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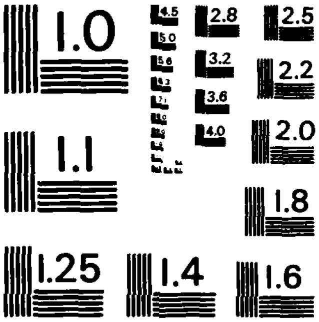

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REPORT DOCUMENTATION PAGE

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1. REPORT NUMBER 4

2. GOVT ACCESSION NO.

3. RECIPIENT'S CATALOG NUMBER

4. TITLE (and Subtitle) DETERMINATION OF ELECTRONIC AND ATOMIC STRUCTURAL PARAMETERS DURING FAST ION TRANSPORT

5. TYPE OF REPORT & PERIOD COVERED Final Report July 1, 1983- Nov. 30 1985

6. PERFORMING ORG. REPORT NUMBER

7. AUTHOR(s)
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8. CONTRACT OR GRANT NUMBER(s) N 00014-83-C-0620

ERFORMING ORGANIZATION NAME AND ADDRESS
he Boeing Co., Seattle, WA 98124
Electrochemical Technology Corp., 1601 Dexter Ave. N.
Seattle, WA. 98109

10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS

CONTROLLING OFFICE NAME AND ADDRESS
fice of Naval Research
0 N. Quincy Street
lington, VA 22217

12. REPORT DATE November 1985

13. NUMBER OF PAGES

MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)

15. SECURITY CLASS. (of this report) UNCLASSIFIED

15a. DECLASSIFICATION DOWNGRADING SCHEDULE

DISTRIBUTION STATEMENT (of this Report)

proved for public release; distribution unlimited.

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DEC 0 5 1985
E

17. DISTRIBUTION ST ENT (of abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY TES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Fast ion conductor, vanadium bronze, X-ray absorption spectroscopy, tungsten bronz

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

X-ray absorption spectroscopy was used to determine the valence state and local structure in a vanadium bronze and a tungsten bronze series of fast ion conducting materials. The structure parameters confirm previous x-ray diffraction results; however, the direct determination of valence state(s) is a unique result. X-ray methods appear to offer real advantages for in situ characterization.

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DETERMINATION OF ELECTRONIC AND ATOMIC STRUCTURAL PARAMETERS DURING FAST ION TRANSPORT

Introduction

The goal of this research was to use x-ray absorption spectroscopy as a probe to investigate the electronic and atomic structure of the different elements contained in typical fast ion conductor materials. X-ray absorption spectroscopy is able to single out each different kind of element in a complex material because the core-level absorption edges are well separated in energy. The region near the absorption edge, x-ray absorption near edge structure (XANES) contains information on the valence state and local symmetry of the absorbing atom. The extended x-ray absorption fine structure (EXAFS) above the absorption edge is caused by scattering of the ejected photoelectron and may be used to determine near neighbor structural parameters. As will be shown these techniques were quite successful in their intended applications.

Accomplishments

We prepared and obtained data at SSRL on two series of fast ion conductors, the vanadium bronze, $M_X V_2O_5$ and tungsten bronze, M_XWO_3 compounds. Where M is usually an alkali metal atom. Although the crystal structures of these materials had previously been determined by x-ray diffraction$^{(1)}$ many of the valence assignments had been deduced by chemical arguments.$^{(2)}$ For example, in the compound $Na^{+1}_X\ (V^{+4}_X\ V^{+5}_{2-X})\ O_5$ each Na^{+1} ion which enters the structure is supposed to switch one V^{+5} to V^{+4} in order to maintain charge neutrality as indicated by the formula. We first studied an extensive series of V compounds$^{(3)}$ in order to understand the near edge structure of V in various coordinations and valences. The significant result was that V in a distorted oxygen environment (as occurs in V_2O_5) has a sharp pre-edge peak in its XANES spectrum which shifts in energy with change in valence. In these compounds with a mix of V^{+4} and V^{+5} we were able to isolate the spectrum from each atom site simply by subtracting one spectrum from another. This is shown in figure 1. The spectrum at the top is $Na_{.4}V_2O_5$, underneath is pure V_2O_5 and at the bottom are the results of subtracting V_2O_5 from $Na_{.4}V_2O_5$ by the fractions indicated. The broadened pre-edge peak is observed to resolve into a doublet and uncover the V^{+4} spectrum. We used this type of analysis to determine directly the valence mix and local symmetry of the V_2O_5 lattice. The work is described in the attached pre-print.(4) The charge state and the local structure was determined for the atoms added to V_2O_5. This is illustrated by the case of $Cu_{.26}V_2O_5$. Again we used the subtraction technique to verify that Cu was switching V^{+5} to V^{+4}. Then the spectrum of Cu was compared to many model Cu compounds. A sample of this is shown in figure 2 where Cu in 2-fold, 4-fold and 6-fold coordination is shown. The spectra are noted in detail in the caption; however, the result was that Cu in V_2O_5 has a unique environment agreeing in detail with none of the model compounds. It is Cu^{+1} but the environment is not simple. The EXAFS was analyzed in detail to determine the structure. The result shows Cu^{+1} to be in a distorted octahedral environment with 3 oxygen neighbors at 1.88 ± .01 Å and 3 more at 2.11 ± .04 Å. This analysis and others is shown in the attached pre-print.(4)

A similar analysis of the tungsten bronze series of samples is in progress and will be published soon.

The work described above was very successful in determining parameters for the atoms in bulk samples. We wanted to investigate another way to obtain an additional site specificity with the technique, i.e., to measure the structure only around the conducting ion. By analogy with photon stimulated desorption and the Knotek-Feibelman(5) mechanism we suggested the following possibility. The fact that ion conduction is observed in these materials suggests that their bonds may be easily broken and they can move on to the next lattice site. During x-ray absorption a photoelectron plus a number of Auger electrons are ejected leaving the absorbing atom in a multiple charged state, which temporarily breaks its bonds to nearby atoms. We believe that this effect would perturb sufficient M^+ ions so that the cell current would be proportional to the x-ray absorption spectra of the M^+ ion. <u>This effect could provide an important site specific probe of the conducting ion</u>.

Rather than begin experiments with a complicated high temperature cell we modeled the effect with an aqueous Cs^+ Cl^- cell using Ag, AgCl electrodes, arguing that the effect should also perturb the hydration sphere, thereby increasing the conductivity of the cell. The arrangement and a preliminary result are shown in figure 3. The normal spectrum of Cs^+ in H_2O is shown by the dotted line with the cell current shown by the solid line as the x-ray energy was scanned through the Cs absorption edge. The delayed onset of the edge is expected since multiple ionization must be present for the effect to occur. The data is too noisy to be useful but the effect is there. We plan further experiments in this area in order to optimize our experimental parameters. If quality spectra can be obtained in this way the results will be important not only for fast ion conductors but for all of electrochemistry.

