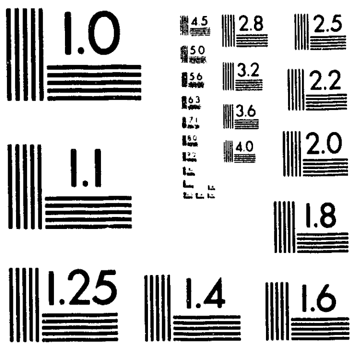
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DOE/ER/45162--T1

Final Performance Report for Contract DOE-FG02-84ER45162

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1 Introduction

This report summarizes the work performed under contract DOE-FG02-84ER45162. During the past ten years, our study of electron emission from laser-illuminated field emission tips has taken on a broader scope by addressing problems of direct interest to those concerned with the unique physical and chemical properties of nanometer-size clusters. The work performed has demonstrated that much needed data can be obtained on individual nanometer-size clusters supported on a wide-variety of different substrates. The work was performed in collaboration with R.P. Andres in the School ofd Chemical Engineering at Purdue University. The Multiple Expansion Cluster Source developed by Andres and his students was essential for producing the nanometer-size clusters studied. The following rereport features a discussion of these results.

The following report provides a motivation for studying the properties of nanometer-size clusters and summarizes the results obtained. The level of graduate student participation is indicated and the published papers and talks related to the DOE funded work are enumerated. Copies of published work are also included under Appendix A.

2 Background

The study of nanometer-size clusters has received increased attention from a wide variety of different research disciplines. The resulting multi-disciplinary effort has blended a healthy mixture of physics, chemistry and materials science focussed on the goal of better understanding the unique properties possessed by aggregates or clusters of atoms.[1] It is now known that the finite size of clusters leads to new atomic packing geometries not realized in the bulk state. These new geometries in turn give rise to size-dependent electronic, chemical, and thermodynamic properties that are of considerable interest to a wide variety of disciplines. Although no practical solid state devices have yet been fabricated from nanometer-size clusters, the study of such clusters now will provide important information about their electronic and thermodynamic properties far in advance of the time that nano-structures will be required by the electronics industry. The potential for optical, electronic, and magnetic applications is a prime reason why an increasing emphasis on the properties of 'supported clusters' and 'cluster-assembled materials' is emerging.[2-4] This trend is evident from recent topical cluster conferences that have been organized within the past few years.[5-7]

The study of supported clusters and cluster-assembled materials rests on a foundation of results obtained from prior molecular beam studies of small, gas-phase clusters which have elucidated a number of size-dependent properties. Research on free-space clusters has clearly established that in the nanometer size range, clusters exhibit unique, size-dependent structural, chemical and electronic features different from bulk samples of the same atoms.[8-11]

The emergence of new techniques, sensitive to the properties of supported clusters and not readily available some ten years ago,[9] has cleared the way for supported cluster studies. As examples, the known surface sensitivity of synchrotron photoemission has been exploited to study the properties of thin cluster films deposited on substrates.[12,13] High resolution transmission electron microscopy has also been used to study the geometry of supported clusters.[14-17] Even more dramatic has been the use of the scanning tunneling microscope,[18-24] and more recently the atomic force microscope,[25] to study the properties of individual supported clusters. Unfortunately, the STM results on metallic clusters have not been as exciting as initial expectations, due partly to the fact that individual atoms are not usually resolved. Nonetheless these newer spectroscopies and scanning probe techniques are providing useful information on supported clusters that can not be obtained from well-established cluster-beam measurements like mass-abundance,[26-28] electron affinity,[29] ionization potential,[30] gas-phase photoelectron spectroscopy,[31-36] and gas-phase reactivity.[37-43]

It seems clear that a knowledge of the properties of individual supported clusters provides insight into important physical processes and will be useful for a better understanding of nanophase materials synthesized from clusters.[3,4] The challenge for the experimentalist is to develop new and reliable tools to better probe the properties of an individual supported cluster. The emerging emphasis on the study of an individual cluster is fast becoming a prerequisite for experiments on supported clusters, since studies of cluster-assembled materials are often complicated by a distribution of cluster sizes which smears out any size effects under investigation. Our research supported by DOE has adressed this crucial problem head-on and has produced some much needed data on the properties of individual supported nanometer-size clusters.

