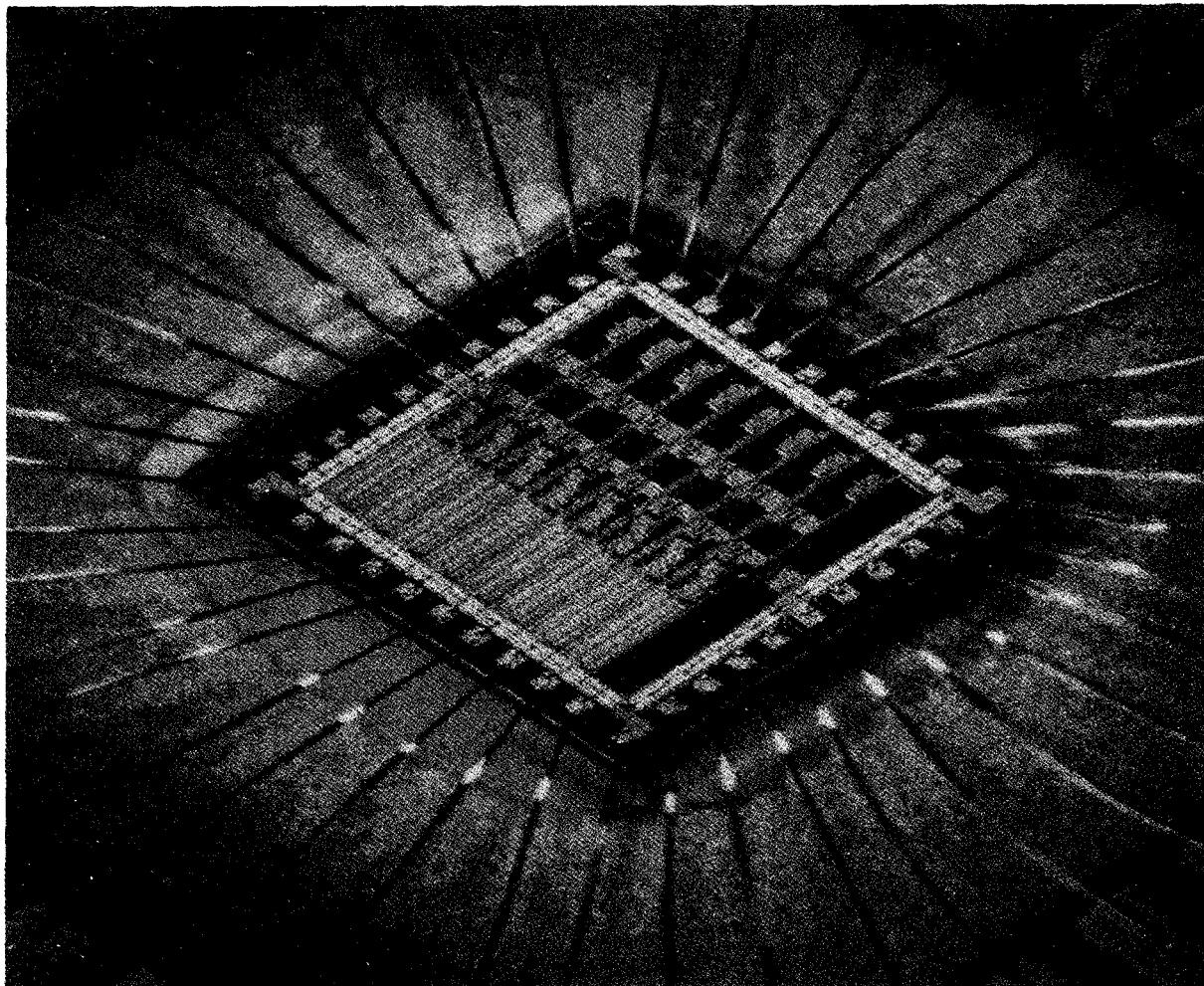


LABORATORY TECHNOLOGY RESEARCH

Abstracts of FY 1997 Projects



U. S. Department of Energy
Office of Energy Research
Office of Computational and Technology Research
Advanced Energy Projects and Technology Research Division

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LABORATORY TECHNOLOGY RESEARCH PROGRAM

Program Overview

The Laboratory Technology Research (LTR) program supports high-risk, multidisciplinary research partnerships to investigate challenging scientific problems whose solutions have promising commercial potential. These partnerships capitalize on two great strengths of our country: the world-class basic research capability of the DOE Energy Research (ER) multi-program national laboratories and the unparalleled entrepreneurial spirit of American industry.

