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SPECTROSCOPIC STUDIES OF THE ELECTRONIC STRUCTURE OF
METAL-SEMICONDUCTOR A. (U) IBM THOMAS J WATSON RESEARCH
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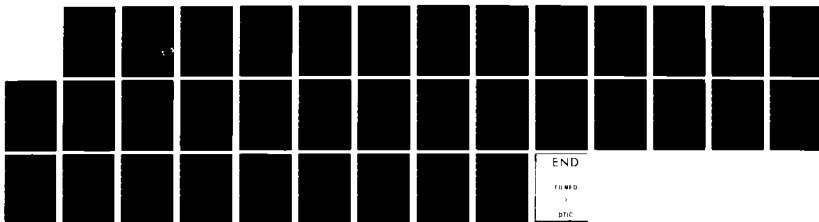
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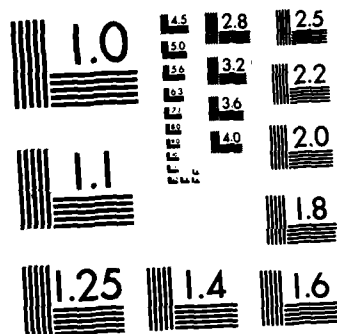
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etc. Extensive studies have been performed as a function of surface preparation (including laser annealing and molecular beam epitaxy) doping, thickness of adsorbed overlayers, etc. The principal techniques used have been photoelectron spectroscopy using synchrotron radiation and inverse photoemission. Our research (33 completed papers) has included studies of well-characterized surfaces of Si, Ge, diamond, GaAs, ZnO, oxidation of Si, W, and Ta surfaces, many rare earth and actinide compounds (involving uranium and thorium). Major achievements of our research program include world-leading contributions to (1) the electronic structure of Si surfaces and interfaces, (2) the development of inverse photoemission into a valuable surface technique which is complementary to photoelectron spectroscopy, and (3) the development and application of surface core-level spectroscopy for the study of complex interfaces.



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**SPECTROSCOPIC STUDIES OF THE ELECTRONIC STRUCTURE
OF
METAL-SEMICONDUCTOR AND VACUUM-SEMICONDUCTOR INTERFACES**

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P.O. Box 218

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December 31, 1982

Final Technical Report for the Period June 15, 1981-September 15, 1982

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I. HIGHLIGHTS

Significant and extensive progress in understanding the electronic structure and properties of semiconductor surfaces and semiconductor interfaces as well as selected compounds has been accomplished under Air Force Office of Scientific Research Contract F49620-81-C-0089 (June '81-Sept. '82). Techniques which have been used include photoelectron spectroscopy, inverse photoemission (bremsstrahlung spectroscopy), low energy electron diffraction (LEED), and Auger spectroscopy, as well as novel materials preparation methods such as laser annealing and molecular beam epitaxy. In summary, 33 research papers have been published under this contract (see Section III) on (a) the electronic structure of semiconductor surfaces and interfaces, (b) chemisorption and oxidation at semiconductor and transition metal surfaces, (c) inverse photoemission (bremsstrahlung spectroscopy), (d) electronic structure of rare earth and actinide compounds, and (e) studies of other materials of particular interest.

Major achievements of this program for the past year include:

- (a) Nine papers on the electronic structure of Group IV (diamond, silicon, germanium), III-IV (GaAs grown by molecular beam epitaxy) and II-VI (ZnO) semiconductor surfaces. The experiments on diamond and silicon surfaces have led to a complete rethinking of the nature of group IV semiconductor surfaces by disproving the widely accepted ionic buckling models.
- (b) Six papers on chemisorption and oxidation at surfaces. This program has just been started and is aimed at studies of the more complex electronic structures which occur in semiconductor-oxide and semiconductor-metal interfaces and at catalyst surfaces. Our unmatched experimental capabilities for high-resolution and good-statistics core-level spectroscopy have produced important new results such as the coexistence of up to 4 oxidation states of silicon even in the submonolayer regime and the transition from chemisorbed oxygen to an oxide.
- (c) Four papers on inverse photoemission (or bremsstrahlung spectroscopy) in the ultraviolet. This is a new technique which is complementary to photoemission in probing unoccupied states which are not accessible by other techniques. We are world leaders in this technique (best resolution, tunability of the photon energy) and have discovered unoccupied adsorbate states and semiconductor surface states.
- (d) Eight plus three papers on the electronic structure of rare earth and actinide compounds, respectively. These systematic studies have led to an understanding of the valence change at the surface of certain rare-earth compounds in terms of a binding energy shift of the 4f electrons at the surface. For the actinides (uranium and thorium compounds), the interaction between the 6d- and 5f-like valence electrons has been studied. The transition from localized state to delocalized (band-like) 5f states has been seen by choosing compounds with different spacing of the actinide atoms.

(e) Three papers on various materials. These include the first observation of level splitting due to hydrogen bonding in water clusters adsorbed on surfaces.

As selected examples, three major highlights are described in this section:

- (1) Oxidation of silicon and transition metals.
- (2) Inverse photoemission.
- (3) Hydrogen bonding.

(1) Oxidation of silicon and transition metals

Oxygen Chemisorption and Oxide Formation on Si(111) and Si(100) Surfaces We have used photoemission techniques with synchrotron radiation to study the adsorption of oxygen on the Si(111)-(7×7) and Si(100)-(2×1) surfaces at room temperature in the monolayer and submonolayer regimes. High resolution Si 2p core-level spectra, valence band spectra, work function and Fermi level pinning positions have been measured (see Fig.1) for exposure between 1 L and 1000 L and after annealing at 700°C. Four oxidation states have been detected for the silicon surface atoms with Si 2p core-level shifts of 0.9, 1.9, 2.6, and 3.4 eV which are assigned to silicon atoms bonded to 1, 2, 3, and 4 oxygen atoms. Both (111) and (100) surfaces are characterized by about the same sticking coefficient, a simple adsorption process for 1 L exposures, the quenching of the surface states after 15 L and a saturation of the amount of oxygen after 100 L. Significant differences exist in the initial bonding geometries for the (111) and (100) surfaces. After 15 L exposure, oxygen atoms are incorporated below the Si(111)-(7×7) surface to form SiO₄ units.

Chemisorption-induced 4f-core electron binding-energy shifts for surface atoms of W(111), W(100), and Ta(111) Hydrogen- and oxygen-induced chemical shifts have been resolved in Ta and W substrate 4f core levels by high resolution synchrotron-radiation-excited photoemission spectroscopy. The surface levels shift continuously by typically 100 to 200 meV to higher binding energy for increasing hydrogen coverage. This is due to the quenching of delocalized surface states in the valence band and a charge transfer from the substrate to the hydrogen atoms. Upon adsorption of oxygen, new core peaks appear indicating the formation of more localized chemisorption bands. Substrate chemical shifts for element Z correlate with chemisorption-induced changes in the heat of surface segregation of the (Z+1) constituent in a dilute (Z+1)_x Z_{1-x} alloy.

(2) Inverse photoemission

Electronic states above the Fermi level play an important role, e.g., in ferromagnetism (minority spin states) and in chemisorption (electron acceptor states of adsorbates). These states can be probed by inverse photoemission (or bremsstrahlung spectroscopy) which is the time-reversed photoemission process. Inverse photoemission is unique for sampling states between the Fermi level and the vacuum level without electron hole interaction which affects other spectroscopies of those states. In addition, the momentum of band-like states can be measured. Recently, we have advanced the experimental technique to a level which is

desirable for work on surface and bulk electronic states (e.g., by improving the energy resolution to 0.3 eV, tunable photon energy and variable light polarization).

We have determined the position of the lowest empty orbitals of CO and O chemisorbed on Ni(111) by using inverse photoemission in the ultraviolet. The $2\pi^*$ orbital of CO is lowered by 4.5 eV upon chemisorption and broadened to a wide resonance centered 3.5 eV above E_F (see Fig.3). Chemisorbed oxygen has sharp $2p_{xy}$ states at 1.4 eV above E_F at $k_{\parallel} = 0$ with the total density of states extending from E_F to $E_F + 2$ eV in agreement with band calculations. This state disappears upon oxide formation where the O $2p$ shall become filled.

