ cm^{-1}$) Electron Energy Loss Spectroscopy (EELS)
    c. Other surface science tools (XPS, UPS, ...)
  C. Example of determining an activation energy: NO(a) dissociation on Rh(111) or OH(a) formation on Rh(100)

IV. Carbon Monoxide Oxidation Reaction over Rhodium
  A. High pressure (1-100 Torr) kinetics over single crystal and supported Rh
  B. Low pressure (10^{-8}-10^{-4} Torr) kinetics over single crystal Rh
  C. UHV determination of activation energies of desorption and dissociation
  D. UHV determination of sticking coefficients for oxygen in the presence of CO(a)
  E. Derivation of a steady state reaction model based on UHV results
  F. Comparison of agreement between model results and reaction data
  G. What is the Rh surface like during CO oxidation?

V. Nitric Oxide Reduction Reaction over Rhodium
  A. High pressure (1-100 Torr) kinetics over single crystal and supported Rh
  B. Low pressure (10^{-8}-10^{-4} Torr) kinetics over single crystal Rh
  C. UHV determination of activation energies of desorption and dissociation
  D. Discussion of the steady state reaction model based on UHV results
  E. Comparison of agreement between model results and reaction data
  F. What is the Rh(111) surface like during the NO-CO reaction?
  G. How is the reaction different on supported Rh?

VI. The NO-CO-O_2 Reaction Over Rhodium
  A. High pressure (1-100 Torr) kinetics over single crystal and supported Rh
  B. Manifestations of NO inhibition of CO oxidation

VII. Conclusions

The similarity in the coverages of strongly-bound adsorbed species like NO and CO under UHV and high pressure conditions allows us to use UHV-obtained data to usefully explain high pressure kinetic data. We find that rate expressions based on UHV-determined intermediate steps using UHV-determined rate constants quantitatively predict the rates at high pressures for both the CO-O_2 and NO-CO reactions over single crystal Rh and supported Rh catalysts. Understanding the relative importance of intermediate steps in all of these reactions clarifies which steps need modification to improve overall reaction rates.

A good reference to begin with concerning these lectures is by S. H. Oh, G. B. Fisher, J. E. Carpenter, and D. W. Goodman in the Journal of Catalysis, Vol. 100 (1986), pp. 360-376.

LECTURE SUMMARY FOR ICTP WORKSHOP ON SURFACE SCIENCE AND CATALYSIS

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LECTURE 1. Introduction, The ELECTRONIC FACTOR in Catalysis

Definitions, The **electronic factor** and its dominance in chemistry.
The **electronic factor** and the **geometric factor** as historical concepts in catalysis.

The **electronic factor as a** local phenomenon: the importance of density of states at the Fermi level.

The **importance** of charge mobility in catalysis.

The **electronic factor** in alloy catalysis.

Lectures 2 and 3. The FISCHER-TROPSCH SYNTHESIS

The **Fischer-Tropsch synthesis**:-

$$nCO + (2n + 2)H_2 = C_nH_{2n+2} + nH_2O$$

The **Fischer-Tropsch synthesis** as a polymerisation process: variations in product selectivity and the Schultz-Flory alpha number, (1).

Suitable catalysts: iron, cobalt, nickel and ruthenium.

The mechanism of the reaction and the role of surface science in its elucidation:-

1) The molecular adsorption of carbon monoxide and hydrogen.
2) The **dissociation of carbon** monoxide: dissociation as an activated process, the role of promoters and poisons, (briefly).
3) The polymerisation steps, the mechanism of McCandlish, (2) and the possible role of metallocycles. A challenge for surface science.
4) Reaction rates on real and model catalysts: methanation studies of Goodman et al, (3).
5) Alcohol synthesis: CO insertion as a chain terminator.

The Fischer-Tropsch synthesis under industrial conditions:-

1) The role of oxides and other selectivity promoters.
2) The role of carbide and other bulk phases in catalyst deactivation.

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1) M. E. Dry, in 'CATALYSIS, Science and Technology,' Volume 1, Ed. J. R. Anderson and M. Boudart, Pub. Springer-Verlag, Berlin, 1981.
2) L. E. McCandlish, J. Catalysis, 83, (1983) 362.
3) see e. g. D. W. Goodman, Acc. Chem. Res. 17, (1984) 194.