A distinguishing feature of the ER multi-program national laboratories is their ability to integrate broad areas of science and engineering in support of national research and development goals. The LTR program leverages this strength for the Nation's benefit by fostering partnerships with U.S. industry. The partners jointly bring technology research to a point where industry or the Department's technology development programs can pursue final development and commercialization.

Collaborative research projects supported by the LTR program are partnerships: the program funds only the national laboratory's research, while the industrial partner supports its research and often provides equipment, funds, or supplies to the laboratory. Thus, a laboratory and its industrial partners can explore scientific and technical approaches that would be too risky for industry to undertake alone. Such work leverages the resources of both partners, since each frequently has unique and complementary facilities and expertise. The LTR program enhances opportunities to pursue technology research that is of value to industry, complements basic research program goals, and seeks to enhance public benefit from investment in scientific research at the national laboratories.

The scientific impact of the LTR program has already been dramatic. Since its inception in 1992, the program's technologies have won 17 R&D-100 Awards, 15 Federal Laboratory Consortium Awards, and seven other awards, such as those from Popular Science and Discover magazines. The record of R&D-100 Awards exemplifies the steadily increasing success of these cooperatively developed technologies.

Projects supported by the LTR program are conducted by the five ER multi-program laboratories: Argonne, Brookhaven, Lawrence Berkeley, Oak Ridge, and Pacific Northwest National Laboratories. These projects explore the applications of basic research advances relevant to DOE's mission over a full range of scientific disciplines. The program presently emphasizes three critical areas of mission-related research: advanced materials, intelligent processing/manufacturing research, and sustainable environments.

Program Focus Areas

Advanced Materials

The Advanced Materials portion of the LTR program will provide a strong foundation for advances in many areas of science and technology including energy, transportation, manufacturing, health, and the environment. Using synthesis, processing, and characterization techniques and advanced computational tools for design and modeling coupled with the integration of basic and applied disciplines, this research will result in the improvement of existing materials and the development of new materials and knowledge of their properties. Research focuses on a broad range of materials problems related to ceramics and composites, metals and alloys, surfaces and thin films, nanomaterials, polymers and biomaterials, and superconducting materials. Results support DOE missions in basic science, energy efficiency, fossil energy, fusion energy, environmental management, and national security. The research is intended to complement, enhance, and leverage existing DOE materials programs through research partnerships.

Advanced materials research focuses on four major subtopics.

- Intelligent Design of Materials: Emphasis is placed on modeling and characterization; alloying and doping; composite and functional graded materials; biomaterials; and nanostructures and films.
- Advanced Synthesis Technologies: Emphasis is placed on advanced techniques such as ion, plasma, laser, and MBE techniques and environmentally friendly processing techniques that reduce waste and/or energy consumption.
- Films and Coatings: Emphasis is placed on surface modification, corrosion and wear resistance, and multilayered films.
- Intelligent or Adaptive Materials: Emphasis is placed on developing materials that respond to external stimuli, such as shape memory alloys, and magneto-resistant, piezoelectric, and electro-rheologic materials.

Intelligent Processing/Manufacturing Research

Intelligent Processing/Manufacturing Research (IPMR) is a multidisciplinary activity which integrates and builds upon the results of DOE basic research to develop new and advanced processing and manufacturing technologies required to meet DOE missions. Much of the work performed under IPMR will ultimately lead to applications in private industry.

The goal of IPMR is to perform technology research projects that apply core DOE laboratory capabilities to advance the state of intelligent processing and manufacturing. To meet this goal,

research is conducted on a range of technology areas that include advanced sensors and controllers, computational technologies, and algorithms coupled with manufacturing processes. Research projects typically have applications in multiple manufacturing sectors and support DOE missions in science and technology, as well as energy and environment. IPMR projects also benefit national initiatives related to manufacturing such as Technologies Enabling Agile Manufacturing (TEAM), Next Generation Manufacturing (NGM), and the Partnership for a New Generation of Vehicles (PNGV).