Angle-resolved inverse photoemission (or bremsstrahlung spectroscopy) with a tunable light detector was used to determine the energy band dispersion of unoccupied minority spin 3d states in Co and Ni. For Co, a ferromagnetic exchange splitting of 0.93 ± 0.1 eV between the unoccupied spin-down and occupied spin-up states (measured with photoemission) is derived (see Fig.4). The full width at half maximum of empty 3d states in Ni is 0.25 ± 0.1 eV.

Unoccupied surface states have been observed for Si(111)-(7x7). These states extend from the Fermi level to the conduction band minimum.

(3) Hydrogen Bonding

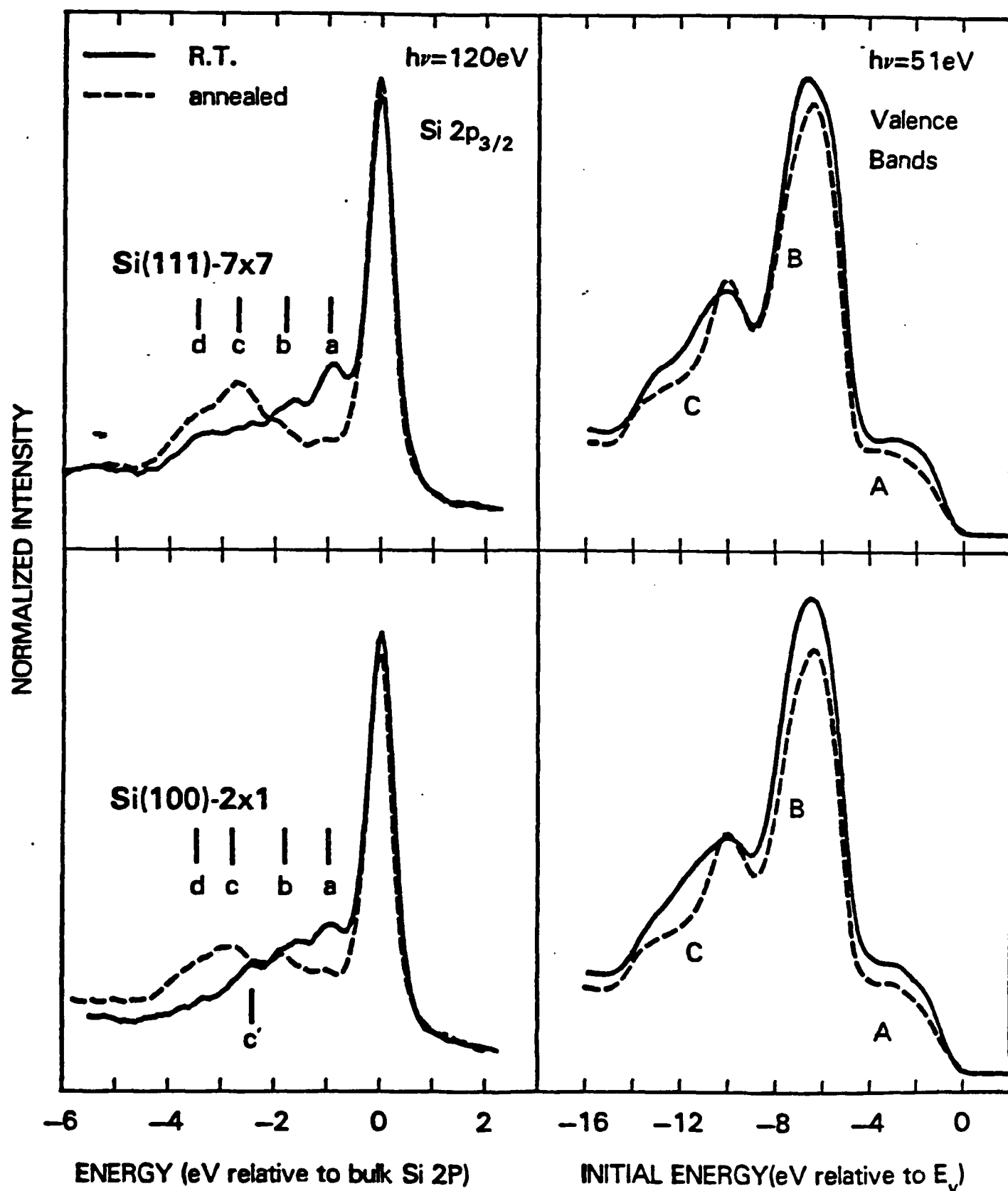
The Electronic Structure of Hydrogen Bonded H_2O . The electronic structure of water-metal interfaces is interesting for the study of electrolytic cells. Our photoemission study reveals for the first time how the electronic structure of water changes when the density of H_2O molecules adsorbed on a surface is increased until hydrogen bonds are formed. Single adsorbed water molecules are found only at submonolayer coverage and substrate temperatures around the desorption temperature in vacuum (160 K). Their molecular orbital structure is similar to the free H_2O molecule except for a rigid shift towards lower binding energies due to final state screening. For a monolayer of adsorbed water, H_2O molecules are bonded to each other by hydrogen bonds. We can distinguish between hydrogen acceptor and hydrogen donor molecules. The molecular orbitals of the hydrogen acceptor are lowered by 1.5 - 2 eV with respect to the donor by the electrostatic field of the positively-charged hydrogen which forms the bridge between donor and acceptor (see Fig.5). For multilayers of adsorbed H_2O (i.e., for ice) the splitting disappears and only a broadening of the molecular orbitals occurs. This is explained by the fact that all H_2O molecules are equivalent in ice and represent donors as well as acceptors. Our findings explain the unusual peak widths in the photoemission spectra of adsorbed water. Also, we can explain the variations in peak separations observed in earlier work as depending on the ratio of proton donors and acceptors.

Figure Captions

- Fig.1: Comparison of Si $2p_{3/2}$ and valence band spectra from (111) and (100) surfaces exposed to 10^3 L at room temperature and after annealing at 700°C . The differences seen between the room temperature spectra for the two surfaces disappear after annealing.
- Fig.2 Angle-integrated 4f core-level emission spectra for Ta(111) as a function of oxygen coverage, showing the quenching of the intrinsic surface core level features S_1 and S_2 and the growth of new chemically-shifted substrate peaks A, C, D, and E. Note that D is a chemically-shifted $4f_{7/2}$ level.
- Fig.3 Angle-resolved inverse photoemission spectra for a clean Ni(111) surface (dash) and $1/3$ monolayer of CO on Ni(111) (line) taken with an initial state energy of 20 eV in the geometry sketched in the upper part of the figure. The CO-induced peak increases its intensity with polar angle θ and is attributed to states derived from the empty CO $2\pi^*$ orbital. The observed angular dependance can be explained by polarization selection rules.
- Fig.4 Using bremsstrahlung spectroscopy and photoemission to determine the ferromagnetic exchange splitting of Co (left column). Bremsstrahlung spectroscopy observes the radiative transitions into the minority spin state above the Fermi level, whereas photoemission detects occupied majority spin states. Right column: By varying the photon energy we change the electron momentum $\vec{k}$ and are able to map out the energy dispersion of the Δ_5, Δ_6 bands.
- Fig.5 Analysis of the photoemission spectrum of a monolayer of adsorbed water at 41 eV photon energy. The observed splitting of the three molecular orbitals is explained by comparison with the calculated binding energies²⁰ of the proton-acceptor (A) and proton-donor (D) levels for the linear water dimer which is shown schematically on the right. For comparison, the spectrum of a multilayer is shown in B where the splitting disappears since most of the molecules are equivalent (such as in ice).