X-ray absorption spectroscopy is usually done by measuring the x-ray transmission through the sample or the fluorescent x-rays re-emitted from the surface of a thick sample. These are bulk techniques because x-rays penetrate deeply. However, it is well known that many electrons are emitted as a consequence of x-ray absorption and they arise from the near surface region. Kordesch and Hoffman(6) demonstrated a simple detector in which the sample was placed in a He atmosphere and the emitted electrons collected on a positively-biased grid. We designed and built a similar detector as shown in figure 4. The performance was beyond our expectations.(7) Signal levels were 10^2 - 10^3 above the fluorescent x-ray level. This is because more electrons are emitted than x-rays (e-yield vs fluorescent yield) plus the 2π collection geometry. The e-yield signal was successful in localizing the structure of the near surface as shown by the data in figure 5 which compares fluorescent x-ray (bulk) and e-yield (surface) spectra for anodized Ti, 6Al, 4V. The e-yield spectrum is that of TiO_2. The inset shows an enlargement of the V-edge also visible in the spectra. This spectrum is plainly not V metal. This data will be analyzed in detail and published.

As a member of the National Academy Panel on In Situ Characterization of Electrochemical Processes a report was prepared discussing the capabilities of x-ray techniques for problems of interest in electrochemistry. This paper may be of interest to the ONR-funded electrochemistry community and is appended to this report.

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6. M. E. Kordesch and R. W. Hoffman, Phys. Rev. B29, 491 (1984).
7. F. W. Lytle, R. B. Greegor, G. P. Huffman and F. E. Huggins, "Electron Yield EXAFS at Atmospheric Pressure", Proceedings 1985 SSRL Users Meeting.

Papers Published

1. J. Wong, F. W. Lytle, R. P. Messmer, and D. H. Maylotte, "K-edge Absorption Spectra of Selected V Compounds", Phys. Rev. B30, 5596 (1984).
2. F. W. Lytle, R. B. Greegor, G. P. Huffman, and F. E. Huggins, "Electron Yield EXAFS at Atmospheric Pressure", Proceedings 1985 SSRL Users Meeting.

Papers in Process

1. "Direct Determination of Valence and Local Structure in $M_XV_2O_5$ by X-ray Absorption Spectroscopy."
2. "An Analysis of the Structure of M_XWO_3 by X-ray Absorption Spectroscopy."
3. "Determination of the Structure of the Anodized Surface Layer of Ti, 6Al, 4V by Electron Yield EXAFS."

Papers Presented:

1. F. W. Lytle, "The Diverse Industrial Applications of EXAFS", 1983 SSRL Users Meeting.
2. F. W. Lytle, "Applications of Synchrotron Radiation to Materials Science", Invited Seminar at Los Alamos National Laboratory, Oct. 1984.
3. F. W. Lytle, "In Situ Characterization with X-ray Techniques", Workshop of National Academy of Science Panel on In Situ Characterization of Electrochemical Processes, Denver, Oct. 1985.
4. F. W. Lytle, "Electron Yield EXAFS at Atmospheric Pressure", SSRL Users Meeting, Stanford, Oct. 1985.

Funded Research Personnel: T. R. Beck, F. W. Lytle, R. B. Greegor, V. A. Biebesheimer, P. Viren

Other Collaborators: I. Raistrick, J. Wong, R. P. Messmer, D. H. Maylotte, G. P. Huffman, F. E. Huggins