IPMR focuses on three major subtopics.

- Intelligent Design: Emphasis is placed on modeling and simulation and on rapid prototyping.
- Intelligent Manufacturing Processes: Emphasis is placed on joining; forming, forging, and casting; and microfabrication.
- Enabling Technologies: Emphasis is placed on intelligent measurements, intelligent controls, and agile automation.

Sustainable Environments

A new generation of environmental technologies is needed that supports pollution prevention, efficient resource use, and industrial ecology. Such technologies can help companies become more competitive by lowering resource and energy needs, reducing waste and emissions control costs, and fostering sustainable development. ER supported programs in biotechnology, chemical and materials sciences, and novel energy concepts provide a fertile ground for further investigation for potential commercial application. Priorities for research in the sustainable environments area stress technologies that emphasize sustainable use of natural resources and avoidance of environmental harm. These may include technologies to control and minimize environmental harm (particularly hazardous wastes), an improved environmental technologies information infrastructure, monitoring technologies, and remediation technologies focused on areas such as manufacturing, transportation, materials, water, and energy.

Research on sustainable environments focuses on three major subtopics.

- Biotechnology: Emphasis is on furthering developments in understanding the microbial and biochemical mechanisms that can contribute to solving complex bioprocessing problems. Topics in molecular biology, biochemistry, microbiology, and biomedicine fall into this category. A potential area for investigation may be the application of extremophile bacteria to the degradation of toxic wastes.
- Chemical Process Modeling: This area fosters teaming with the Mathematical, Information, and Computational Sciences Division (MICS) of the DOE Office of Computational and Technology

Research to apply modeling and simulation capability in development of environmentally benign chemical processes. This topic may span a spectrum from the application of modeling tools in the development, at the structural level, of new classes of catalysts, to large scale industrial process modeling.

- Novel Energy Devices: New developments in mechanical engineering and materials sciences are progressing toward the miniaturization of motors, pumps, and compressors to a microscale. Further investigation is required to make these devices economically feasible. This area has potential to revolutionize the way heating and cooling are produced and used in commercial and home settings.

PROGRAM IMPLEMENTATION

The LTR program conducts research using three different mechanisms:

- **Multi-Year Projects.** These cost-shared projects between ER multi-program laboratories and private industry are performed in support of DOE missions but also are relevant to industry needs. LTR program funding to ER laboratories for these projects is typically from \$100,000 to \$250,000 per year for a three-year period. The industrial partner supports its research in at least an equivalent amount. Cooperative Research and Development Agreements (CRADAs) are used to implement these projects. CRADAs provide for protection of proprietary data and disposition of intellectual property. Projects that were initiated in Fiscal Year (FY) 1997 were chosen by an external peer review of proposals on the basis of scientific/technical merit and commercial potential. The reviewers were practicing experts in the subject area of the proposal, from at least three different institutions, who did not have a conflict of interest. Each reviewer provided comments on four evaluation criteria: scientific/technical quality, qualifications of key personnel and facilities, the work plan, and commercial potential. A fifth criterion on the industry partner commitment to the project (either funds-in or in-kind support) was evaluated by the DOE LTR office based on commitment letters from the industry partner. Proposals which received the strongest evaluations overall were chosen for funding.

All of the multi-year projects which received FY 1997 funding from the LTR program (Total = \$14.4 million) are included in this book of abstracts. The following ID codes for each abstract identify the national laboratory conducting the project: ANL - Argonne National Laboratory, BNL - Brookhaven National Laboratory, LBL - Lawrence Berkeley National Laboratory, ORL - Oak Ridge National Laboratory, and PNL - Pacific Northwest National Laboratory.

- **Quick Response Projects.** These projects provide private industry, especially small businesses, with a means to solve difficult technical problems rapidly by tapping the unique expertise of ER laboratory scientists and engineers. These projects are implemented through a variety of flexible mechanisms, such as personnel exchanges, technical assistance to and consultations with small businesses, and small collaborative projects (CRADAs). Funding is allocated on the basis of a merit review that emphasizes scientific/technical quality, commercial potential, and contribution to DOE's missions. These projects last from a few days to one year with LTR program funding to ER laboratories from \$3,000 to \$100,000 per project.