Fig. 1

OXYGEN ON SILICON - 10^3 L exposure -



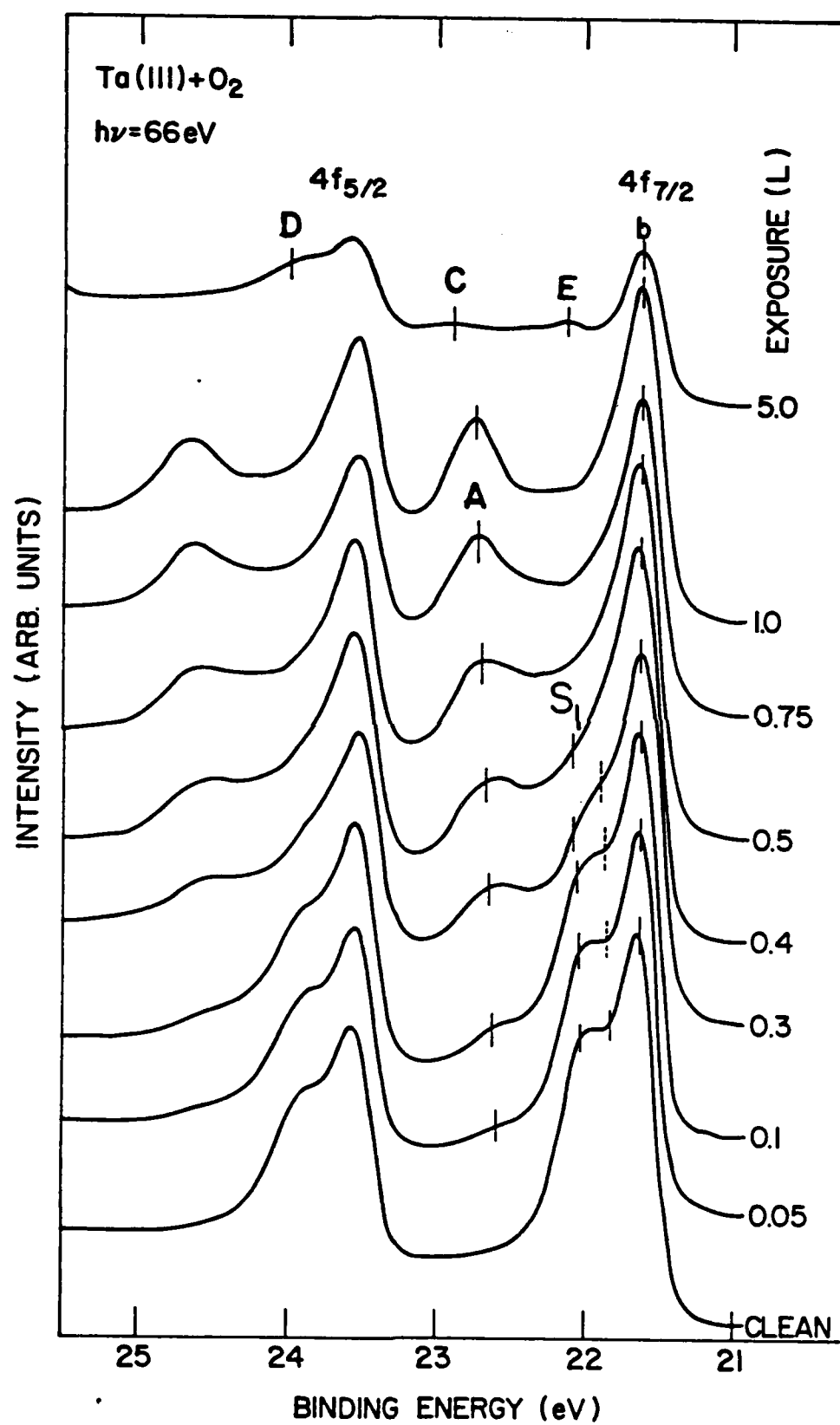


Fig. 2

CO on Ni (111)

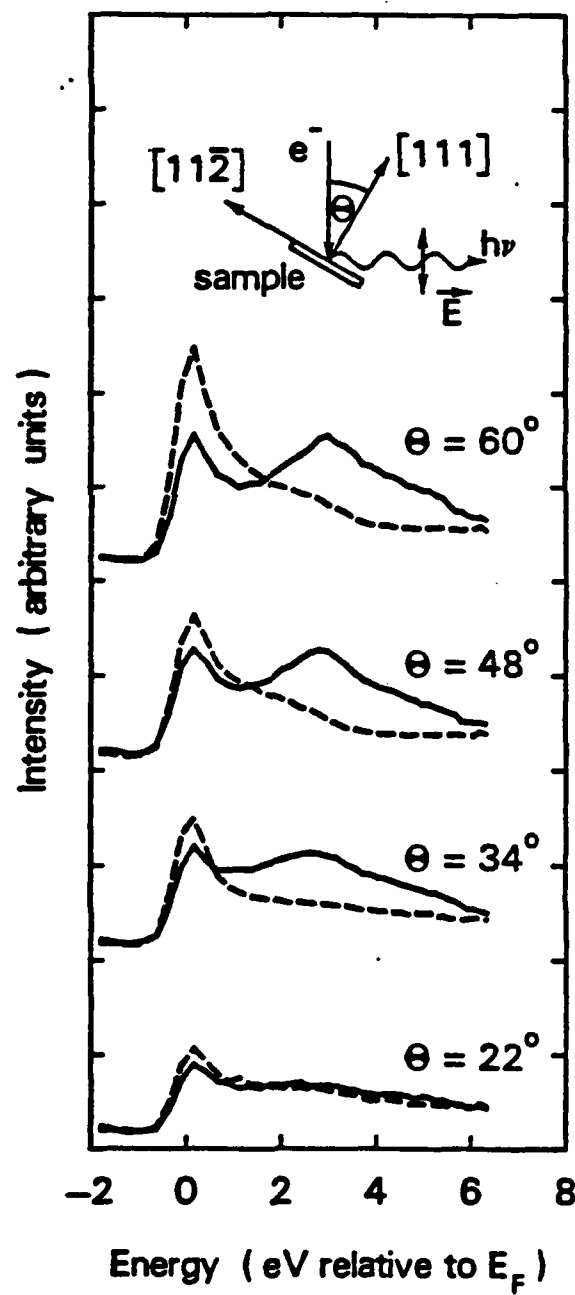
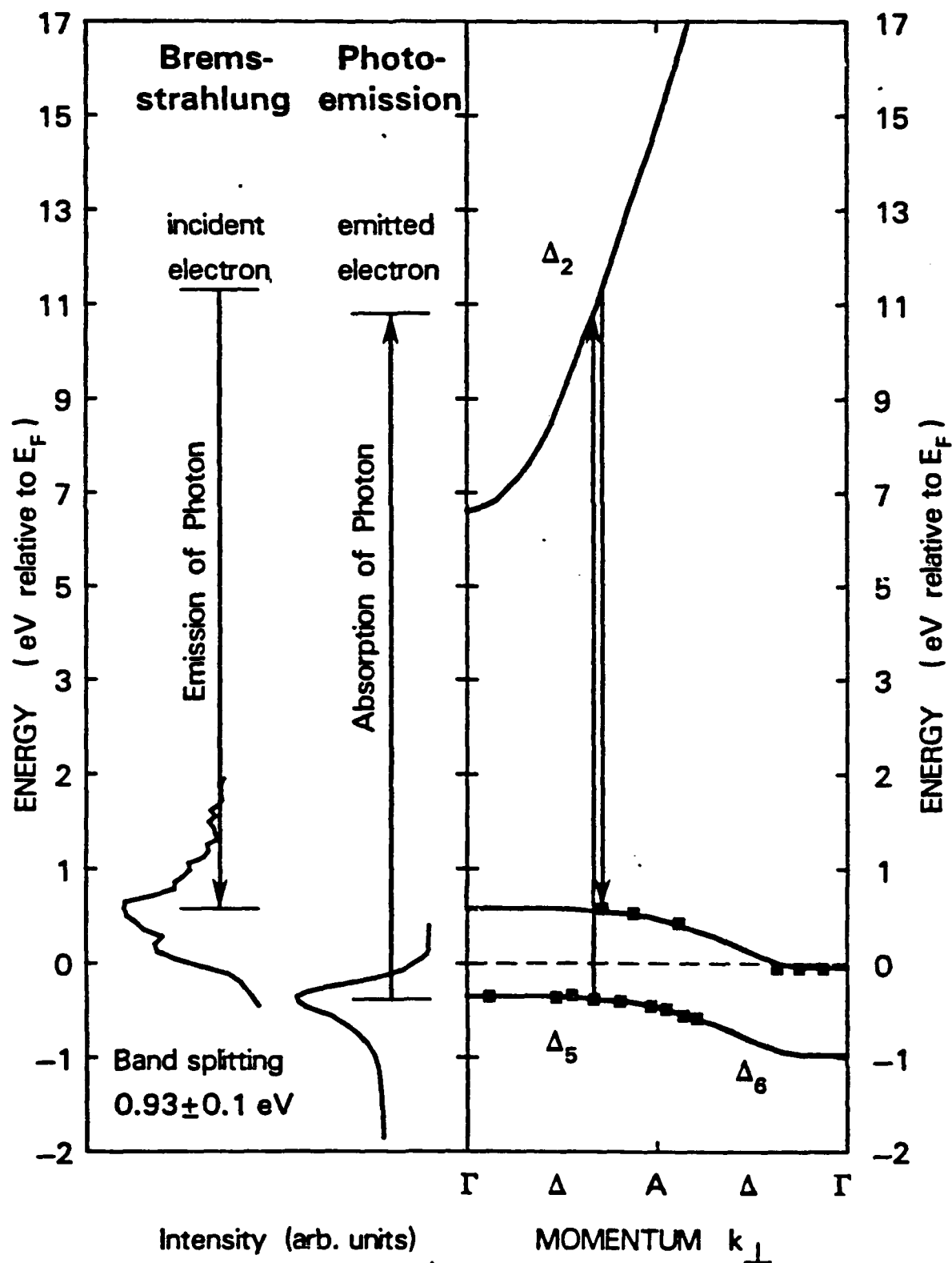