A goal that formed the central theme of our research was to measure the size-dependent properties of a cluster after it is deposited onto a surface. A prerequisite for continued progress is the development of new experimental techniques sensitive to the properties of an *individual* supported cluster. As summarized below, a wide variety of results have been obtained on the electronic and structural properties of nanometer-size clusters under DOE support. Examples include the melting temperature as a function of cluster size,[44-46] the shape of supported clusters on flat substrates,[24,47] and the size-dependent electronic structure of individual supported clusters.[48-51] Most of this prior work has been focussed on Au clusters because of their weak interaction with adsorbed gases. Unfortunately, continued DOE funding was not available to further extend these studies to other systems of interest.

3 Summary of Results on Supported Nanometer-size Clusters

3.1 Size-Dependent Melting

We have obtained a number of interesting results by applying our expertise in areas related to field emission and photo-field emission[52-59] to questions related to the nanometer-size cluster problem. In this section, we summarize the more important results obtained on the size-dependent melting transition.

The details of how a melting transition in a solid occurs has been a topic of long-term interest. Early theories of melting have focussed on the role of vibrational instabilities,[60,61] lattice-shear instabilities,[62] catastrophic generation of dislocations,[63] or the presence of thermal vacancies and other point defects.[64] The dominant role played by an external surface in the melting transition has also recently been emphasized.[65-72] Clearly, interesting effects are expected in materials with large surface to volume ratios. From molecular-dynamic studies, there is considerable evidence that melting is initiated at external surfaces or at internal surfaces such as grain boundaries or dislocations. Computer simulations of melting have nicely demonstrated the influence of these features on the melting transition.[73,74] As cluster size decreases, the number of surface atoms increases and any role played by the surface in the melting transition will be accentuated. It may not be surprising, therefore, that small Au clusters in the nanometer-size range show a melting point depression.[75]

The study of melting in small nanometer-size clusters is also relevant to a better understanding of the melting transition in general[76] and the issue of size dependent melting of small clusters has therefore received increasing theoretical attention in the last few years.[77-82] It will continue to be a topic of considerable interest because it encompasses a number of important issues. The melting depression in small metallic clusters is also of considerable technological importance.[83,84] For these reasons, any systematic data on this melting transition should be clearly welcomed.

We have already demonstrated the feasibility of using the field emission current from an individual cluster to measure the melting temperature of that cluster. To do this, we have shown[85,44,45] that *individual* nanometer-size clusters can be reliably soft-landed on the apex of sharp field emission tips in a controlled way. This new technique has the flexibility of studying a single isolated cluster over a wide range of sizes (~ 1.0 - 20 nm in diameter). Moreover, the technique appears to be as sensitive to small clusters as to large ones.

3.2 Size-Dependent Electronic Structure

The question whether the quantized electronic states of a free space cluster survive the deposition process is an important issue with vast implications. It has been difficult to address this question experimentally. Recently, however, structure in the energy distribution of electrons emitted from a 1 nm Au cluster supported on a W tip has been successfully measured at Purdue.[50] A resonant tunneling model to explain this data in terms of the cluster's quantized electronic structure has been proposed.[48] The observed emission spectra provided good evidence that the quantized electronic states in a 1 nm diameter Au cluster can survive at room temperature when the cluster is supported on a W surface. A resonant-tunneling model based on first-order perturbation of the supported cluster by the applied electric field was used to provide an estimate for the position in energy of the peaks observed in the field emission spectrum.[48,50] A reasonable account for the experimental observations was obtained and a tentative identification of the cluster shell-levels responsible for the observed structure was proposed.

This novel field emission technique has been further exploited to probe the electronic structure of supported Au clusters in the 1 nm to 3 nm size range and the evolution of the size-dependent electronic structure of individual, supported metal clusters has been studied.[49] A number of interesting observations can be made from the data. The electron energy distribution shifts toward the substrate E_F with increasing cluster size, with all detectable structure disappearing for clusters with diameters greater than ~3 nm. The position in energy of the peaks in the electron energy distribution from an individual cluster were reproducible over a period of time spanning a few weeks. The relative height of the peaks changed when measured on a time scale of a few hours, a fact attributed to variation in the coupling of the cluster states to the substrate.[48]

4 Graduate Student Participation

A number of graduate students have contributed to the research discussed above. These students are listed below. Those supported by DOE research funds are marked with an asterisk.