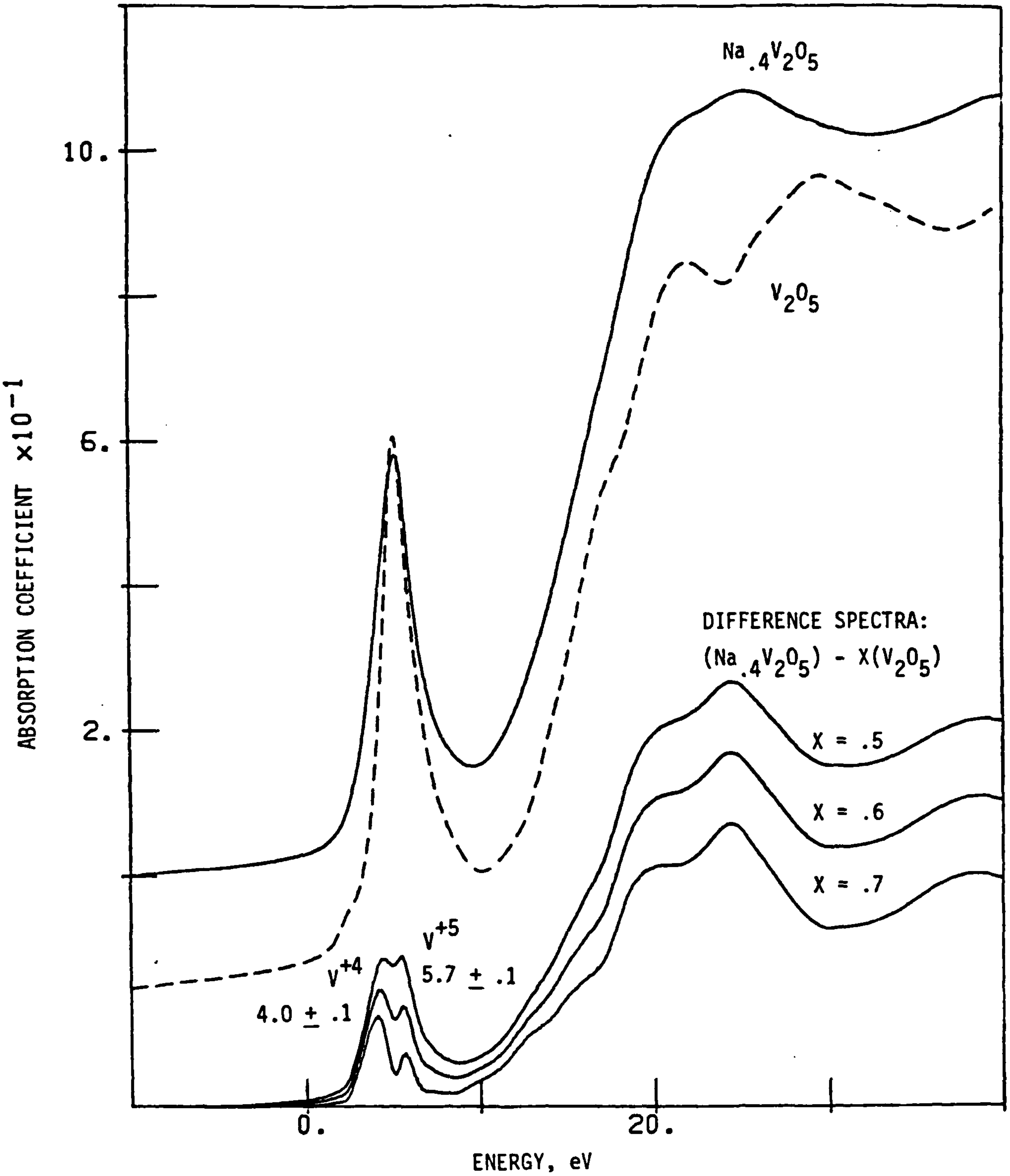

Fig. 1 Comparison of the K x-ray absorption edge of V in $Na_{.4}V_2O_5$ and V_2O_5 (shifted downwards). The three bottom curves illustrate uncovering of the V^{+4} pre-edge spectra obtained by subtracting away the V^{+5} spectrum, i. e. V_2O_5.

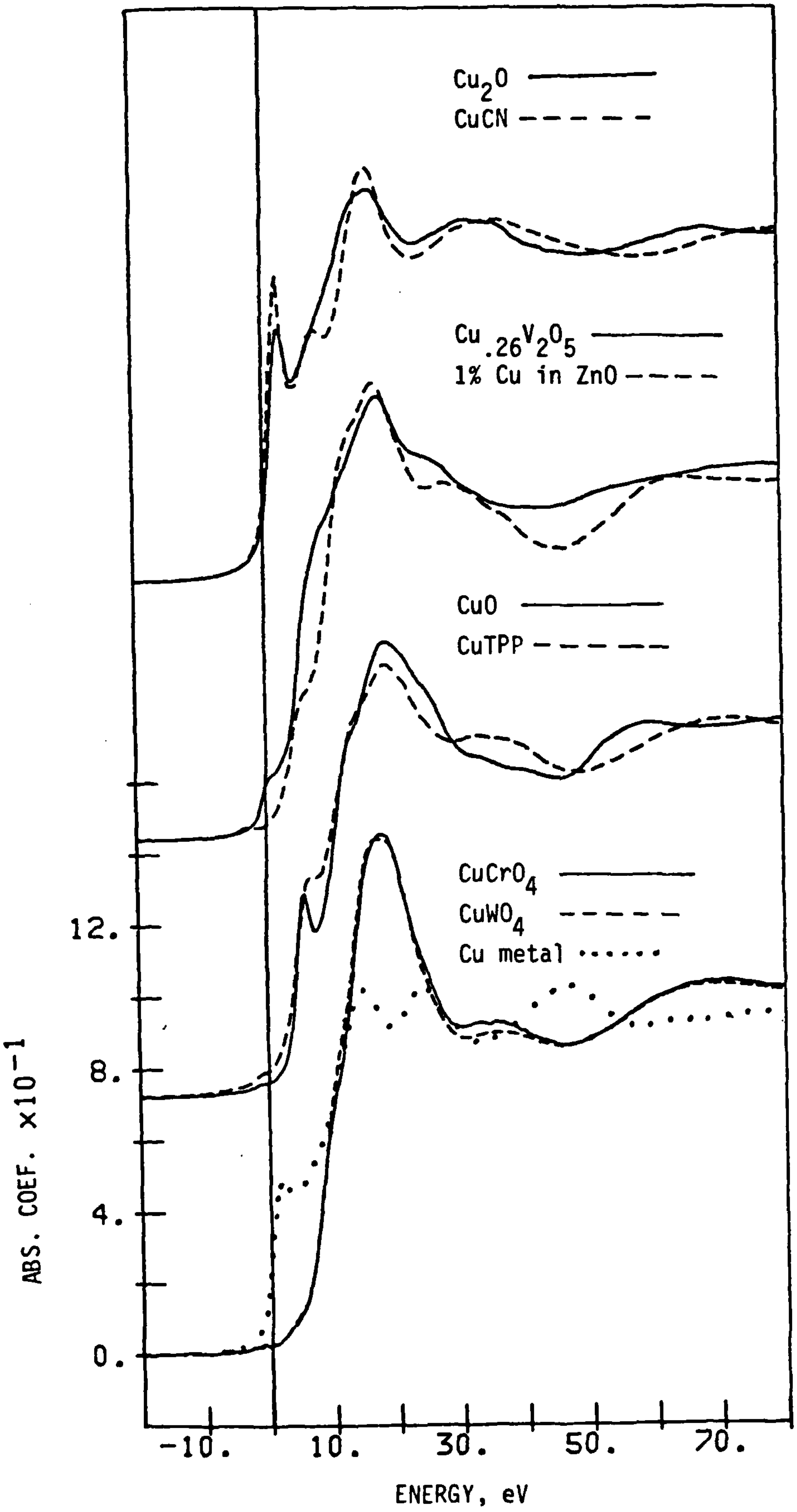

Fig. 2 Comparison of the Cu K-absorption edges of Cu$_{.26}$V$_2$O$_5$ with Cu reference compounds. Beginning at the top Cu in Cu$_2$O and CuCN is 2-fold coordinated, 1% Cu in ZnO is 4-fold (tetrahedral) coordinated, also 4-fold coordinated in CuO and Cu tetra phenyl porphyrin (square planar), and in CuCrO$_4$ and CuWO$_4$ it is in 6-fold (octahedral) coordination. The zero of energy is the first inflection point for Cu metal shown for comparison. The lack of the small pre-edge feature at ~ 2 eV common to all the Cu^{+2} compounds and the first absorption rise of Cu$_{.26}$V$_2$O$_5$ mark it as Cu^{+1}; however, the other absorption features are plainly different from any of the reference compounds.