A DESCRIPTION OF THE HIGH-PRESSURE AMMONIA SYNTHESIS REACTION BASED ON SURFACE SCIENCE

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ABSTRACT

A kinetic description is given of the high-pressure ammonia synthesis reaction over a Fe based catalyst. The description is based on a statistical mechanical treatment of the competitive adsorption of the reactants, intermediates, and product on identical surface sites. The kinetic expression with parameters taken from ultra-high vacuum for the reactants on single-crystal surfaces is able to describe the observed ammonia kinetics for an industrial catalyst over a very broad range of conditions (1-300 atm, 375-500°C). This provides a direct verification that the 'pressure gap' between surface science and heterogeneous catalysis can be bridged. Further, the present description gives a detailed picture of the working catalyst. Variations in surface coverages down through the reactor bed and the origin of the activation enthalpy of the overall reaction are discussed. We also show that the microscopic picture of poisoning and promotion whereby the effect of surface impurities is to stabilize or destabilize the adsorbing molecules can account quantitatively for the observed promotion and poisoning of the high pressure ammonia synthesis by potassium and water, respectively.

SURFACE ANALYTICAL TECHNIQUES I

I. Introduction to fundamentals of surface analysis

A. A brief review of interactions of photons and charged particles with the surface region of materials

B. Surface analytical techniques developed using:

1. Photons
2. Electrons
3. Atoms and molecules
4. Thermal and other means of excitation

C. Basic concepts of energy and momentum analyzers for charged particles and photons

D. Sample preparation

II. Surface analytical techniques commonly used for the study of catalysis and related phenomena

A. Angular resolved photoelectron spectroscopy (ARPES)

1. Instrumentation
2. Application of ARPES for determining the surface and bulk valence band electronic structure
3. Polarization selection rules

B. Inverse photoemission spectroscopy (IPS), a complementary technique to ARPES

1. Basic concepts and instrumentation
2. Application to determination of unoccupied states

C. X-ray photoelectron spectroscopy (XPS)

1. Core level photoemission and chemical shifts
2. Auger transitions
3. Quantitative analysis

D. (Surface) Extended X-ray absorption fine structure ((S)EXAFS) and photoelectron diffraction (PD)

1. Basic concepts
2. Local order (< 6 A)

E. Low energy electron spectroscopies (< 200 eV)

1. Electron energy loss spectroscopy (EELS)

   a. Instrumentation
   b. Energy loss mechanisms and dipole selection rules
   c. Application to chemical analysis of adsorbate species

2. Low energy electron diffraction (LEED), and surface symmetry in reciprocal space

F. High energy electron spectroscopies (> 1000 eV)

1. Auger electron spectroscopy (AES)

2. Scanning electron microprobe (SEM)

   a. Instrumentation
   b. Quantitative analysis
   c. Depth profiles
   d. Small spot size analysis

G. Secondary ion mass spectroscopy (SIMS), and elemental depth profile analysis

III. Closing remarks

REFERENCES ON SURFACE ANALYTICAL TECHNIQUES

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5. E.W. Plummer, W. Eberhardt, Angle-Resolved Photoemission as a Tool for Study of Surfaces, in I. Prigoyine and S.A. Rice (Eds.), Advances in Chemical Physics, Wiley-Interscience, New York (1982), p. 533.
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WORKSHOP ON "SURFACE SCIENCE AND CATALYSIS" G. Pirug

ALKALI METAL ADDITIVES

OUTLINE

I. Introduction

- Importance of alkali metals as coatings for electron emitters or as promotors in heterogeneous catalysis

- fundamental interest in the adsorption of alkali metals as 'simple' metals in basic research

II. Adsorption of alkali metals on transition metal substrates

- experimental details of alkali metal deposition

- characterization of the alkali metal layer

  electronic structure: work function change, electron binding energies of core levels, comparison of submonolayer coverages and massive layers (plasmon excitation, electron binding energy shifts, adsorption and sublimation energy)

  geometric structure: repulsive interaction and long range order, commensurable and incommensurable surface structure, compression structures and rotational epitaxy, phase diagrams

III. Influence of the alkali metal additives on the coadsorption of other gases

- CO coadsorption as a model system

  influence of the alkali metal on: the electron core and valence level binding energy (XPS, UPS, IPS, MDQS), C-O stretch frequency and vibrational properties (EELS, IRS), adsorption kinetics and adsorption energy (TDS), dissociation probability, molecular orientation and bond lengths (XPD, ESDIAD, NEXAFS, PARUPS), work function changes (ΔΦ).