Fig. 3

Fig. 4

BAND MAPPING FOR Co (0001)



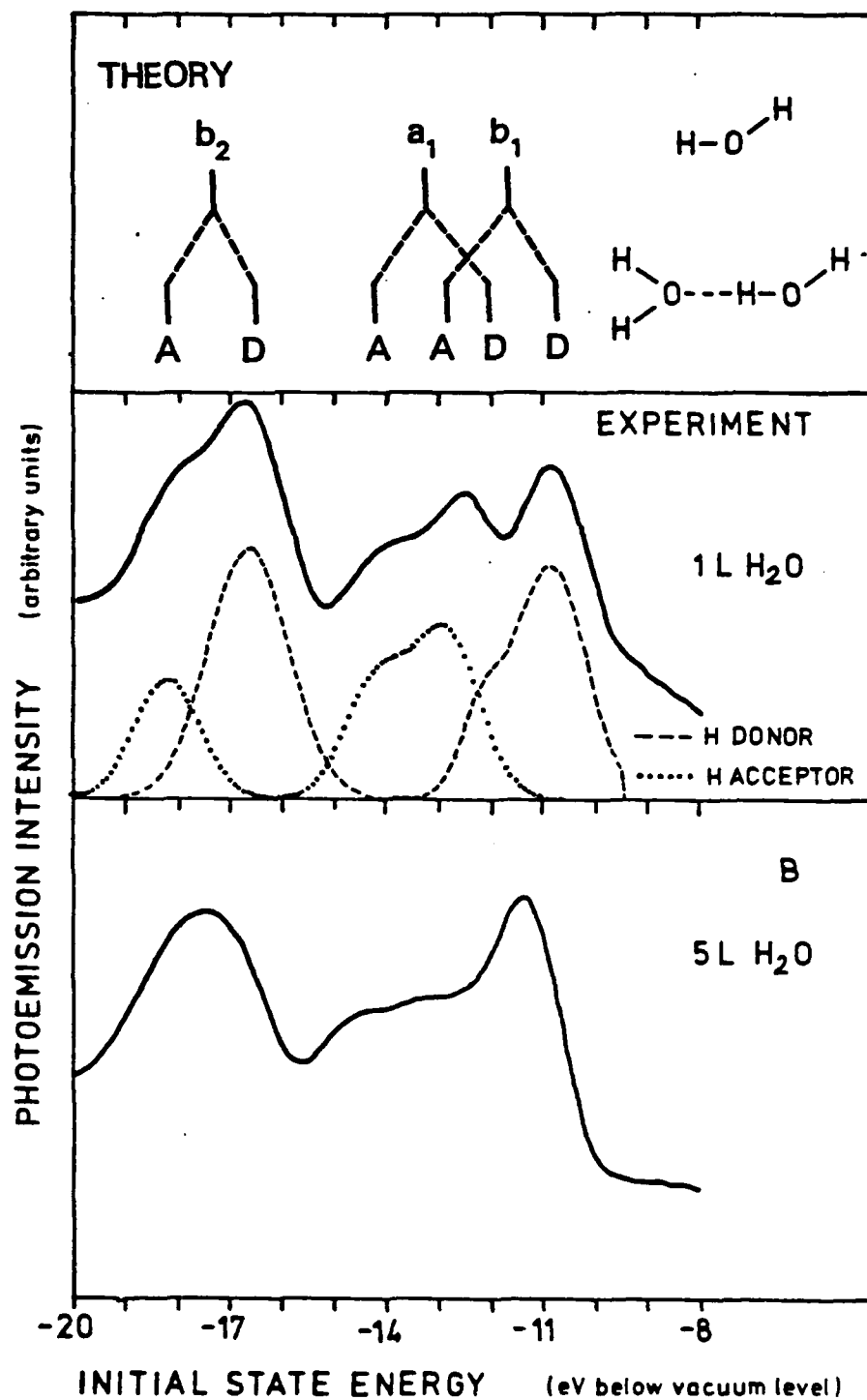


Fig 5

II. SUMMARY OF WORK COMPLETED UNDER CONTRACT

In the following, a brief summary of published research papers that were supported in part by the Air Force Office of Scientific Research under Contract F49620-81-C0089 during the period June 15 1981 to September 15, 1982 (including the three-month cost-free extension). For clarity, these research papers (33 in all) have been grouped into five sub-areas of research:

- A. Electronic structure of semiconductor surfaces and interfaces (Refs.1-9).
- B. Chemisorption and oxidation at semiconductor and transition metal surfaces (Refs.10-15)
- C. Inverse photoemission (bremsstrahlung spectroscopy) (Refs.16-19).
- D. Electronic structure of rare earth and actinide compounds (Refs.20-30).
- E. Other surface/interface studies (Refs.31-33).

A. Electronic Structure of Semiconductor Surfaces and Interfaces

1. Electronic Structure of Si(111) Surfaces

The relation of surface states and surface core levels to the surface geometry is discussed for Si(111) surfaces by comparing photoemission and inverse photoemission results with calculations for different geometries. It is shown that several sets of recent angle-resolved photoemission data for Si(111)-(2x1) and Si(111)-(7x7) are very similar to each other despite the fact that they have been interpreted in terms of different geometries. This indicates that no unique conclusions about the surface geometry can be drawn from the surface electronic structure. At present, only the traditional ionic buckling models can be ruled out rather safely. New information about unoccupied surface states in the gap of Si(111)-(7x7) is obtained from inverse photoemission data. The quenching behavior of occupied surface states under hydrogen exposure shows an unexpected band narrowing which helps identify the origin of surface state bands from the energy level of a localized state at 0.2 eV below the valence band maximum (F.J. Himpsel, Th. Fauster, and G. Hollinger, Surf. Sci., in press).

2. Time Effects in Laser Annealing of the Si(111) Surface

Previous photoemission studies of the surface states on laser-annealed Si(111) have concluded that this surface is (a) similar to the thermally-annealed Si(111)-(7x7) surface for a 2 J/cm² ruby laser pulse but (b) similar to the cleaved Si(111)-(2x1) surface for a 0.4 J/cm² Nd: YAG laser anneal which has a factor-of-two-faster quenching rate than case (a). While it has been suggested that the surface structure depends on the quenching rate and the time scales for the rearrangement of silicon surface atoms, experimental considerations, however, complicate comparison of (a) and (b). In order to examine such time effects and eliminate differences due to

different experimental conditions, we have performed a photoemission experiment identical to case (a) using a XeCl excimer UV laser. An energy density range of 1-4 J/cm² was investigated and both RBS studies of ion implanted samples annealed with this laser and computer simulation model calculations indicate that the range of regrowth quenching rates employed spans that range between case (a) and (b). For all laser-annealing conditions investigated, we find surface state spectra which are similar to case (a) (R.A. Pollak, F.J. Himpsel, G. Hollinger, R.F. Marks, B. Reihl, C.W. White, D.M. Zehner, and R.F. Wood, Phys. Rev. B (Rapid Commun.), in press).

