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22a. NAME OF RESPONSIBLE INDIVIDUAL D. R. Sandstrom			22b. TELEPHONE NUMBER (Include Area Code) (206) 773-2272		22c. OFFICE SYMBOL

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Project Title: Study of Chemical Systems in Static and Time
Resolved Mode by X-ray Absorption Spectroscopy

Principal Investigator: Donald R. Sandstrom

Institution: The Boeing Company, Seattle, WA 98124

Telephone: (206) 773-2272

DESCRIPTION OF PROJECT AND SIGNIFICANT RESULTS

The project objective was to develop new experimental x-ray absorption (XAS) techniques for application to studies in metal-containing coordination complexes in dynamic systems. Two new XAS approaches were advanced: (1) single pulse time resolved XAS (5,10) and 2) gas phase XAS (6-8). Our investigations of the time-resolved technique involved design of advanced circuitry for accurate single-pulse x-ray detection (10), definition of requirements for sufficiently accurate x-ray measurements and evaluation and understanding of time structure modulations and sources of noise in synchrotron radiation beams (5). Our progress resulted in a system with the potential for time resolved sensitivity for changes in chemical state and bond distance for transient

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chemical intermediates on a sub-microsecond time scale. Development of the gas phase XAS method expanded the potential range of the research on metal-containing complexes to metal clusters and gas phase complexes. This study could provide information on metal complexes in the absence of solvent interactions vis-a-vis our previous solution studies and to the growth of long range interactions in larger clusters. Initial studies were made on easily vaporized materials (8) and then in a high temperature, high flux vapor source where the evaporated metal flowed along the x-ray beam between the plates of an electron yield detector (6,7).

All the x-ray experiments were conducted at the Stanford Synchrotron Radiation Laboratory (SSRL) during three trips to the facility; however, during the past two years SSRL has not operated frequently or reliably and it was necessary to hold in abeyance the time resolved and gas phase experiments. With the limited beam time available we were able to complete studies which involved determination of the chemical state and environment of the catalytic atoms during reduction of a catalyst (2), determination of the chemical state and environment of rare earth ions substituted into high temperature superconductors (4) and a dynamic study of the formation of PdH_x and PdD_x (14).

Many of the experiments involved chemical systems exhibiting high structural and/or thermal disorder for which existing methods of treating the disorder became inadequate.

Improved models for incorporating atomic disorder were developed and incorporated into our data analysis programs (1). In our work with the L edges of the rare earth elements a significant advance was made in understanding the nature of the x-ray absorption near edge structure (XANES) which is primarily atomic in nature and how it joins to the extended x-ray absorption fine structure (EXAFS) (3). All of the publications, reports and presentations have been sent previously to ONR and described in our end of the year reports.

PUBLICATIONS

1. D. R. Sandstrom, E. C. Marques, V. A. Biebesheimer, F. W. Lytle and R. B. Greegor, "Thermal Disorder in Pt and Ir Metal: Comparison of Model and Experimental EXAFS", Phys. Rev. B32, 3541 (1985).
2. F. W. Lytle, R. B. Greegor and E. C. Marques, "Catalyst Research With X-ray Absorption Spectroscopy", Proceedings of the 9th International Congress on Catalysis, Vol. 5, Chemical Institute of Canada, 1989.
3. F. W. Lytle and R. B. Greegor, "Investigation of the 'Join' Between the Near Edge and Extended X-ray Absorption Fine Structure", Appl. Phys. Lett., to be published, Jan. 1990.
4. F. W. Lytle, G. van der Laan, R. B. Greegor, E. M. Larson and J. Wong, "Determination of the Valence of Pr, Gd and Ho, in $\text{YD}_{0.2}\text{Cu}_{0.7}\text{O}_7$ by X-ray Absorption Spectroscopy", Phys. Rev. B, accepted.

OTHER REPORTS

5. D. R. Sandstrom, V. A. Biebesheimer and F. W. Lytle, "Time Resolved X-ray Absorption Spectroscopy", SSRL Activity Report 86/01.
6. E. C. Marques, D. S. Olson, D. R. Sandstrom, F. W. Lytle and R. B. Greegor, "X-ray Absorption of Gas Phase Metal Clusters", Proceedings of 13th annual SSRL User's Conference, Oct. 1986.

7. E. C. Marques, D. R. Sandstrom, F. W. Lytle and R. B. Greegor, "XAS of Gas Phase Metal Containing Chemical Systems", SSRL Activity Report 87/01.
8. F. W. Lytle, R. B. Greegor, E. C. Marques and D. R. Sandstrom, "Investigation of Chlorine Compounds by X-ray Absorption Spectroscopy", SSRL Activity Report 87/01.
9. E. C. Marques, D. R. Sandstrom and F. W. Lytle, "M-M Structure in a New Type of Transition Metal Dimer by X-ray Absorption Spectroscopy", Proceedings 14th Annual SSRL User's Conference, Oct. 1987.
10. W. L. Brown and E. C. Marques, "Techniques for Digitizing and Storing Amplitude Levels of High Frequency Radiation Pulses", Boeing Document D180-31205-1, Sept. 1988.

PRESENTATIONS

11. E. C. Marques, D. S. Olson, D. R. Sandstrom, F. W. Lytle and R. B. Greegor, "X-ray Absorption of Gas Phase Metal Clusters", 13th Annual SSRL User's Conference, Oct. 1986.
12. E. C. Marques, D. R. Sandstrom and F. W. Lytle, "M-M Structure in a New Type of Transition Metal Dimer by X-ray Absorption Spectroscopy", 14th Annual SSRL User's Conference, Oct. 1987.
13. F. W. Lytle, R. B. Greegor and E. C. Marques, "Catalyst Research With X-ray Absorption Spectroscopy", Plenary Address, 9th International Congress on Catalysis, Calgary, 1988.
14. R. Frahm, G. Meitzner, R. B. Greegor and F. W. Lytle, "In Situ Investigation of the Electrochemical Formation of PdH_x and PdD_x", presented at Workshop on Cold Fusion Phenomena, Santa Fe, 1989.

FUNDED RESEARCH PERSONNEL

V. A. Biebescheimer, R. B. Greegor, F. W. Lytle, E. C. Marques, D. S. Olson and D. R. Sandstrom

STUDENTS

V. A. Biebescheimer and D. S. Olson

OTHER COLLABORATORS

W. L. Brown, R. Frahm, G. van der Laan, E. M. Larson,
G. Meitzner and J. Wong