Examples of each type of quick response project are included in this book of abstracts. The total funding for quick response projects was \$1.4 million in FY 1997.

- **Major Industry Partnerships.** These partnerships team scientists and engineers in DOE national laboratories with an industry sector to research generic problems facing the

industry. The LTR program supports the American Textiles (AMTEX) Partnership and the Advanced Computational Technology Initiative (ACTI) Partnership. The objective of AMTEX is to transfer advanced technology into the Integrated Textile and Apparel sector. ACTI focuses on the discovery and recovery of oil and gas by supporting borehole seismic, oil recovery, drilling and completion, and computational technologies. In both partnerships, DOE and industry research generic, pre-competitive technologies for their mutual benefit. DOE supports national laboratory scientists and engineers, and industry supports its own researchers.

Major industry partnership projects funded by the LTR program are part of a large group of projects focused on one industrial segment. Abstracts of multi-year projects in support of the ACTI Partnership, which received FY 1997 funding from the LTR program, are listed as project sequence numbers 87 - 94. For information on the AMTEX program, contact the AMTEX Laboratory Program Office at the Oak Ridge Y-12 Plant, Oak Ridge, Tennessee, 37831, Telephone (423)576-5526.

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ABSTRACTS OF MULTI-YEAR PROJECTS SUPPORTED IN FY 1997**ADVANCED MATERIALS****Synthesis and Processing**

1. Title: **Cold Cathode Electron Emission from Diamond and Diamond-Like Carbon Thin Films for Flat Panel Computer Displays**

ID: ANL95-02

PI: Alan R. Krauss
Materials Science and Chemistry Division

Phone: 630 252-3520

Partner: SI Diamond Technology, Inc.
Houston, Texas

FY 97 Funding: \$140K

Total Project Funds: \$420K

Cold cathode electron emission has been observed from a number of diamond and diamond-like carbon thin films. It is expected that this phenomenon can be used for the development of high visibility displays for critical applications such as avionics, high reliability microelectronics applications for operation in harsh environments where maintenance is not feasible, and flat panel computer displays. In this latter application, it is expected that cold cathode emission technology will result in a significant reduction of plant cost, unit cost, and energy consumption compared with current active matrix liquid crystal displays. Development of diamond field emission technology for reliable displays will also provide a means for U.S. industry to enter the multi-billion dollar flat panel display market which is currently dominated by Asian industries. However, several of the key physical properties which affect field emission performance are normally interrelated so that it is difficult to perform controlled measurements of the effects of these properties on electron emission. The development of devices like flat panel computer displays which use cold cathode electron emission has been hampered by a lack of basic understanding of the emission process. A method has been developed at ANL for the growth of diamond films in the near-absence of atomic hydrogen, using Ar-C₆₀ or Ar-CH₄ plasmas. This method produces films which respond differently to variations in growth conditions compared with films grown

in large quantities of hydrogen. The differences manifest themselves in the manner in which the nucleation density, grain size, grain boundary width, surface roughness, crystallographic orientation, and the extent and localization of regions of sp₂ and sp₃ electronic bonding character vary with the hydrogen concentration in the plasma. ANL has related several of these properties to the effective work function, turn-on voltage, and emission site density by comparing the electron emission behavior and physical properties of conventional micro-and nano-crystalline, and low-hydrogen nano-crystalline, diamond films. Work will continue in two areas: (1) The emission sites will be identified, and a determination of the site density will be made, using photo-electron emission microscopy (PEEM) for several varieties of electron emitting diamond and diamond-like carbon films. The project team has found that post-deposition treatment is critical in controlling emission properties, and the PEEM data will be studied in conjunction with oxygen and hydrogen plasma post-deposition processing. (2) Studies of the grain morphology of films produced both at SI Diamond and at ANL are being conducted using transmission electron microscopy. The ANL portion of these tasks will continue with funding from the Advanced Projects Research Agency as part of a program for improvement of diamond cathode materials for high resolution displays. Research in diamond structures and applications to electronics support DOE's long standing mission in materials sciences.