3. **Angle-resolved Photoemission Studies of GaAs(100) Surfaces Grown by Molecular Beam Epitaxy**

Angle-resolved photoemission techniques were used to study the GaAs(100) surfaces grown by molecular beam epitaxy. Three well-ordered surfaces with the c(4×4), c(2×8), and c(6×4) reconstructed surface structures and a disordered As-covered (1×1) surface were investigated in the photon energy range of 12-70 eV. All major sharp peaks in the normal-emission spectra were found to be transitions from bulk valence bands; therefore, transitions due to surface states were either weak or broad. The variations in bulk-peak intensities for different surface structures were related to surface diffraction effects. For photon energies greater than about 20 eV, primary-core peaks for transitions from the upper bulk valence bands to a broadened and shifted free-electron-like final band were identified, and energy dispersion relations for the upper valence bands were determined along the [100] direction. Surface umklapp transitions were quite strong due to the presence of many short surface reciprocal lattice vectors. To characterize these surfaces, angle-integrated photoemission measurements of valence bands and core levels were also made, and the positions of the Fermi level for these surfaces were also determined (T.-C. Chiang, R. Ludeke, M. Aono, G. Landgren, F.J. Himpsel, and D.E. Eastman, Phys. Rev. B, submitted for publication).

4. **Angle-resolved Photoemission from Polar and Nonpolar Zinc Oxide Surfaces**

We report the first angle-resolved photoemission spectra (ARPES) of polar and nonpolar surfaces of an ionic wurtzite-type compound semiconductor. We present experimental results obtained at photon energies between 20 and 80 eV on the polar zinc and oxygen and the nonpolar (10 $\bar{1}$ 0) surface of ZnO and identify surface-induced features by comparison with recent theoretical calculations. Comparison between ARPES experiments and theory confirms the determination of the surface-atom geometry at ZnO (10 $\bar{1}$ 0) by low-energy electron diffraction studies (W. Göpel, J. Pollmann, I. Ivanov, and B. Reihl, Phys. Rev. B26, 3144 (1982)).

5. **Surface States on Reconstructed Diamond(111)**

Using angle-resolved photoemission with synchrotron radiation, surface states on the (2×1)/(2×2) reconstructed diamond(111) surface are characterized. These states cover an energy range of about 2 eV and exhibit maximum emission intensity at 1 eV

below the valence band maximum in normal emission (i.e., at the center $\bar{\Gamma}$ of the surface Brillouin zone). The symmetry is determined to be Λ_1 (s, p_z -like) by using polarization selection rules. For off-normal emission an upwards energy dispersion by about 1 eV is seen. This behavior is similar to the energy position, dispersion and symmetry of surface states on the Si(111)-(2x1) cleavage plane and in contrast with the momentum distribution of the π -band of graphite which is peaked at the boundary of the surface Brillouin zone (F.J. Himpsel, D.E. Eastman, P. Heimann, and J.F. van der Veen, Phys. Rev. B24, 7270 (1981)).

6. A New Model of the Si(111)-(2x1) Surface

Experimental energy dispersions are reported for the surface states of a single-domain Si(111)-(2x1) surface that contradict the results of band calculations for the widely accepted buckled surface model. We present evidence based on *ab initio* calculations using cluster models that the Si(111) surface, if it buckles, should be viewed as only slightly buckled with the singly-occupied dangling bond orbital on each Si coupled into a two-dimensional "Mott insulator" system (A. Redondo, W.A. Goddard III, T.C. McGill, F.J. Himpsel, and D.E. Eastman, submitted to Phys. Rev. Lett.).

7. Surface Electronic Properties of Ion-implanted Laser-annealed Si(111)

Highly-degenerate As-doped n-type and B-doped p-type Si(111)-(1x1) surfaces have been prepared via ion implantation and laser annealing and studied using photoemission. For As concentrations of ~4-7%, surface states become very different from those for intrinsic Si(111)-(1x1) and the Fermi level E_F at the surface moves to the conduction band minima resulting in a zero height n-type Schottky barrier. Emission from the conduction band minima has been directly viewed in momentum space (D.E. Eastman, P. Heimann, F.J. Himpsel, B. Reihl, D.M. Zehner, and C.W. White, Laser and Electron-Beam Interactions with Solids, ed. by B.R. Appleton and G.K. Celler, Elsevier, 1982, p.261.).

8. Photoemission Results for Laser-annealed Si(111) and Ge(111) Surfaces

We have studied the valence band and surface-core-level states for laser-annealed, thermally-annealed, and cleaved Ge(111) and Si(111) surfaces with high resolution photoelectron spectroscopy using synchrotron radiation. For the annealed surfaces we find two surface states near the top of the valence band as well as characteristic surface core level spectra. These indicate the existence of a common local bonding geometry for all these surfaces. We observe that the (1x1) and cleaved (2x1) surfaces are not related as recently reported for Si (F.J. Himpsel, D.E. Eastman, P. Heimann, B. Reihl, C.W. White, and D.M. Zehner, Laser and Electron-beam Interactions with Solids, ed. by B.R. Appleton and G.K. Celler, Elsevier, 1982, p.267.).

9. Electronic Properties of Laser-annealed (111)-(1x1) Surfaces of Highly Doped Silicon

Highly-degenerate As-doped n-type and B-doped p-type Si(111)-(1×1) surfaces have been prepared via ion implantation and laser annealing and studied using photoemission. For As concentrations of ~4-7%, surface states become very different from those for intrinsic Si(111)-(1×1) and the Fermi level E_F at the surface moves to the conduction band minima. For this "flat band" Si surface, the deposition of Au results in a zero height n-type Schottky barrier. Also, emission from the conduction band minima has been directly viewed in momentum space (D.E. Eastman, P. Heimann, F.J. Himpsel, B. Reihl, D.M. Zehner, and C.W. White, Phys. Rev. B24, 3647 (1981)).

B. Chemisorption and Oxidation at Semiconductor and Transition Metal Surfaces

10. Chemisorption-Induced 4f-core-electron Binding-energy Shifts for Surface Atoms of W(111), W(100), and Ta(111)

Hydrogen- and oxygen-induced chemical shifts have been resolved in Ta and W substrate 4f core levels by high resolution synchrotron-radiation-excited photoemission spectroscopy. The surface levels shift continuously by typically 100 to 200 meV to higher binding energy for increasing hydrogen coverage. This is due to the quenching of delocalized surface states in the valence band and a charge transfer from the substrate to the hydrogen atoms. Upon adsorption of oxygen, new core peaks appear at 0.4 to 1.1 eV higher binding energy w.r.t. the bulk level, indicating the formation of more localized chemisorption bands. Substrate chemical shifts for element Z correlate with chemisorption-induced changes in the heat of surface segregation of the (Z+1) constituent in a dilute $(Z+1)_x Z_{1-x}$ alloy (J.F. van der Veen, F.J. Himpsel, and D.E. Eastman, Phys. Rev. B25 7388 (1982)).

11. Photoemission Studies of Surface Core-level Shifts and Their Applications

With the use of synchrotron radiation and a high resolution electron spectrometer, shifts in the core level binding energies of surface atoms relative to bulk atoms have been measured for a variety of metals and semiconductors. Surface core level shifts for transition metals show strong dependencies on surface crystallography (Ir, W, Au) and on the number of valence d electrons; e.g., surface core level shifts to higher binding energies are observed for rare earth metals and Ta(111) which have less than half-filled d-shells, and half-filled d-shells. Surface core level shifts are large for materials with valence d-electrons and for covalent semiconductors (~0.2-1.0 eV) and are smaller for metals with s, p electrons (e.g., $\delta E_g \approx 0.057$ eV for Al(001)). Applications of surface core level shifts to be discussed include: surface reconstruction on metals (W(100)) and semiconductors (Si(111)-(2×1) and (7×7)), surface valence changes on mixed-valent rare earths (YbAl₂), and high resolution chemisorption studies (Ta, Si, and rare earth metals) (D.E. Eastman and F.J. Himpsel, J. Vac. Sci. Technol. 20 609 (1982)).