Chemical Engineering Graduate Students

Seung-Bin Park (PhD in 1988)

Eugene Choi (PhD in 1989)

Amar Ramachandra (PhD in 1992)

Atul Patil (PhD candidate)

Physics Graduate Students

Stacy Mogren (*) (PhD in 1989)

Tom Castro(*) (PhD in 1989)

Yun Zhong Li(*) (PhD in 1989)

Mong-ea Lin(*) (PhD in 1991)

David Schaefer (PhD candidate)

5 DOE Related Publications

"Thermionic and Threshold Photoemission Study of $LaB_6(110)$", S. Mogren and R. Reifenberger, Surf. Sci. **186**, 232-46 (1987).

"A Retrievable UHV Deposition Source for Investigation of Low Melting Point Metals on Solid Surfaces", T. Castro and R. Reifenberger, Rev. Sci. Instrum. **58**, 289-92 (1987).

"Direct Imaging of 13 Å-diam Au Clusters Using Scanning Tunneling Microscopy", A.M. Baro, A. Bartolome, L. Vazquez, N. Garcia, R. Reifenberger, E. Choi, and R.P. Andres, Appl. Phys. Lett. **51**, 1594-6 (1987).

"Studies of Individual Nanometer-Sized Metallic Clusters Using the Scanning Tunneling Microscope, Field Emission and Field-ion Microscopy", T. Castro, Y.Z. Li, R. Reifenberger, E. Choi, S.B. Park, and R.P. Andres, J. Vac. Sci. Technol. **A7**, 2845- (1989).

"Writing Nanometer-Scale Features in Gold Using the Scanning Tunneling Microscope", Y.Z. Li, L. Vazquez, R. Piner, R.P. Andres and R. Reifenberger, Appl. Phys. Lett. **54**, 1424-26 (1989).

"Computer Control of the Tunnel Barrier Width for the Scanning Tunneling Microscope", R. Piner and R. Reifenberger, Rev. Sci. Instrum. **60**, 3123-7 (1989).

"A Field Emission Technique to Measure the Melting Temperature of Individual Nanometer-Sized Clusters", T. Castro, R. Reifenberger, E. Choi, and R.P. Andres, Surf. Sci. **234**, 43-52 (1990).

"Size Dependent Melting Temperature of Individual Nanometer-Sized Metallic Clusters", T. Castro, R. Reifenberger, E. Choi, and R.P. Andres, Phys. Rev. **B42**, 8548-56 (1990).

"STM and FEM Studies of Individual Supported Metal Clusters", T. Castro, Y.Z. Li, R. Reifenberger, E. Choi, S.B. Park, and R.P. Andres, *Novel Materials in Heterogeneous Catalysis*, R.T.K. Baker and L.L. Murrell, Eds., American Chemical Society Symposium Series No. 437, American Chemical Society, Washington D.C., pgs. 329-41 (1990).

"Thermodynamic and Structural Properties of Individual Nanometer-Size Supported Metallic Clusters", T. Castro, E. Choi, Y.Z. Li, R.P. Andres and R. Reifenberger, Mat. Res. Soc. Symp. Proc. **206**, 159- , (1991)

"Field Emission and Photofield Emission from LaB_6, S. Mogren and R. Reifenberger, Surf. Sci. **254**, 169-81 (1991).

Shape of Nanometer-Size Supported Gold Clusters Studied Using the Scanning Tunneling Microscope", Y.Z. Li, R. Reifenberger, E. Choi, and R.P. Andres, Surf. Sci. **250**, 1-7 (1991).

"Observation of the Discrete Electron Energy States of an Individual Nanometer-Size Supported Gold Cluster", M.E. Lin, R.P. Andres, R. Reifenberger, Phys. Rev. Lett. **67**, 477-80, (1991).