2. Title: Giant Magnetoresistance Wire Sensor**ID:** ANL95-07**PI:** Samuel D. Bader
Material Sciences Division**Phone:** 630 252-4960**Partner:** Sagax Technology
Columbia Heights, MN**FY 97 Funding:** \$75K**Total Project Funds:** \$425K

Giant magnetoresistance (GMR) materials are a class of materials that consist of a ferromagnetic phase interleaved with or otherwise nanodispersed through a nonferromagnetic phase. They have the potential to outperform conventional magnetoresistive materials by one to two orders of magnitude. Magnetoresistive devices are currently being developed for many magnetic-field-sensing applications, such as automotive, power distribution, and information storage systems. Lowering the cost of fabrication and increasing the sensitivity will no doubt remove barriers for use of magnetic field sensing in both industrial processing and consumer products. ANL has been at the forefront in probing the underlying physics of the GMR effect in ultrathin magnetic films. Among major

accomplishments is the realization of record-setting GMR values for epitaxial Fe/Cr superlattices. The ER-LTR program in GMR wires at ANL has taken a bulk-processing approach. The objective is to develop inexpensive GMR materials based on granular magnetic systems, which consist of nanometer-sized magnetic particles uniformly dispersed in a conducting, nonmagnetic matrix. The approach utilizes the immiscibility of the constituent materials of a metastable alloy and the phase-separation induced by heat treatment. Metastable alloys of Co and Cu in the form of long ribbons have been fabricated via melt-spinning, which is widely adopted in industry for producing rapidly quenched amorphous alloys. The ribbons are subsequently subjected to heat treatment in order to control the size and distribution of the magnetic particles, and to optimize the GMR effect. A room-temperature GMR effect of 7% is observed in ribbons after suitable annealing, but the field sensitivity needs to be improved. Toward this end, the effect of mechanical processing on GMR has been explored. It is hoped that uniaxially deforming the ribbons would induce a shape anisotropy to stabilize the magnetic particles against thermal agitation and improve the field sensitivity. Under certain circumstances, ribbons deformed prior to heat treatment show small improvements in field sensitivity. The challenge is to prepare metastable alloys whose tensile strength is better able to sustain the large mechanical deformation needed to tune the GMR field sensitivity. This project supports the DOE mission in advanced materials research applications to new energy devices.

3. Title: High Performance Tailored Materials for Levitation and Permanent Magnet Technologies

ID: ANL97-02

PI: George W. Crabtree
Materials Science Division

Phone: 630 252-5509

Partner: Superconductive Components, Inc.
Columbus, OH

FY 97 Funding: \$125K

Total Project Funds: \$750K

The high temperature superconductor $Nd_{1+x}Ba_{2-x}Cu_3O_{7-y}$ has recently been recognized as a powerful new material in which strong magnetic flux pinning has been observed. This material can be used for the development of levitators and trapped-field permanent magnets, creating an opportunity to drive substantial advances in the performance and technical competitiveness of these technologies. The development of this material will shift the leading edge of materials research in this area from the two foreign laboratories (ISTEC, Japan and FZK, Germany) now dominating the field to the U.S.