- physical picture of the CO-alkali metal interaction

  long range versus short range interaction, atomistic models e.g., charge transfer, hybridization, electrostatic interaction.

- special aspects on the coadsorption of other gases: NO, N_2, O_2, H_2O, hydrocarbon.

- comparison of submonolayer and massive alkali metal layer influences on the surface reactivity.

IV. Influence of the alkali metal on surface reactions

UHV model experiments related to the Fischer/Tropsch and Ammonia Synthesis.

V. Outlook

Leiden, December 1986

Catalysis, and its relation to the surface chemistry of metals.

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1st hour - Definitions - rate, catalyst, support, promoter;
which reactions on metals are important in practice;
characterization of clean metal surfaces (remarks on)
i) powders, ii) flat surfaces

Characterization of catalytic intermediates
i) physical methods (just a list ...) and their pitfalls
(example - a study on hydrocarbons by IR)
ii) indirect use of physical methdos
(example - ethylene oxidation)
iii) chemical methods (just a list)

2nd hour Characterization of catalytic intermediates by chemical methods
Isotopes: ^{13}C labelling and D_2-exchange reactions of hydrocarbons
Archetype molecules: neohexane, methylcyclopentane, substituted pentanes - for a use in hydrogenolysis, isomerization and dehydrocyclization reactions.
Transient state techniques
Pulse techniques
Displacement reactions

K. Christmann :

Hydrogenation and Hydrogenolysis: Model Studies

1. Introduction

- Definition of hydrogenation/dehydrogenation and hydrogenolysis reactions.
- Listing of principle reaction types.
- Some important hydrogenation/dehydrogenation and hydrolysis reactions in practical chemistry (hydrocracking, methanation, hydrogenation of oils, ammonia synthesis, etc.).
- The concept of heterogeneous catalysis (activity, selectivity, deactivation, regeneration of catalyst materials).
- The reaction mechanisms, elementary steps (Eley-Rideal, Langmuir-Hinshelwood).
- The need for model studies in order to elucidate ensemble effects, ligand effects, spill-over effects.
- Brief description of spectroscopic methods to monitor hydrogenation and hydrogenolysis reactions and to detect surface intermediates (LEED, UPS, TDS, EELS).

2. The adsorption of hydrogen as an elementary step

- Sticking and dissociation of H_2, binding energies, electronic states, geometrical requirements , long-range order effects (lateral interactions).
- Some selected typical results for transition metal surfaces.

3. The coadsorption of hydrogen and other molecules on single crystal surfaces

- General principles of coadsorption (competitive, cooperative adsorption).
- Hydrogen and carbon monoxide.
- Hydrogen and hydrocarbons.
- Hydrogen and nitrogen.

4. The role of catalyst additives

- H-coadsorption on promoted surfaces
- H-coadsorption on bimetallic surfaces (ensemble -, ligand - and spill-over effects)

5. Conclusions:

What can we learn from model studies about the mechanism of hydrogenation/dehydrogenation and hydrolysis reactions?
How can we improve activity and selectivity?

LECTURES FOR TRIESTE : DAVID KING, LIVERPOOL.

Lecture 1 :
Classification of Chemisorption structures at metal surfaces:

In this lecture, I plan to talk about the electronic and crystallographic structure, first of all of Clean Single Crystal Metal Surfaces, and secondly, of adsorbate structures on these surfaces. The classification would be in terms of chequerboard structures, in which substrate atoms do not change their symmetry; displacive structures, in which substrate atoms are displaced by a small amount compared with the unit cell dimensions, induced by chemisorption; and reconstructed structures, in which there is a gross reconstruction of the substrate induced by adsorption. There will be some discussion on order/disorder phenomena within these three categories.