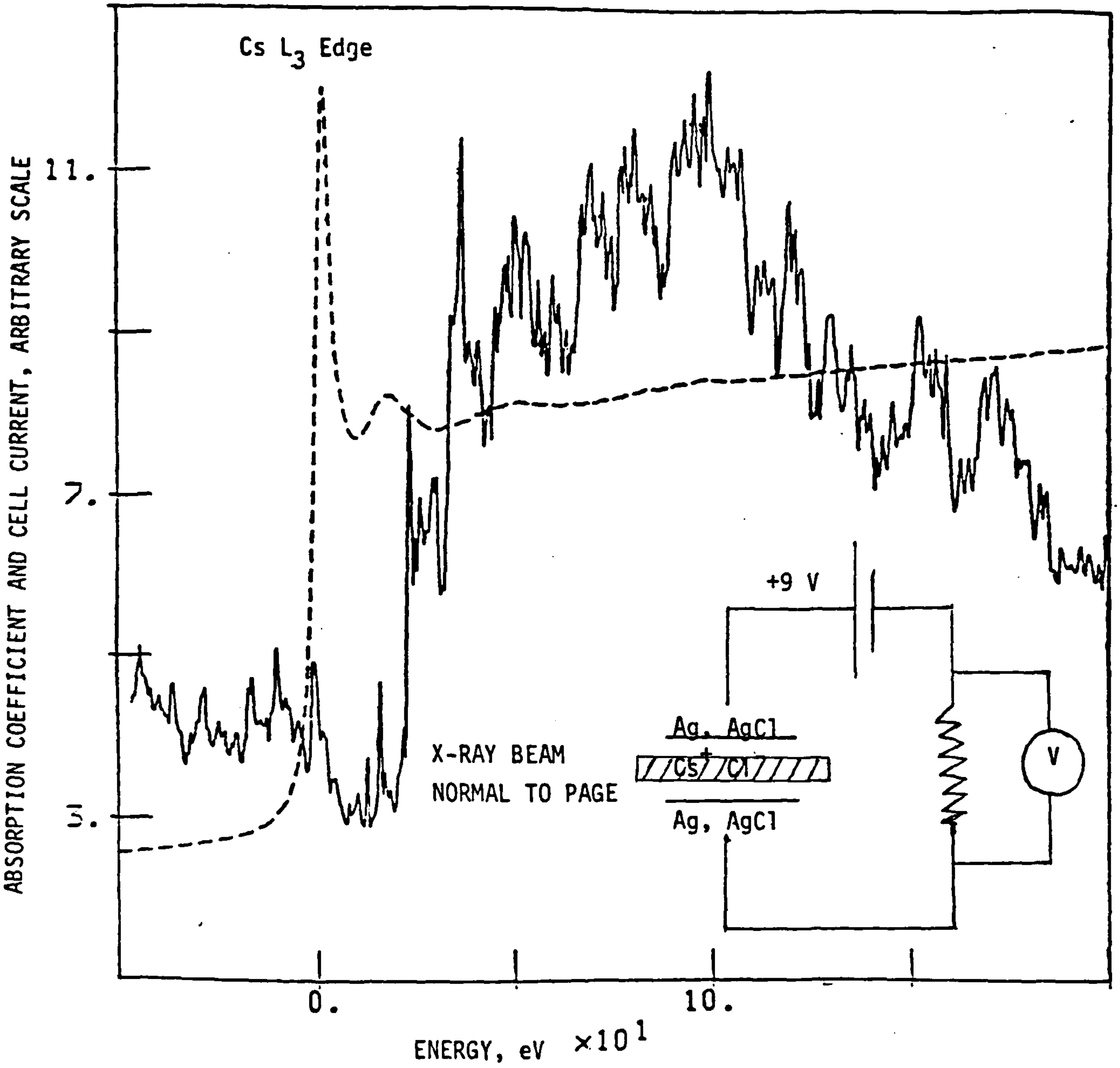

Fig. 3 Comparison of the current induced by x-ray absorption (solid line) with the fluorescent x-ray emission (dashed line) in a thin cell containing 1 N CsCl in H_2O. The image of the x-ray beam across the cell is indicated. The voltage across the dropping resistor increased with increasing x-ray absorption as shown.

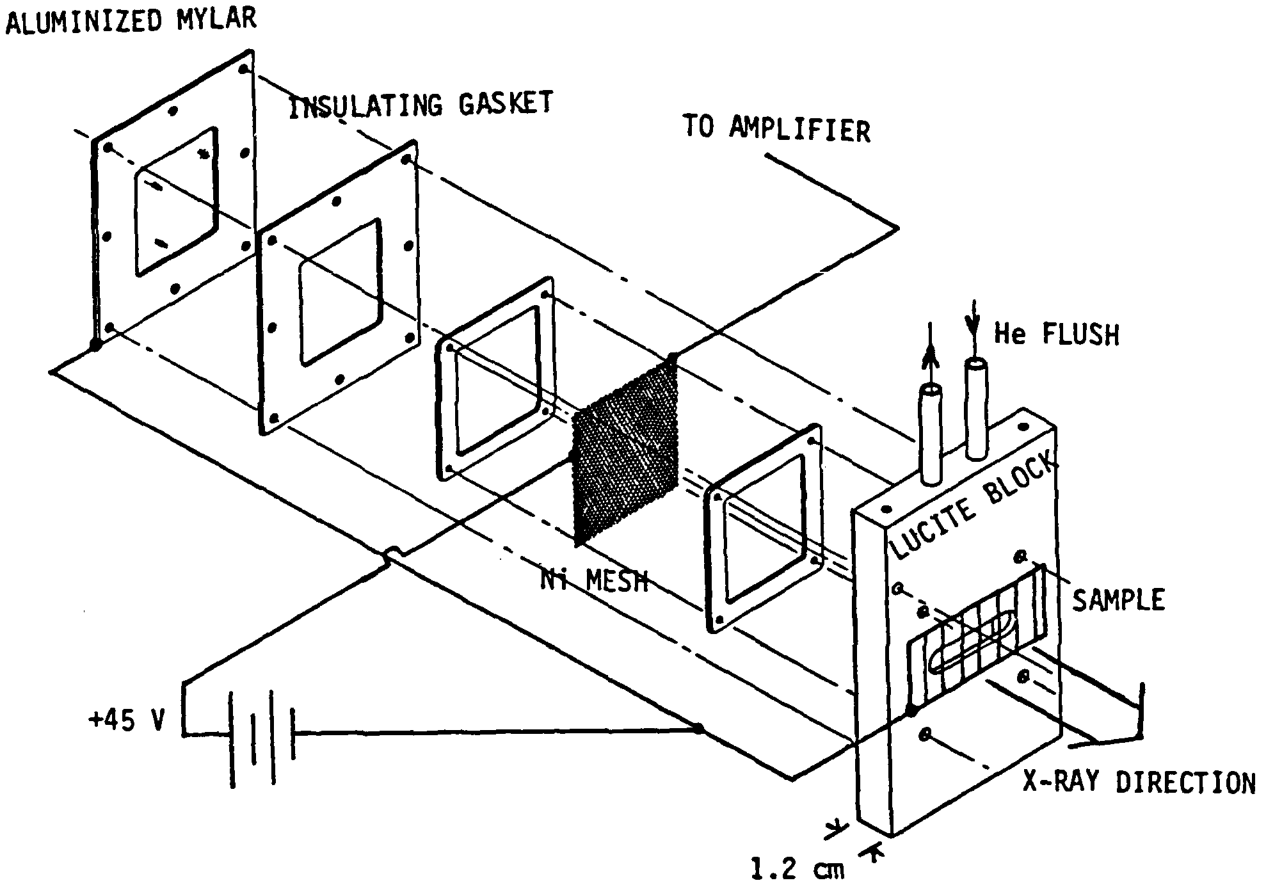

Fig. 4 Exploded view of electron yield detector.