Although the strong pinning characteristics of this material have been recognized, the responsible pinning centers and efficient procedures for large scale fabrication of the material remain relatively obscure. ANL has identified a crucial new processing variable, the high temperature cooling rate during the growth process, which can be used to tailor the flux pinning properties of these materials. In addition, ANL has developed a low temperature oxygen anneal processing technique to control the magnetic field where maximum pinning occurs. An important objective of this project is to understand the origin of the superior flux trapping capabilities and to develop fabrication procedures using top-seeding techniques for making large samples for applications. Processing methodology will be developed based on property measurements using X-ray and neutron diffraction, magneto-optical imaging, magnetization measurements, and scanning tunneling microscopy on melt-textured material and single crystal samples. The best material will be tested in prototype levitating flywheels to assess its value and define problem areas in collaboration with our industrial partner, Superconductive Components, Inc. The development of high performance flux pinning materials will enable a new generation of levitation devices for frictionless bearings and flywheel energy storage, and of permanent trapped-field magnets. The materials performance advances achieved under this project can be applied to other developing technologies, such as coated conductors for high current carrying wires (IBAD and RABiTs), and high power microwave filters for cellular communications. (1) The emission sites will be identified, and a determination of the site density will be made, using photo-electron emission microscopy (PEEM) for several varieties of electron emitting diamond and diamond-like carbon films. The project team has found that post-deposition treatment is critical in controlling emission properties, and the PEEM data will be studied in conjunction with oxygen and hydrogen plasma post-deposition processing. (2) Studies of the grain morphology of films produced both at SI Diamond and at ANL are being conducted using transmission electron microscopy. The ANL portion of these tasks will continue with funding from the Advanced Projects Research Agency as part of a program for improvement of diamond cathode materials for high resolution displays. Research in diamond structures and applications to electronics support DOE's long standing mission in materials sciences.

4. Title: Smooth Diamond Films for Friction and Wear Applications and Chemically Protective Coatings

ID: ANL97-05

PI: Alan R. Krauss, D. M. Gruen
Materials Science and Chemistry Division

Phone: 630 252-3520

Partner: Durametallic Corporation
Kalamazoo, MI

ASTeX, Inc
Woburn, MA

FY 97 Funding: \$150K **Total Project Funds:** \$750K

Diamond has a number of properties which, in principle, make it an exceptional material for a large number of applications. In particular, the extreme hardness (harder than any other known material), chemical inertness (it resists attack by almost all known acids and bases), and low coefficient of friction (comparable with that of Teflon™) make it an ideal candidate for a wide range of applications involving sliding or rolling contact between moving surfaces. However, conventional diamond chemical vapor deposition (CVD) methods produce coatings with extremely rough surfaces. This roughness has limited the development of diamond film technology for tribological applications, and penetration of diamond film technology into these markets has been disappointingly slow. This project concerns the use of a process developed at Argonne National Laboratory for the production of ultra-smooth diamond coatings on rotating and sliding mechanical parts in order to reduce energy consumption, improve product reliability, and reduce toxic emissions into the environment. Films produced by this process have been shown to possess tribological properties which eliminate the problems which have so far limited the use of diamond coatings for applications involving moving parts. The work to be performed addresses adaptation of the process for the production of diamond coatings that are 10-100 times smoother than those produced by existing processes. End face mechanical seals, used to prevent the leakage of gases and liquids in equipment with rotating shafts, have been chosen as the area of application. The benefits obtained in terms of energy savings, increased productivity, reduced maintenance, and reduced release of environmentally hazardous materials for this single application will be substantial, but the technology which will be developed will also be directly applicable to a large number of application areas in manufacturing and transportation, in most cases with similar benefits. This project supports DOE mission in the application of basic research developments in material sciences to improved processing technologies.