"Size-dependent Thermodynamic and Electronic Properties of Individual Nanometer-size Supported Gold Clusters", M.E. Lin, A. Ramachandra, R.P. Andres, and R. Reifenberger, in **Physics and Chemistry of Finite System; Vol. I**, Eds. P. Jena, S.N. Khanna, and B.K. Rao, Kluwer Academic Publishers, Boston, 1992; pgs. 309-22.

"Improved Method for Fractal Analysis Using Scanning Probe Microscopy", S. Miller and R. Reifenberger, J. Vac. Sci. Technol. **B10**, 1203-7 (1992).

"The Field Emission Spectrum of a Nanometer-size Supported Gold Cluster: Theory and Experiment", M.E. Lin, R. Reifenberger, R.P. Andres, Phys. Rev. **B46**, 15490-7 (1992).

"Size-dependent Field Emission Spectra from Nanometer-size Supported Gold Clusters", M.E. Lin, R. Reifenberger, A. Ramachandra, and R.P. Andres, Phys. Rev. **B46**, 15498-502 (1992).

"Electron Emission from an Individual, Supported C_{60}", M.E. Lin, R.P. Andres, R. Reifneberger, and D.R. Huffman, Phys. Rev. **B47**, 7546-59 (1993).

"Electron Emission from Nanometer-size Metallic CLustrers: Electronic States and Structural Stability of Supported Au Clusters", M.E. Lin, A. Ramachandra, R.P. Andres, R. Reifenberger, Proceedings of the NATO Advanced Research Workshop on Nanosources and Manipulation of Atoms under High Fields and Temperatures, Lyon, France (in press).

6 DOE Related Talks

A Scanning Tunneling Microscope Study of Au Clusters, R. Reifenberger, A.M. Baro, N. Garcia, L. Vazquez, A. Bartolome, R.P. Andres, and E. Choi, presented at the Scanning Electron Microscopy '87 Conference;

Hamilton Ontario, Canada; May 4, 1987; presented by R. Reifenberger (30 min.); **Invited**.

Scanning Tunneling Microscope Studies of Nanometer-Sized Au and Ag Clusters, Y.Z. Li, E. Choi, R.P. Andres and R. Reifenberger, March Meeting of the American Physical Society, New Orleans LA; March 21-25. 1988; presented by Y.Z. Li (10 minutes).

Field Emission and Field-ion Microscopy Studies of 10-20 Å Diameter Au Clusters, T. Castro, E. Choi, R.P. Andres and R. Reifenberger, March Meeting of the American Physical Society, New Orleans LA; March 21-25, 1988; presented by T. Castro (10 minutes).

Studies of Individual Nanometer-Sized Gold Clusters, T. Castro, Y.Z. Li, R. Reifenberger, E. Choi, S. B. Park, and R.P. Andres, American Vacuum Society, Illinois Chapter 6th Annual Fall Symposium, Argonne National Laboratory, Chicago IL; September 8 1988; presented by T. Castro (20 minutes).

Scanning Tunneling Microscope and Field-ion Microscope Studies of Nanometer-Sized Clusters, Y.Z. Li, T. Castro, E. Choi, R.P. Andres, and R. Reifenberger, American Vacuum Society 35th National Symposium - Topical Conference "Probing the Nanometer Scale Properties of Surfaces/Interfaces", Atlanta GA; October 4 1988; presented by R. Reifenberger (20 minutes).

Electronic Properties of Individual Nanometer-Size Gold Clusters, Y.Z. Li, T. Castro, R. Reifenberger, E. Choi, S.B. Park, and R.P. Andres, 36th Midwest Solid State Conference, W. Lafayette IN; October 10-11, 1988; presented by Y.Z. Li (15 minutes).

Electronic Properties of Individual Nanometer-Size Gold Clusters, R. Reifenberger, Illinois Institute of Technology, Chicago ILL; November 16 1988; (1 hour).

Size Effect in the Melting of Au Clusters, T. Castro, E. Choi, S.B. Park, R.P. Andres, R. Reifenberger, March Meeting of the American Physical Society; St. Louis MO; March 20-24, 1989. Presented by T. Castro (10 minutes).

Writing Nanometer-Scale Features in Gold Using the Scanning Tunneling Microscope, R.P. Andres, Y.Z. Li, R. Reifenberger, March Meeting of the American Physical Society; St. Louis MO; March 20-24, 1989. Presented by R.P. Andres (10 minutes).