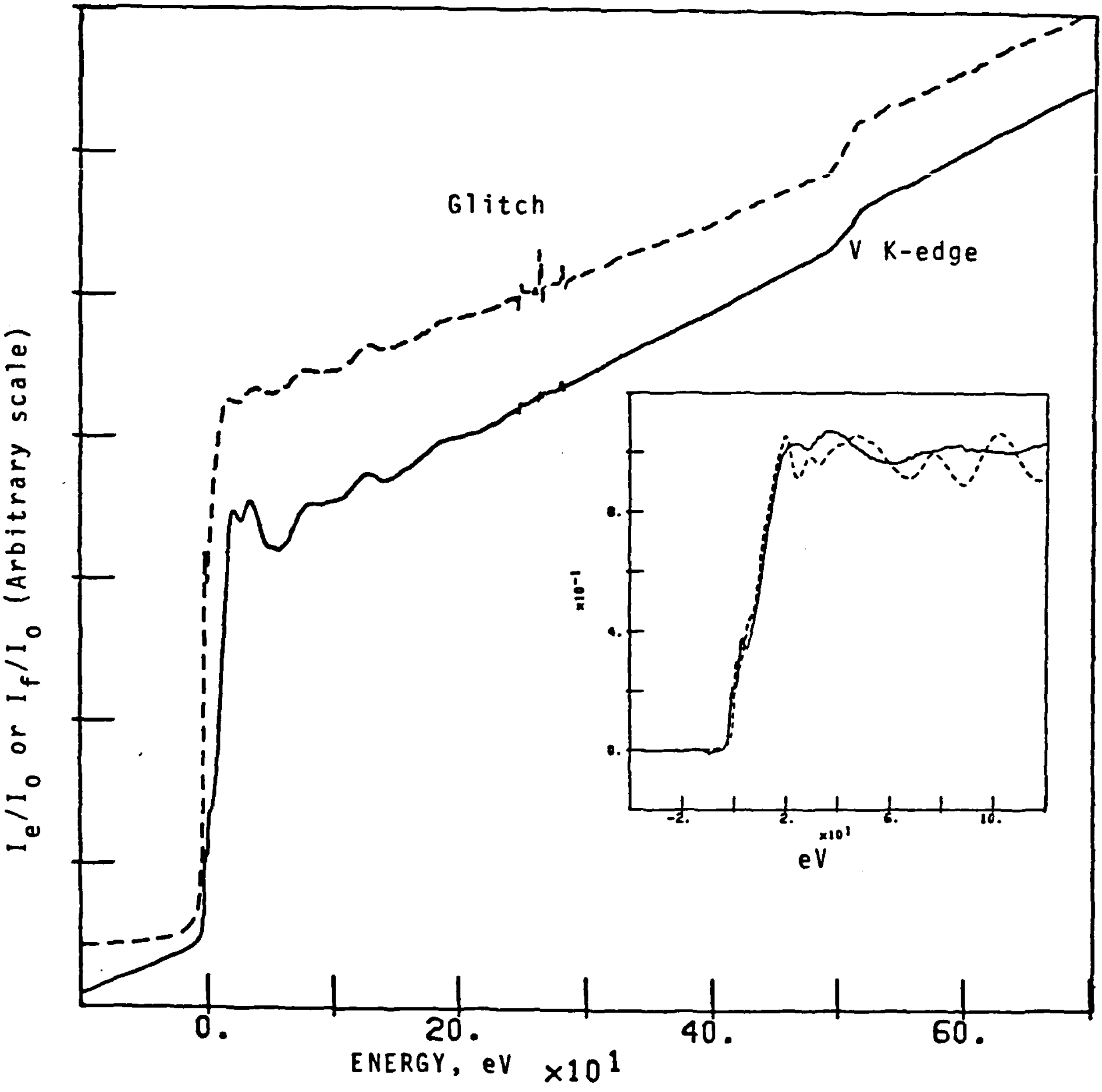

Figure 5 Fluorescent x-ray (dashed line) and e-yield (solid line) Ti K-edge spectra for anodized Ti-6Al-4V alloy. Monochromator glitches and the V K-edge are also apparent. The inset shows a scaled up view of the V e-yield spectrum (solid line) and a normal absorption mode metallic V K-edge spectrum (dashed line).

REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS
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1. REPORT NUMBER
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2. GOVT ACCESSION NO.

3. RECIPIENT'S CATALOG NUMBER

4. TITLE (and Subtitle)
ELECTRON YIELD EXAFS AT ATMOSPHERIC PRESSURE

5. TYPE OF REPORT & PERIOD COVERED
Technical report

6. PERFORMING ORG. REPORT NUMBER

7. AUTHOR(s)
F. W. Lytle, R. B. Greegor, The Boeing Co.
G. P. Huffman, F. E. Huggins, U. S. Steel Corp.
Monroeville, PA 15146

8. CONTRACT OR GRANT NUMBER(s)
N 00014-83-C-0620

9. PERFORMING ORGANIZATION NAME AND ADDRESS
The Boeing Co., Seattle WA 98124

10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS

11. CONTROLLING OFFICE NAME AND ADDRESS
Office of Naval Research
800 N. Quincy Street
Arlington, VA 22217

12. REPORT DATE
October 1985

13. NUMBER OF PAGES

14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)

15. SECURITY CLASS. (of this report)
UNCLASSIFIED

15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)
X-ray absorption spectroscopy, EXAFS, surface structure

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)
Extended abstract on following page.

Presented at and published in the proceedings of Oct. 1985 SSRL Users Meeting,
Stanford University, Stanford, CA 94305

ELECTRON YIELD EXAFS AT ATMOSPHERIC PRESSURE

F. W. Lytle and R. B. Greegor, The Boeing Co., Seattle, WA 98124
G. P. Huffman and F. E. Huggins,
U. S. Steel Corp., Monroeville, PA 15146

We used a He-flushed ion chamber containing the sample similar to that reported by Kordesch and Hoffman[1] to measure a variety of absorption spectra. The device was inserted in our fluorescent detector[2] so that simultaneous absorption, fluorescent and e-yield data could be taken. The technique was successful beyond our most optimistic expectations: 1) Signal level was 10^2 - 10^3 higher than for the fluorescent channel (2π collection efficiency plus e-yield versus fluorescent yield). 2) Data scans did not contain diffraction spikes from crystalline samples. 3) The amplitude of data from concentrated samples was not diminished by self-absorption (e.g., there is an enormous, sharp white line at the K-edge of elemental sulfur). 4) Excellent data from gas samples was obtained by mixing ~1% with the He flush. 5) The technique is sensitive only to the top 10 - 1000 Å. Disadvantages include: 1) The technique is sensitive only to the top 10 - 1000 Å. 2) Care must be taken to prevent the sample from charging or the data shows a continuous drift with discharge glitches. Results will be illustrated with S, Ar, Ti, Cu, Sn and Ta data.