12. Oxygen Chemisorption and Oxide Formation on Si(111) and Si(100) Surfaces

We have used photoemission techniques with synchrotron radiation to study the adsorption of oxygen on the Si(111)-(7×7) and Si(100)-(2×1) surfaces at room temperature in the submonolayer and monolayer regimes. High resolution Si 2p core-level spectra, valence band spectra, work function and Fermi level pinning positions have been measured for exposure between 1 L and 1000 L and after annealing at 700°C. Four oxidation states have been detected for the silicon surface atoms with Si 2p core-level shifts of 0.9, 1.9, 2.6, and 3.4 eV which are assigned to silicon atoms bonded to 1, 2, 3, and 4 oxygen atoms. Both (111) and (100) surfaces are characterized by about the same sticking coefficient, a simple adsorption process for 1 L exposures, the quenching of the surface states after 15 L and a saturation of the amount of oxygen after 100 L. Significant differences exist in the initial bonding geometries for the (111) and (100) surfaces. After 15 L exposure, oxygen atoms are incorporated below the Si(111)-(7×7) surface to form SiO₄ units (G. Hollinger and F.J. Himpsel, J. Vac. Sci. Technol., in press).

13. Adsorbate Band Dispersions for C on Ru(0001)

We have studied carbon-induced two-dimensional energy bands on Ru(0001) using angle-resolved photoelectron spectroscopy and have compared them with *ab initio* calculations. We find a nearly parabolic band (bottom at $E_F - 9.8$ eV at $\vec{k}_\parallel = 0$, effective mass $\sim 1.5 m_e$) which we assign to the C2p_z valence states of a graphitic carbon overlayer. Compared to graphite, these states are bound more tightly by 2.3 eV (F.J. Himpsel, K. Christmann, P. Heimann, D.E. Eastman, and P.J. Feibelman, Surf. Sci. 115, L159 (1982)).

14. Adsorbates of a Charge-Transfer Instability Exist for Rare-Gas Atoms Adsorbed on Metals?

Recent optical-absorption experiments on rare-gas atoms bonded to metals dramatically segregate various rare-gas/metal systems into two classes. Cunningham, Greenlaw and Flynn have hypothesized that these two classes are characterized by the presence or absence of charge transfer from the (excited) rare-gas atom to the metal, and that such charge transfer is controlled by the sign of the difference $\Phi - I^*$, where Φ is the metal work function and I^* is the energy required to ionize the rare-gas atom in its lowest excited state. Flynn and Chen have, in addition, collected data describing the dipole moments of adsorbed Xe; these also suggest a dramatic dependence on the quantity $\Phi - I^*$. As a test of this hypothesis, we have measured the dipole moment of Xe adsorbed on a low-work-function substrate (Gd(0001)). Both the new measurement, and new calculations using the atom-on-jellium model, are *inconsistent* with the interpretation emphasizing $\Phi - I^*$. Our analysis suggests that the behavior seen in the optical-absorption measurements could represent a physical effect even more unusual than the charge-transfer instability proposed by Flynn *et al.* (N.D. Lang, A.R. Williams, F.J. Himpsel, B. Reihl, and D.E. Eastman, Phys. Rev. B20, 1728 (1982)).

15. Photoemission Spectroscopy of Surfaces and Adsorbates

Core level photoelectron spectroscopy is providing new information concerning the electronic properties of adsorbates and surfaces. For rare gas multilayers adsorbed on metal surfaces, the photoelectrons and Auger electrons exhibit well-resolved increases in kinetic energy with decreasing distance between the excited atom and the substrate, allowing a direct labeling of the layers. These energy shifts are mainly due to the substrate screening effects, and can be described well by an image-charge model. For a Kr/Xe bilayer system prepared by first coating a Pd substrate with a monolayer of Kr and then overcoating with a layer of Xe, a thermally-activated layer inversion process is observed when the temperature is raised, with Xe coming in direct contact with the substrate. For rare gas submonolayers adsorbed on the Al(111) surface, coverage-dependent core level shift and work function measurements provide information about the adatom spatial distributions, polarizabilities, and dipole moments for the ground and excited states. We have also studied core level shifts for a clean Al(001) surface relative to the bulk. The shifts have a large contribution from the initial-state effects (T.-C. Chiang, G. Kaindl, F.J. Himpsel, and D.E. Eastman, AIP Conference Series, in press).

C. Inverse Photoemission (Bremsstrahlung Spectroscopy)

16. Empty Orbitals of Adsorbates Determined by Inverse UV Photoemission

We have determined the position of the lowest empty orbitals of CO and O chemisorbed on Ni(111) by using inverse photoemission in the ultraviolet. The $2\pi^*$ orbital of CO is lowered by 4.5 eV upon chemisorption and broadened to a 5.5 eV wide resonance centered 3.5 eV above E_F . Chemisorbed oxygen has sharp $2p_{xy}$ states at 1.4 eV above E_F at $k_{\parallel} = 0$ with the total density of states extending from E_F to $E_F + 2$ eV in agreement with band calculations. This state disappears upon oxide formation (F.J. Himpsel and Th. Fauster, Phys. Rev. Lett. 49, 1583 (1982)).

17. Mapping of Unoccupied Minority Spin Bands Using Inverse Photoemission

Angle-resolved inverse photoemission (or bremsstrahlung spectroscopy) with a tunable light detector is used to determine the energy band dispersion of unoccupied minority spin 3d states in Co and Ni. For Co, a ferromagnetic exchange splitting of 0.93 ± 0.1 eV between the unoccupied spin-down and occupied spin-up states (measured with photoemission) is derived. The full width at half maximum of empty 3d states in Ni is 0.25 ± 0.1 eV (F.J. Himpsel and Th. Fauster, Phys. Rev. B26, 2679 (1982)).

18. $2\pi^*$ -derived States for CO on Ni(111) Studied by UV Bremsstrahlung Spectroscopy

Angle-resolved and polarization-dependent UV bremsstrahlung spectroscopy (inverse photoemission) is used to determine the energy and symmetry of the $2\pi^*$ -derived states for CO on Ni(111). We observe a 3 eV wide band centered at 3 eV above the Fermi level. This shows that the electron hole interaction for transitions $C1s \rightarrow 2\pi^*$ is reduced from 10 eV in the gas phase to 1 eV for chemisorbed CO due to screening by metal electrons. Compared with physisorbed and gas phase CO, chemisorption on

Ni(111) lowers the energy of the $2\pi^*$ orbital and increases its width (Th. Fauster and F.J. Himpsel, Phys. Rev. B, submitted for publication).

19. **Momentum-Resolved Bremsstrahlung Spectroscopy with a Tunable Photon Detector**

The first momentum-resolved bremsstrahlung (or inverse photoemission) experiments using a tunable light detector in the energy range from 10 to 40 eV are reported. The experimental setup comprises Pierce-type electron gun, Seya monochromator, and position-sensitive channel plate device. An energy resolution of 0.3 eV at 20 eV has been achieved and the angular resolution is estimated to 6° . The apparatus has been used to determine the energy band dispersion of unoccupied minority spin 3d states in Co. A ferromagnetic exchange splitting of 0.93 ± 0.1 eV between the unoccupied spin-down and occupied spin-up states (measured with photoemission) is derived. Another application is the measurement of unoccupied adsorbate levels of CO and O on Ni(111). Detecting the polarization of the photons (using the polarization-dependent reflectivity of the monochromator) enables us to identify the symmetry character of the adsorbate orbitals (Th. Fauster and F.J. Himpsel, J. Vac. Sci. Technol., submitted for publication).