Electronic Properties of Au Clusters Using Scanning Tunneling Microscopy and Spectroscopy, Y.Z. Li, E. Choi, S.B. Park, R.P. Andres, R. Reifenberger, March Meeting of the American Physical Society; St. Louis MO; March 20-24, 1989. Presented by Y.Z. Li (10 minutes).

Nanometer Scale Writing in Gold with a Scanning Tunneling Microscope, Y.Z. Li, R.P. Andres, and R. Reifenberger; STM Spring Workshop, Laboratory for Research on the Structure of Matter, University of Pennsylvania, Philadelphia PA, May 15-16 1989. Presented by R. Reifenberger (20 minutes).

Writing Nanometer-sised Symbols with the Scanning Tunneling Microscope, R. Reifenberger, Seminar in the Solid-State Seminar Series, Department of Electrical Engineering, Purdue University, W. Lafayette IN, Sept. 27 1989 (1 hour).

Structure and Properties of Individual Nanometer-sized Metallic Clusters, R. Reifenberger, ASM/TMS Conference, Indianapolis IN, October 4, 1989 (30 mins). **Invited.**

Size-dependent Shape and Melting Temperature of Supported Nanometer-size Gold Clusters, T. Castro, Y.Z. Li, E. Choi, R.P. Andres, and R. Reifenberger, presented by R. Reifenberger at the 36th American Vacuum Society Conference, Boston MA, October 23-27, 1989, (20 mins).

Size-dependent Properties of Individual Nanometer-size Clusters, T. Castro, E. Choi, Y.Z. Li, R.P. Andres, and R. Reifenberger, presented by R. Reifenberger at the March Meeting of the American Physical Society, Los Angeles CA, March 12-16, 1990 (10 mins).

Size-Dependent Properties of Individual Nanometer-Size Supported Clusters, R. Reifenberger, presented by R. Reifenberger; Kodak Research Laboratory Invited Lecture Program, Rochester N.Y.; Sept. 14, 1990 (1 hour).

Size-dependent Properties of Individual Nanometer-size Supported Clusters, E. Choi, R.P. Andres, T. Castro, Y.Z. Li, and R. Reifenberger, presented by R. Reifenberger at the 1990 Meeting of the Materials Research Society, Boston MA, November 26- December 1, 1990, (30 mins). **Invited.**

Observation of Discrete Electronic States from an Individual Nanometer-Size Supported Au Cluster, M.E. Lin, A. Ramachandra, R.P. Andres, and R. Reifenberger, presented by M.E. Lin at the 1991 March Meeting of the American Physical Society, Cincinnati OH, March 18-22, 1991 (10 mins).

New Characterization Techniques to Probe Individual Supported Nanometer-Size Metallic Clusters, R. Reifenberger, 1991 March Meeting of the American Physical Society, Cincinnati OH, March 18-22, 1991 (30 mins) **Invited**.

Field and Photo-field Emission from Sharp Tips, R. Reifenberger, presented at the Amoco Research Center, Naperville IL; April 16, 1991 (1 hour).

Thermodynamic, Structural and Electronic Properties of Individual Nanometer-Size Clusters, R.P. Andres and R. Reifenberger, presented by R. Reifenberger at the International Symposium on the Physics and Chemistry of Finite Systems: From Clusters to Crystals, Richmond VA, Oct. 8-12, 1991 (30 minutes) **Invited**.

Observation of Single Electron Tunneling at Room Temperature, M.E. Lin, R.P. Andres, and R. Reifenberger, presented by M.E. Lin at the 1992 March Meeting of the American Physical Society, Indianapolis IN, March 16-20, 1992 (10 mins).

Electron Emission from Nanometer-size Metallic Clusters, presented by R. Reifenberger at the NATO-Advanced Research Workshop, July 6-10, 1992; Lyon, France (1 hour) **Invited**.

Electronic States and Structural Stability of Supported Au Clusters, presented by R. Reifenberger at the Sixth International Symposium on Small Particles and Inorganic Clusters, Sept. 16-22, 1992; Chicago IL (1 hour) **Invited**.

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