The research of Lytle and Greegor was supported in part by grants from NSF and ONR. The research of Huffman and Huggins was supported by a grant from the Gas Research Institute.

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[1] M. E. Kordesch and R. W. Hoffman, Phys. Rev. B29, 491 (1984).

[2] F. W. Lytle, et al., Nucl. Inst. and Meth. 226, 542 (1984).

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER 3	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) X-RAY TECHNIQUES FOR IN SITU CHARACTERIZATION OF ELECTRO CHEMICAL PROCESSES		5. TYPE OF REPORT & PERIOD COVERED Technical report 6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) Farrel W. Lytle		8. CONTRACT OR GRANT NUMBER(s) N 00014-83-C-0620
9. PERFORMING ORGANIZATION NAME AND ADDRESS The Boeing Co., Seattle, WA 98124		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
11. CONTROLLING OFFICE NAME AND ADDRESS Office of Naval Research 800 N. Quincy Street Arlington, VA 22217		12. REPORT DATE November 1985 13. NUMBER OF PAGES
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		15. SECURITY CLASS. (of this report) UNCLASSIFIED 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

Report was prepared as input to the National Academy of Sciences "Panel on In Situ Characterization of Electro Chemical Processes."

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

X-ray techniques, structure determination at electrodes,

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

The concepts and techniques of x-ray absorption spectroscopy which may be useful for in situ characterization of electrodes are discussed. Many techniques appear to have unrealized potential for determing quantities of fundamental importance to electrochemistry.

X-RAY TECHNIQUES FOR IN SITU CHARACTERIZATION OF ELECTRO CHEMICAL PROCESSES

by

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Seattle, WA 98124

"Electronic and molecular structure is basic to understanding reactivity....", Henry Taube.

Understanding of the structure of chemical systems at the atomic level must come from probes of the structure with wavelengths of atomic dimensions, e.g., x-ray, electron and neutron diffraction techniques are well known. In addition certain indirect information at the atomic or molecular level may be obtained by various spectroscopies, spanning the electromagnetic spectrum. In this discussion x-ray techniques including variants of x-ray absorption spectroscopy (XAS) will be described which have unrealized potential for *in situ* atomic level characterization of chemical processes at electrodes. X-ray absorption near edge structure (XANES) from the region near the onset of the absorption edge has the usual capabilities for spectroscopic characterization, i.e., spin state, valence and local symmetry. Extended x-ray absorption fine structure (EXAFS) comprises the region above the absorption edge and is a modulation of the absorption coefficient extending 1000-2000 eV above the edge, caused by elastic scattering of ejected photoelectrons from neighboring atoms. Thus XAS involves the absorption of x-rays but the actual structure probe is the photoelectron where the source of electrons is the atom involved in the x-ray absorption event. This imparts element specificity because the x-ray absorption edges of different elements are separated in energy. Simply by choosing the energy range of measurement, the various elements present in a material may be characterized in turn. The structural information obtainable from EXAFS consists of local bond distances, coordination number, chemical identity of coordinating atoms and a measure of structural disorder. This is in addition to the spectroscopic information obtainable from XANES previously mentioned. As an example the L-absorption edges of Pt metal are shown in figure 1. The EXAFS wiggles are apparent on each edge. In the inset is shown the Fourier transform of the EXAFS from the L_3 edge. The coordination shells