D. Electronic Structure of Rare Earth and Actinide Compounds

20. **Photoemission of Spin-Polarized d States in Gadolinium**

Recently spin-polarized photoemission from gadolinium films was interpreted in terms of a $4f \rightarrow 5d$ resonant scattering mechanism. In view of our photoemission data we believe that this mechanism is very unlikely. Instead, the spin-polarization data can be explained in a straightforward manner taking into account the Gd band structure and simple photoemission escape cone rules (B. Reihl and F.J. Himpsel, Solid State Commun. 44, 128 (1982)).

21. **Systematics of the 4f Ionization Energies in the Rare Earth Prictides**

The 4f binding energies have been measured for several trivalent rare earth antimonides: NdSb, GdSb, ErSb and LuSb. It is shown that the 4f level shift, relative to the pure metals, is essentially a constant over the lanthanide series. The shift for CeSb is extrapolated and used to identify the screened $4f^0$ photoionized final state in the spectra from γ -Ce and CeSb. In a similar way the measured 4f position in GdP is used to show that the Ce^{3+} to Ce^{4+} excitation in CeP requires an energy of about 3 eV. This seems to suggest that the high-pressure phase of CeP, and in analogy the ground state of CeN, should not be described as mixed-valent systems within the framework of the promotional model. Instead we suggest that these systems are analogous to the α -phase of Ce which by f-counting methods is shown to have essentially a 4f configuration (N. Mårtensson, B. Reihl, and F.Holtzberg, Valence Instabilities, ed. P. Wachter, North-Holland, Amsterdam (1982)).

22. **Nature of the Ground State in α -Cerium as Studied by Photoemission**

We have studied, through temperature-dependent resonant photoemission experiments, the changes in the 4f electronic states of the system $Ce_{0.9}Th_{0.1}$ produced by the first order γ - α valence transition. The results rule out the commonly held interpretation that the γ - α transition in cerium arises from the promotion of a 4f atomic-like state near the Fermi surface to the 5d, 6s conduction band (N. Mårtensson, B. Reihl, and R.D. Parks, Solid State Commun. 41, 573 (1982)).

23. **Highly-resolved Surface Shifts in a Mixed-valent System: $EuPd_2Si_2$**

Synchrotron-radiation-excited photoelectron spectroscopy is employed to study the bulk and surface states of the mixed-valent system $EuPd_2Si_2$, which undergoes a valence transition from $Eu^{2.2+}$ at room temperature to $Eu^{2.9+}$ at low temperature (≤ 100 K). The high resolution employed, coupled with a relatively large bulk-to-surface energy shift to the Eu^{2+} emission, allows a definitive identification of the bulk and surface states of Eu^{2+} . The Eu^{3+} emission is not observed at room temperature, but is readily apparent in the low-temperature spectra (N. Mårtensson, B. Reihl, W.D. Schneider, V. Murgai, L.C. Gupta, and R.D. Parks, Phys. Rev. B25, 1446 (1982)).

24. **Single-site Mixed Valence of Thulium: A Comparative Photoemission Study of $Tm_xY_{1-x}Si$ and $Tm_xY_{1-x}S$**

The dilution of Tm by Y in mixed-valent TmSe and trivalent TmS has been studied by photoelectron spectroscopy using synchrotron radiation. TmSe is found to stay mixed-valent down to at least 5% Tm while Tm is trivalent in all the studied $Tm_xY_{1-x}S$ compounds. The results point towards a single-site description of mixed-valence. We find that both the selenides and the sulfides have a divalent surface layer, due to a surface shift at the 4f level. In TmSe this shift is resolved and found to be -0.4 eV. In the trivalent sulfides only one monolayer is divalent while in the mixed valent selenides the divalent layer penetrates deeper (N. Mårtensson, B. Reihl, R.A. Pollak, F. Holtzberg, G. Kaindl, and D.E. Eastman, Phys. Rev. B26, 648 (1982)).

25. **Surface Effects on Core-level Binding Energies and Valence in Thulium Chalcogenides**

Vacuum-cleaved (100) surfaces of mixed-valent TmSe, divalent TmTe, and trivalent TmS were studied by high-resolution, angle-integrated photoelectron spectroscopy with the use of synchrotron radiation in the energy range $30 \leq h\nu \leq 110$ eV. In the topmost surface layers of TmSe and TmTe the 4f levels are found to be shifted to higher binding energies by 0.32 ± 0.04 and 0.41 ± 0.05 eV, respectively. In both TmSe and TmS the topmost surface layers are divalent. In the case of TmSe a separation of $Tm^{2+}4f^{12}$ spectral feature into surface and bulk contributions allows a determination of the bulk mean valence $\bar{v} = 2.55 \pm 0.05$. While a shift of the Se 3d levels to lower binding energy is observed for Se atoms in the topmost surface layer of TmSe, no such shift can be resolved for the Te 4d levels of TmTe. The surface-derived divalent spectral features can be quenched completely in all three cases by exposure of the surfaces to submonolayer amounts of oxygen, resulting in the formation of trivalent surface oxides. Values for the electron mean free path ℓ are derived from the ob-

served surface- and bulk-spectral intensities, with I decreasing with decreasing electron kinetic energy down to ≈ 45 eV. Smaller singularity indices α of the Doniach-Sunjić line shapes as well as smaller extrinsic losses are observed for photoemission from the divalent surface layers as compared to the bulk (G. Kaindl, C. Laubschat, B. Reihl, R.A. Pollak, N. Mårtensson, F. Holtzberg, and D.E. Eastman, Phys. Rev. B26, 1713 (1982)).

26. **Bulk and Surface Valence States in Rare Earth Monosulfides**

We have studied the 4f photoelectron spectra of SmS, EuS, DyS, TmS, and YbS using high resolution ($\Delta E < 150$ meV) photoemission with synchrotron radiation. By means of a systematic treatment of the 4f ionization energies the bulk and surface valence states of these and several other rare earth sulfides are determined. In particular, we find in TmS that the trivalent state is stable by only 0.2 eV and we show that, due to this low stability, the surface in TmS is divalent (N. Mårtensson, B. Reihl, R.A. Pollak, F. Holtzberg, and G. Kaindl, Phys. Rev. B25, 1713 (1982)).

27. **Eu Valence and Surface Core Level Shifts in EuPd_x Compounds as Studied by Photoelectron Spectroscopy**

For the EuPd_x system (with the addition of EuPd_2Si_2), we study how the Eu 4f photoemission spectrum evolves as the Eu valence is brought from a divalent, through a mixed-valent to a trivalent state. In the divalent and mixed-valent compounds, a 4f surface shift is clearly seen. In the trivalent Eu compounds, as well as in mixed-valent EuPd_2Si_2 , we detect a stable-valent Eu^{2+} surface layer. The trivalent compounds EuPd_3 and EuPd_5 form a most interesting class of magnetic materials with a non-magnetic Eu^{3+} bulk-covered by a few Å thick layer of magnetic Eu^{2+} atoms. The 4f surface peak shows a smaller chemical shift as a function of Pd alloying than the bulk peak. This is explained in terms of the reduced number of Eu-Pd bonds for a surface Eu atom (V. Murgai, L.C. Gupta, R.D. Parks, N. Mårtensson, and B. Reihl, Valence Instabilities, ed. P. Wachter, North-Holland, Amsterdam, (1982)).

28. **Resonant Photoemission as a Tool to Study f and d Electrons in Actinide Compounds**

Using synchrotron radiation in the energy range $60 \leq h\nu \leq 130$ eV, we have measured $h\nu$ -dependent photoemission energy distribution curves and constant-initial-state spectra from single crystals of $\text{U}_2\text{Th}_{1-x}\text{Sb}$, UTe , UO_2 , and UPd_3 . The U 5f emission shows the strongest intensity variation, which can be explained by the super-Coster-Kronig character of the decay of the autoionizing core excited state. Thus f and d emission features in the photoemission spectra are unambiguously identified by their different resonance behavior. It is concluded that the spectra are dominated by localized 5f states in all of these compounds (B. Reihl, N. Mårtensson, D.E. Eastman, A.J. Arko, and O. Vogt, Phys. Rev. B26, 1842 (1982)).