Lecture 2 :
Kinetic and Dynamic Processes at Metal Surfaces:

In this lecture, I plan to present an analysis of the current understanding of Adsorption, desorption and diffusion kinetics for chemisorbed species on metal surfaces. An attempt will be made to relate these processes to their surface structure within the chemisorbed overlayer.

Lecture 3 :

A continuation of lecture 2, developing the lecture to include a discussion of the influence of surface crystallography on kinetic processes; and developing the discussions to include recent work from reaction dynamics, including state-selected molecular beam studies. There will be a few examples chosen of catalytic processes at metal single crystal surfaces to illustrate the importance of surface crystallography in relation to catalysis involving simple molecules.

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DAK/JE
12th January 1987.

CHARACTERIZATION OF SUPPORTED CATALYSTS BY SOLID STATE NMR

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I. Introduction: Some relevant spectroscopic considerations

Transient techniques, relaxation processes, dipolar interaction, chemical shift anisotropy, quadrupolar interaction, magic angle spinning, cross-polarization. 'Transient Techniques in NMR of Solids', by G. C. Gerstein and C. R. Dybowski.

II. Solid-state NMR of zeolites.

^{29}Si and ^{27}Al NMR, precursors in zeolite synthesis, composition of the aluminosilicate framework, Si-Al ordering, interaction with adsorbed species. J. M. Thomas and J. Klinowski, Adv. Catal., <u>33</u>, 199 (1985).

III. Structure of aluminas. C. S. John et al. Applied Catal., <u>6</u>, 341 (1983).

IV. Characterization of silica-alumina gels.

Homogeneity of Si-Al interaction in cracking catalysts, effect of silica on alumina porosity and acidity.

V. Surface phases of Ni-Mo/Al_2O_3 hydrodesulfurization catalysts.

^{95}Mo, ^{27}Al and ^{31}P NMR, deactivating surface phases. M. McMillan et al. J. Catal., <u>97</u> 243 (1986).

VI. CO and ethylene adsorption on supported metal particles.

^{195}Pt and ^{13}C NMR, bonding of CO to group VIII metals, structure of adsorbed C_2 hydrocarbons, observation of chemical reaction and reaction products. P.-K. Wang et al., Science, <u>234</u>, 35 (1986).

DYNAMICS OF CHEMISORPTION AND SURFACE REACTION: NON-EQUILIBRIM ENERGY DISTRIBUTION IN REACTANTS AND PRODUCTS

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I. Introduction

Nozzle molecular beams, laser fluorescence, infrared emission, Fourier transform time resolved spectroscopy.

II. Effects of different kinds of energy on reactivity of molecules.

Translational energy effect on chemisorption of O_2, N_2, CO, CH_4 and higher hydrocarbons on transition metals, rotational and vibrational energy effects on CH_4 chemisorption on Ni and Rh, vibrational energy effect on cyclopropane reaction on mica, G. Prada-Silva et al., Surface Sci., 83, 453 (1979), D. J. Auerbach et al., J. Chem. Phys., 81, 2515 (1984), C. T. Retter et al., Phys. Rev. Lett., 54, 2716 (1985), S. L. Tang et al., J. Chem. Phys., 84, 6488 (1986).

III. CO oxidation on Pt and Pd

Translational energy of desorbed CO_2, angular distribution, rotational and vibrational energy excitation in desorbed CO_2, modeling of energy distribution. C. A. Becker et al., J. Chem. Phys., 67, 3394 (1977), J. Segner et al., Surface Sci., 138, 505 (1984), D. A. Mantell et al., Surface Sci., 172 (1986).

IV. Coverages effects on non-equilibrium energy distribution

Pulsed molecular beams, time resolved infrared emission, comparision of CO oxidation on Pt and Pd, D. A. Mantell et al., Chem. Phys. Lett., 102, 37 (1983).