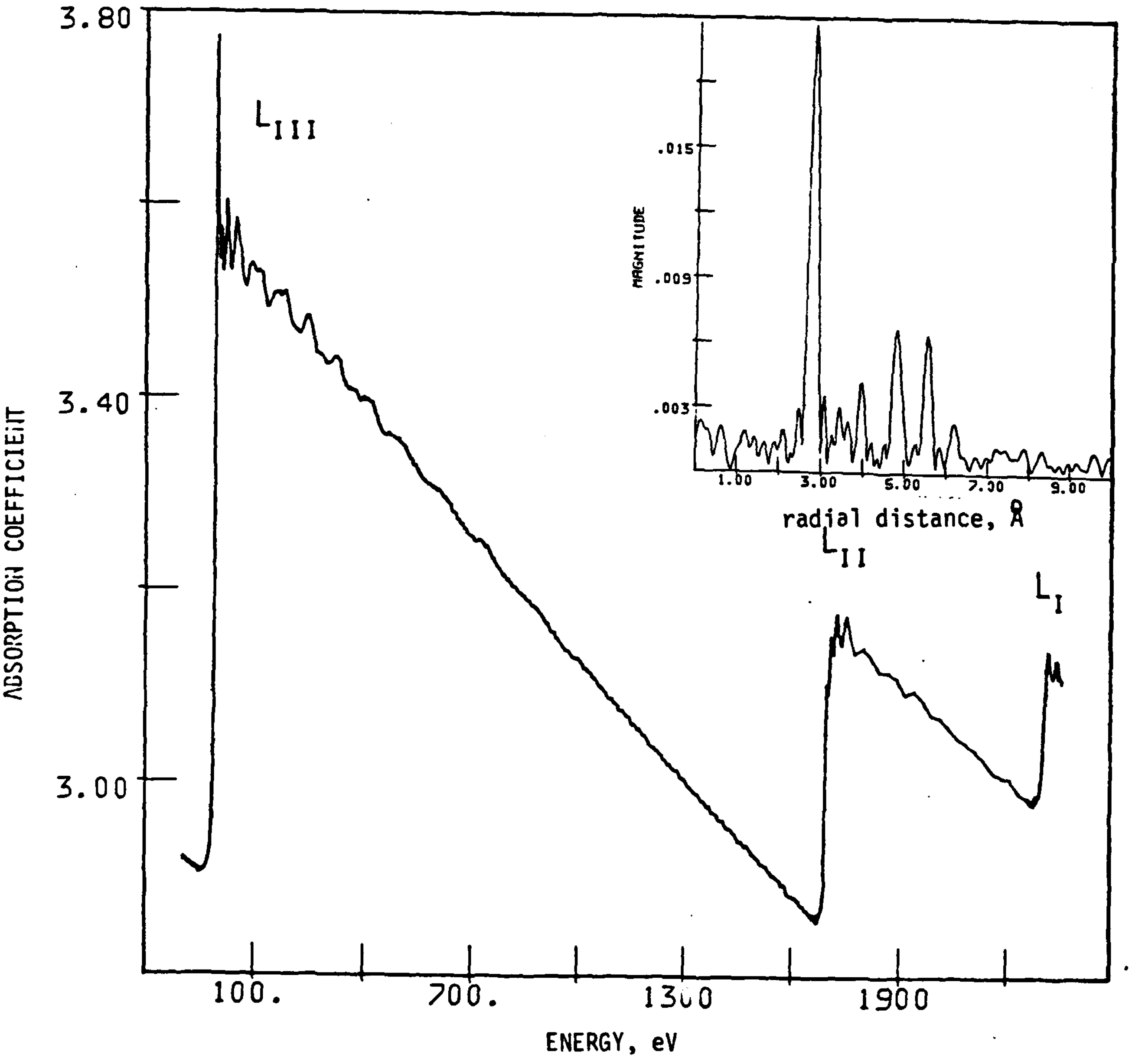

Fig. 1. The L-absorption edges of Pt metal. Zero of energy is Pt L_{III} at 11563.7eV. The inset is the Fourier transform (including phase shift) of the Pt L_{III} EXAFS. Bond distances are accurate for the first five coordination shells to ± .005 Å.

out to number 5 are clearly visible (and can be accurately measured); however, it is apparent that EXAFS is clearly a near neighbor phenomenon. Also note that the structure at the edge, XANES, is different for each edge. This is because the transitions arise from different core electrons; therefore, the favored dipole transition probes appropriate states above the Fermi level which are different in Pt for the electrons of different symmetries.

The fact that x-rays are penetrating is germane to the task of devising useful experimental techniques for in situ characterization. The calculated mean free path for x-ray absorption in water as a function of x-ray energy is shown in figure 2. Also indicated are the K-absorption energies of selected elements in order to place the graph in perspective with the periodic table. Since it is often possible to work through 2-4 mean free paths of extraneous absorbing media, considerable experimental flexibility is possible. Examples will be discussed below. Thus, XAS offers a number of unique advantages for in situ electro chemical characterization:

1. A penetrating x-ray beam to get inside of a material or experimental apparatus.
2. XAS can be combined with reflection or diffraction.
3. Once the x-ray beam is at the desired interface, the x-ray absorption event converts the structure probe to electrons which have a high cross section for interaction with nearby atoms.
4. The technique is element specific.

There is considerable experimental flexibility inherent with XAS techniques. This derives from combining XAS with x-ray diffraction or reflection and/or from detecting various products of the x-ray absorption event. The possibilities include fluorescent x-ray photons, optical photons, electrons, ions and photon desorbed atoms. All of which have different escape depths, thereby isolating the region of the sample characterized. The following is a description of x-ray techniques now in use and their approximate applications. Items 1-6 involve XAS with various geometries and detectors, items 7-8 combine XAS with x-ray diffraction and item 9 is pure x-ray diffraction.

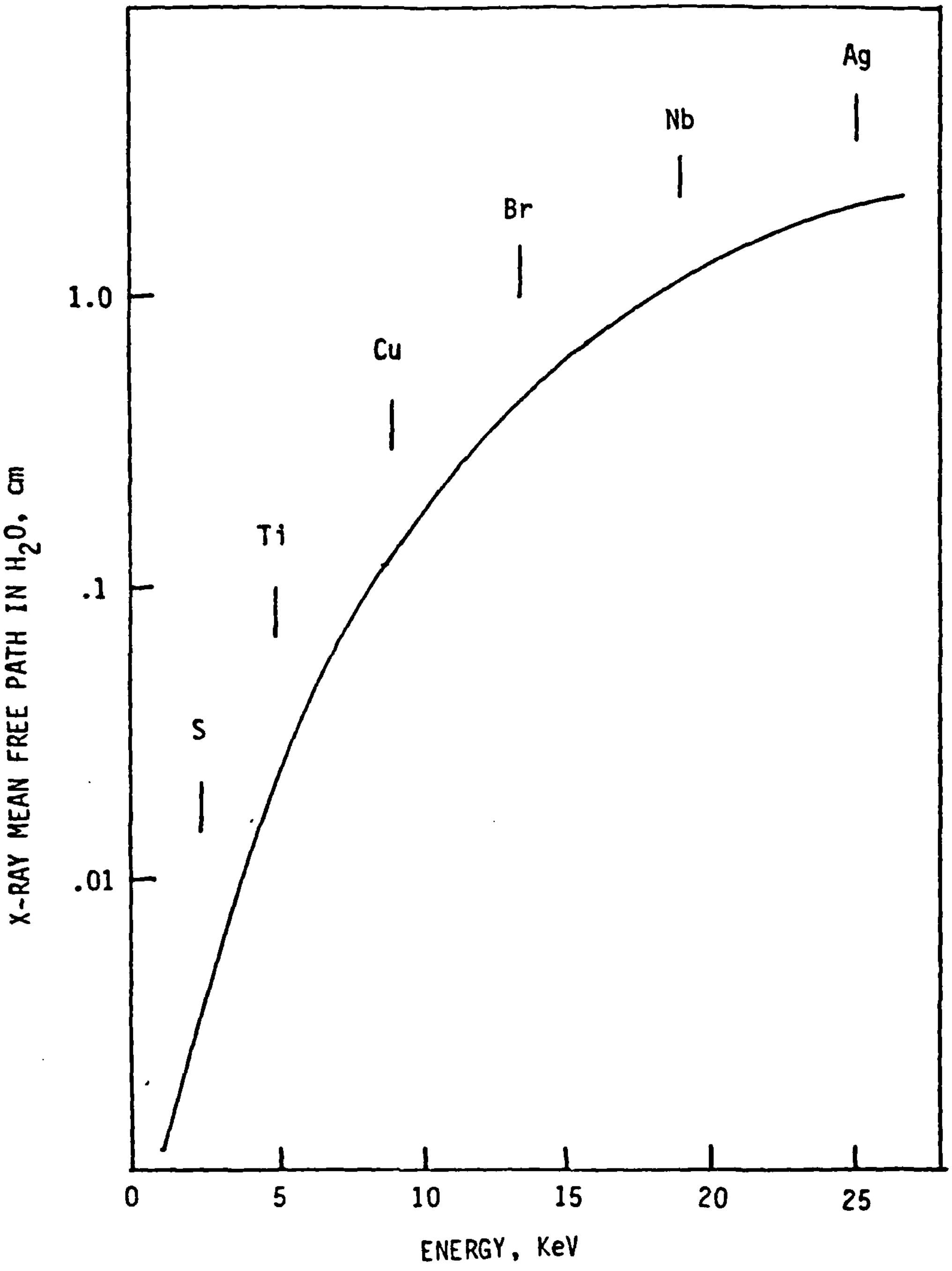

Fig. 2. Calculated mean free path of x-rays in water. The energy position of the K-edges of various elements is indicated.