29. **Resonant Photoemission Study of $\text{UNi}_{5-x}\text{Cu}_x$**

Valence band spectra of the intermetallic compounds $\text{UNi}_{5-x}\text{Cu}_x$ ($x = 0.0, 2.5, \text{ and } 5$) have been measured using synchrotron radiation in the energy range 10 eV to 130 eV. In all compounds the 5f-derived valence band density of states is pinned to the Fermi energy. In contrast to earlier work, no splitting of the U 5f emission due to different final state 5f configurations is observed in UCu_5 . This points towards an itinerant description of the 5f electrons in these compounds. A decrease in the intensity of the Ni two-hole satellite is observed in going from Ni-metal and UNi_5 to $\text{U}(\text{Ni}_{5-x}\text{Cu}_x)_5$, which can be related to a filling of the Ni d-band. Furthermore, a shift of the Ni satellite energy by 1.3 eV towards higher binding energies occurs in $\text{U}(\text{Ni}_{5-x}\text{Cu}_x)_5$ (W.-D. Schneider, B. Reihl, N. Mårtensson, and A.J. Arko, Phys. Rev. B

30. **Binding Energies and Heat-of-Formation Data for $\text{USb}_x\text{Te}_{1-x}$ and $\text{UAs}_x\text{Se}_{1-x}$ Compounds as Derived from Photoelectron Spectroscopy**

Using high-resolution photoelectron spectroscopy with synchrotron radiation we have accurately determined the core-level binding energies in the nonuranium atoms in $\text{USb}_x\text{Te}_{1-x}$ and $\text{UAs}_x\text{Si}_{1-x}$ as a function of the stoichiometric composition. With the use of a Born-Haber cycle a relation between the measured shifts and heat-of-formation data for these compounds is formulated. The analysis demonstrates that photoelectron spectroscopy can become a most valuable method for deriving new heat-of-formation data (N. Mårtensson, B. Reihl, and O. Vogt, Phys. Rev. B25, 824 (1982)).

E. Other Surface/Interface Studies

31. **The Electronic Structure of Hydrogen-Bonded H_2O**

We have studied the electronic structure of H_2O adsorbed on different metal surfaces between 7 K and 200 K using photoelectron spectroscopy. From the valence orbital spectra we are able to distinguish three different phases of adsorbed H_2O : (A) single-adsorbed H_2O molecules at temperatures close to the desorption point, (B) partially hydrogen-bonded H_2O clusters for coverages of a monolayer or less, (C) fully hydrogen-bonded ice at low temperatures and several monolayers of coverage. For A, we find valence molecular orbitals which are almost rigidly shifted upwards relative to the gas phase by a final-state relaxation shift of 1.3 eV. All orbitals are broadened by 1.0 - 1.5 eV relative to the gas phase. For (B) we identify two inequivalent types of H_2O molecules whose orbital energies differ by 1.5 - 2 eV. This splitting is identical to the electrostatic shift of molecular orbital energies as calculated for the hydrogen-bonded H_2O dimer by Umeyama and Morokuma. In this model the set of molecular orbitals with higher binding energy is assigned to the hydrogen acceptor molecule and the set with lower binding energy to the hydrogen donor molecule. At monolayer coverage we find about twice as many donors as acceptors (D. Schmeisser, F.J. Himpsel, G. Hollinger, and B. Reihl, Phys. Rev. B, in press).

32. **Integer vs. Non-Integer Valence Properties Revealed by a Comparative Photoemission Study of Na_xWO_3 and ReO_3**

Fano-type resonances in the 5d conduction band intensity spectra have been observed at the $5p_{1/2}$ and $5p_{3/2}$ photoabsorption thresholds for Na_xWO_3 ($0.4 < x < 0.85$), ReO_3 , WO_2 , and W. The Na_xWO_3 tungsten bronze spectra exhibit a characteristic double resonance structure different from the simple resonance peaks measured for ReO_3 , WO_2 , and W. These results could be related to a mixed valence character of the metallic bronzes revealed in the final state of the photoemission process. The complex structure of the photoelectron core level 4f spectra for Na_xWO_3 and ReO_3 are discussed on the basis of these new findings (G. Hollinger, F.J. Himpsel, N. Mårtensson, B. Reihl, J.P. Doumerc, and T. Akahane, submitted to Phys. Rev. B).

33. On the Applicability of the Rigid Band Model to the Metallic Sodium Tungsten Bronzes: A Photoemission Study Using Synchrotron Radiation

W4f, Na2p photoelectron spectra and valence band spectra are reported for a series of cubic metallic Na_xWO_3 ($0.4 < x < 0.85$) bronzes in the 20-130 eV photon energy range. From a comparison of experimental and theoretical conduction band densities of states it is found that in the measured composition range the trends of the rigid band model behavior are respected (G. Hollinger, F.J. Himpsel, B. Reihl, P. Pertosa, and J.P. Doumerc, submitted to Solid State Commun.).

III. LIST OF PUBLICATIONS UNDER CONTRACT

A. Electronic Structure of Semiconductor Surfaces and Interfaces.

1. F.J. Himpsel, Th. Fauster, and G. Hollinger, Electronic Structure of Si(111) Surfaces, Surf. Sci., in press.
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5. F.J. Himpsel, D.E. Eastman, P. Heimann, and J.F. van der Veen, Surface States on Reconstructed Diamond(111), Phys. Rev. B24, 7270 (1981).
6. A. Redondo, W.A. Goddard III, T.C. McGill, F.J. Himpsel, and D.E. Eastman, A New Model of the Si(111)-(2x1) Surface, submitted to Phys. Rev. Lett.
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B. Chemisorption and Oxidation at Semiconductor and Transition Metal Surfaces

10. J.F. van der Veen, F.J. Himpsel, and D.E. Eastman, Chemisorption-induced 4f-core-electron Binding-energy Shifts for Surface Atoms of W(111), W(100), and Ta(111), Phys. Rev. B25 7388 (1982)).
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13. F.J. Himpsel, K. Christmann, P. Heimann, D.E. Eastman, and P.J. Feibelman, Adsorbate Band Dispersions for C on Ru(0001), Surf. Sci. 115, L159 (1982)).
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15. T.-C. Chiang, G. Kaindl, F.J. Himpsel, and D.E. Eastman, Photoemission Spectroscopy of Surfaces and Adsorbates, AIP Conference Series, in press.

C. Inverse Photoemission (Bremsstrahlung Spectroscopy)

16. (F.J. Himpsel and Th. Fauster, Empty Orbitals of Adsorbates Determined by Inverse UV Photoemission, Phys. Rev. Lett. 49, 1583 (1982).
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31. D. Schmeisser, F.J. Himpsel, G. Hollinger, and B. Reihl, The Electronic Structure of Hydrogen-Bonded H_2O , *Phys. Rev. B*, in press.
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33. G. Hollinger, F.J. Himpsel, B. Reihl, P. Pertosa, and J.P. Doumerc, On the Applicability of the Rigid Band Model to the Metallic Sodium Tungsten Bronzes: A Photoemission Study Using Synchrotron Radiation, submitted to *Solid State Commun.*

IV. Biography of Principal Investigator

Franz J. Himpsel received his Diploma degree in Physics in 1973 and his Ph.D. degree in Physics in 1976 from the University of Munich. He worked at DESY (Hamburg) in the synchrotron radiation group of Prof. W. Steinmann from 1974-1976. From 2/77 to 6/78 he held a post-doctoral position in D.E. Eastman's group at IBM, Yorktown Heights, NY, and from 6/78 to 1/79 he held the position of Visiting Scientist at the Synchrotron Radiation Center, University of Wisconsin, Madison. Currently he is Manager of the Photoemission and Surface Physics group at IBM, Yorktown Heights, NY.

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1. N. Schwentner, F.J. Himpsel, E.E. Koch, V. Saile, M. Skibowski, Energy Distribution of Photoelectrons from the Valence Band of Solid Xenon, *Vacuum Ultraviolet Radiation Physics*, ed. by E.E. Koch, R. Haensel, C. Kunz; Pergamon-Vieweg. p.335 (1974).
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