Fourier transform infrared spectroscopy of adsorbates, M.A. Chesters

Outline

1. Molecular Vibrational Spectroscopy
    - simple harmonic oscillator
    - normal modes of vibration
    - characteristic group frequencies
2. Infrared Spectroscopy
    - excitation mechanism
    - selection rules
    - sensitivity - of routine analytical instrument
      - limitations on
3. Basic priniciples of FTIR spectroscopy
    - general advantages over dispersive spectroscopy
    - particular advantages important for surface studies
4. History of application of infrared spectroscopy to heterogeneous catalysis and surface studies.
5. Reflection-absorption infrared spectroscopy
    - metal surface selection rule
    - sample size and sensitivity
    - comparison with EELS
6. Examples of applications of infrared spectroscopy
    - ethylene on platinum surfaces - 20 years of vibrational spectroscopy
    - kinetic studies - methanol decomposition on Cu(111)
    - vibrational coupling and intermolecular interactions
    - lineshapes and vibrational relaxation
7. Future developments
    - improvements in sensitivity
    - improvements in spectral range
    - application of synchrotron radiation sources

TITLE: THE EFFECT OF ELECTRONEGATIVE ADATOMS ON THE CHEMISORPTIVE AND CATALYTIC BEHAVIOR OF METALS

M.Kiskinova

I.INTRODUCTION

Reasons for deactivation of heterogeneous catalysts and the relevance of the surface science studies to the fundamental understanding of the mechanism involved in the poisoning of metal surfaces.

II.ADSORPTION OF ELECTRONEGATIVE ELEMENTS (S,Cl,P,C,O,N,Se) ON SINGLE METAL SURFACES

Structures,coverages,segregation and bulk diffusion,charge transfer,bond lengths,binding energies and nature of the adatom-surface bonding as measured by means of LEED,AES,ESCA,TPD,work function.

III.EFFECT OF THE ELECTRONEGATIVE ADATOMS ON THE CHEMISORPTIVE PROPERTIES OF THE METAL SURFACES TOWARDS CO,H_2,NO.

Comparison between the strength of the effect and the nature of the poison.

IV.EFFECT OF ELECTRONEGATIVE ADATOMS ON THE SURFACE REACTIONS ON METAL SURFACES AND CORRELATION BETWEEN THE KINETIC DATA AND THE CHEMISORPTIVE PROPERTIES.

Examples : CO + H_2 on Ni(100)
CO + O_2 on Pt(110)
CO + NO on Pt(100)

V. POSSIBLE MECHANISMS OF THE POISONING EFFECT

Geometric,electronic (long-range and short range) effects. Influence of the local geometry,atomic size and Pauling electronegativity factors.

Time-Resolved Electron Energy Loss Spectroscopy

I) Introduction to high resolution electron energy loss spectroscopy [1]

a) Overview and history
  - -HREELS in relation to other electron spectroscopies
  - -basic principles of HREEL spectrometers [2]
  - -overview of various HREEL spectrometers from a historical perspective
  - -design limitations of electron monochromators, intensity vs. energy spread

b) Recent developments
  - -dispersion compensation, concept and implementation [3]
  - -dispersive detection [4]
  - -performance of TREEL spectrometers [5]
  - -time-resolved vs. low signal experiments
  - -the future? (new electron sources)

II) Techniques used to study surface dynamics

a) Overview [6]

b) Established techniques
  - -temperature programmed desorption (HCOOH/Cu(110) [7])
  - -molecular beam scattering [8] (NO/Pt(111))

c) Other new time-resolved techniques
  - -FTIR [9]
  - -Auger, UPS, XPS, laser induced desorption, helium scattering
  - -pico- and femto-second studies

III) TREELS results

a) CO/Cu(100) [10]
  - -adsorption kinetics (mobile precursor vs. Langmuir)
  - -calibration of HREELS intensities
  - -desorption kinetics
  - -combined TREELS/pulsed molecular beam measurements
  - -activation energies and pre-exponential factors (TST)

b) CO/Ni(111) [11]

c) CH_3OH/Ni(110) [12]
  - -combined TREELS/TPD measurements
  - NO/Rh(100) [13]

d) post-deadline results!?

References

[1] For an overview of HREELS see:
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  -W.H.Weinberg in "Methods of Experimental Physics", Vol.22, R.L.Park and M.G.Legally, eds., Acaedmic Press (1985)
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Analysis", H.Ibach, ed., Springer-Verlag (1977)
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