1. Normal x-ray absorption, EXAFS; bulk characterization.
2. Fluorescent x-ray EXAFS; trace quantities, bulk.
3. Optical EXAFS; near surface, optical depth.
4. Electron yield EXAFS; surface to near surface.
5. Photon stimulated desorption EXAFS; surface species.
6. Reflection EXAFS; grazing incidence, surface 10-100 Å.
7. X-ray scattering with anomalous dispersion; atom specific, bulk.
8. Coupled beam x-ray diffraction; single crystal, surface species.
9. Reflection x-ray diffraction; top layer.

The previous discussion suggests that with carefully conceived experiments it is now possible to measure in situ structural parameters for an electrode surface and for the double layer in the contacting solution, to measure the concentration of each atomic species at the interface and to monitor the change in ion concentration $M \rightarrow M^+$ or $X \rightarrow X^-$. This could be done in real time and with full reversibility. In the future it should be possible to obtain atom-specific images at atomic level resolution of selected surfaces by using x-ray holographic techniques followed by reconstruction with optical lasers. X-ray holography already has been demonstrated with conventional sources. X-rays with a high degree of coherence will be available at very high intensity from undulators. The problems of beam splitters, mirrors, etc. for x-ray wavelengths will be difficult but not insurmountable. Wavelength tunability for atomic specificity, penetration to the desired region of the experiment and high, pulsed intensity for snapshots in time would make this the ultimate atomic level structure probe. Few of these techniques were even imagined 10 years ago. What will we find 10 years in the future?

All of the x-ray techniques which have been mentioned are possible only because of the availability of x-ray beams of unprecedented flux, stability and collimation from synchrotron sources. The progress in the science of electron storage rings and insertion devices has resulted in a new era, a renaissance in x-ray physics. The flux available is shown in perspective with other light sources in figure 3. For comparison, rotating anode x-ray sources are 5 orders of magnitude below the bottom of the graph. The synchrotron x-radiation has the following properties; high and stable intensity, broad spectral range, smooth and predictable shape, narrow vertical collimation, high degree of polarization and pulsed time structure. Sources are now

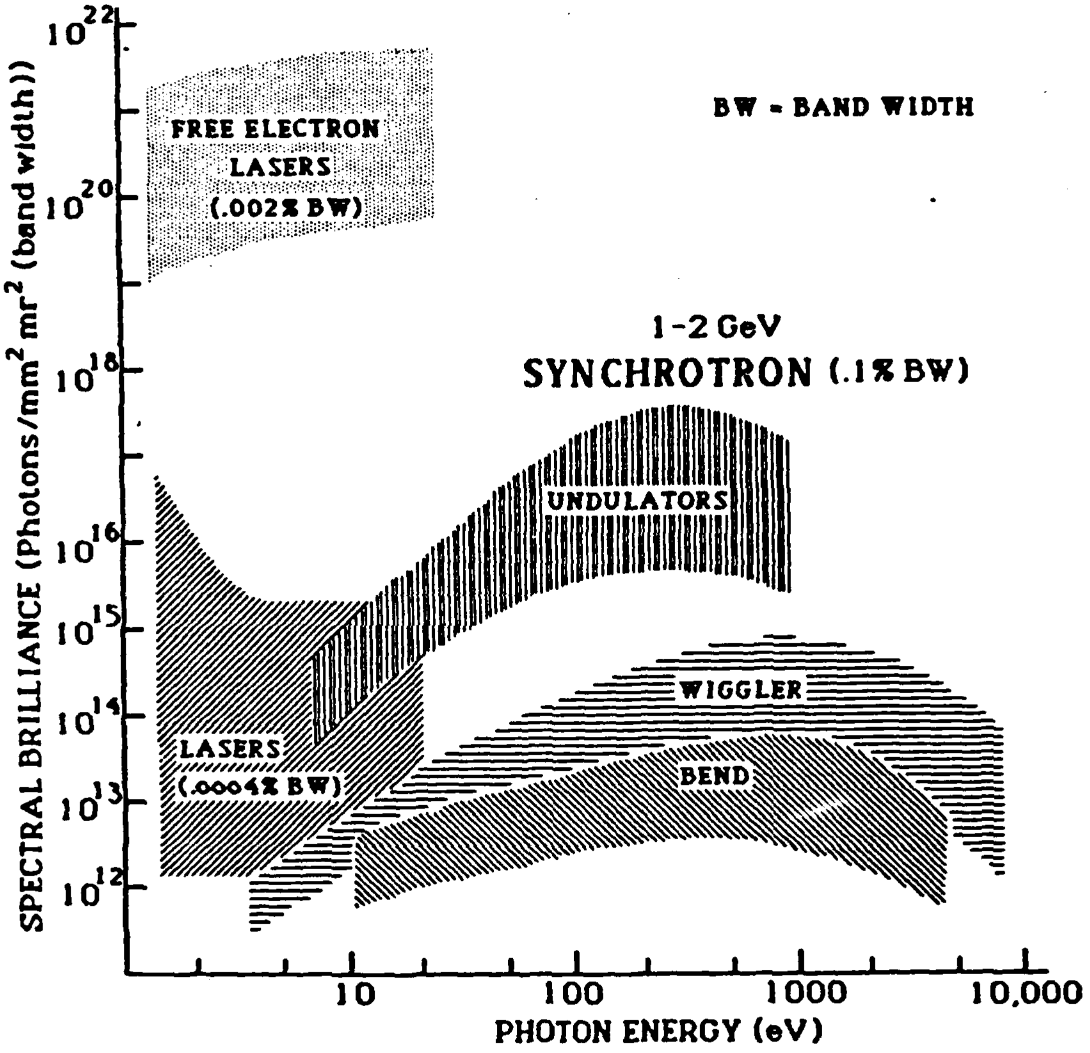

Fig. 3. Spectral brilliance compared for various photon sources. Note that the graphs for the various synchrotron radiation sources all may be extrapolated to the right by 10,000 eV for the projected 6 GeV ring.. (From "Opportunities in Chemistry", National Academy Press, 1985)

available in the United States at Stanford University, Cornell University, University of Wisconsin, and Brookhaven National Laboratories. Experimental time is awarded to qualified investigators without cost by reason of the scientific merit of their proposed research.

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