5. Title: Ionically Conductive Membranes for Oxygen Separation

ID: LBL97-03

PI: Steven J. Visco
Materials Sciences Division

Phone: 510 486-5821

Partner: Praxair, Inc.
Tonawanda, NY

FY 97 Funding: \$125K

Total Project Funding: \$750K

The global market for industrial oxygen is estimated at \$20 billion annually. The dominant technology for the production of commercial oxygen is cryogenic distillation. The high capital equipment costs for cryogenic O₂ separation limits this technology to large installations. Accordingly, industrial suppliers of oxygen are highly motivated to develop technologies that can satisfy increasing demand for oxygen through smaller scale plants. One approach under development elsewhere is the use of mixed ionic-electronic ceramics; when such ceramic electrolytes are exposed to compressed air on one side and ambient pressure on the other, oxygen diffuses through the mixed conductor from the compressed side to the low pressure side due to the chemical potential gradient of oxygen across the membrane. The drawback to this technology is the need for a compressor which raises issues of noise and reliability. Another problem is that permeation delivers ambient pressure oxygen. In contrast, we propose the efficient electrolytic extraction of oxygen from air using novel thin-film structures consisting of high strength ionic membranes supported on porous, catalytic electrodes. Using this technology, high purity O₂ can be electrochemically pressurized as an integral part of the separation process. The simplicity of operation of an electrolytic O₂ generator promises high reliability as well as low cost. Still, to survive as a commercial process, this approach must be cost-competitive to cryogenic production of O₂. Key to success is highly efficient operation (low power consumption) of the device along with low fabrication costs. Power losses in the electrolytic oxygen cell will be related to ohmic losses across the electrolyte membrane, charge transfer polarization at the electrode/electrolyte interfaces, and mass transfer polarization across the electrodes. The LBNL approach addresses the above issues in such a way that both scientific and technical success are likely. The LBNL team has initiated preparation of porous substrates suitable for colloidal deposition. High temperature furnaces are being installed for sintering of bilayer structures suitable for high oxygen flux in an electrolytic oxygen generator. The LBNL investigator recently met with the principal investigator for the industrial partner to discuss the timeline for the development plan. A tentative date of October 1997 has been agreed upon for the LBNL team to travel to industrial partner's laboratory in order to discuss the development plan in detail, and to ensure maximum productivity of the collaborative effort. This research supports the DOE mission in materials research and applications.

6. Title: Combinatorial Discovery and Optimization of Novel Materials for Advanced Electro-Optical Devices

ID: LBL97-18

PI: Xiao-Dong Xiang
Materials Sciences Division

Phone: 510 486-4864

Partner: NZ Applied Technologies (NZAT)
Woburn, MA

FY 97 Funding: \$125K **Total Project Funds:** \$750K

Advanced materials are the building blocks of the emerging photonic technologies which are the foundation for a new industrial base. Complex oxide ceramics (ternaries and higher order compounds) exhibit a wide range of technologically significant properties such as the electro-optic effect. The rapid expansion in the types of phenomena exhibited by modern advanced ceramics has revived interest in the use of complex oxides for advanced optical device applications. This project directly supports DOE's interests in materials research for advanced ceramic applications. However, due to the complexity of multi-component oxides, searching for new materials or optimization of existing materials has become a forbidding task for the materials community. This project will: (1) use the method of combinatorial synthesis and screening, recently developed at LBNL, to evaluate a wide range of oxide materials and compounds and optimize the advanced oxide materials for electro-optical devices; and (2) use heteroepitaxial thin film growth methods, developed at NZAT, to fabricate advanced oxide electro-optical devices based on search and optimization results. The goal of this project is to produce commercially viable advanced electro-optical devices. If successful, this project will play an important role in forming a strong foundation for the emerging large scale integrated optics device industry.

7. Title: Development of High-Temperature Superconducting Wire Using RABiTS Coated Conductor Technologies

ID: ORL97-02

PI: David K. Christen
Solid State Division

Donald M. Kroeger
Metals and Ceramics Division

Phone: 423 574-6269

Partner: 3M
St. Paul, MN

Southwire Corporation
Carrollton, GA

FY 97 Funding: \$100K

Total Project Funds: \$750K

This project is developing a recent breakthrough at Oak Ridge National Laboratory that offers a potential new route to the fabrication of high-temperature superconducting (HTS) wires for power applications. The new process produces high-T_c coatings that have high critical-current densities at liquid nitrogen temperatures, and enable operation in substantial magnetic fields. The present approach exploits the growth of crystalline biaxially-aligned coatings on oriented metal tapes produced by simple thermo-mechanical processing. The tapes start with inexpensive polycrystalline metals or alloys that are biaxially aligned by deformation rolling and thermal annealing treatment, followed by the epitaxial deposition of thin, passivating buffer layers. The project team is investigating the scientific and technical feasibility of making long-length coated conductors that can provide operating characteristics that are currently unattainable by any electrical conductor, including present prototype HTS tapes that utilize the "powder-in-silver-tube" fabrication approach. The research focuses on both the simplification and optimization of oxide buffer layers on reactive metals, and specifically will evaluate (co)evaporation techniques for both the buffer layer(s) and the superconductor coatings. 3M has an established experience base in high-rate deposition of other materials using this manufacturing technology. Southwire is the leading U.S. manufacturer of utility wire and cable, and is a retailer of underground transmission cables (a prime first candidate for HTS insertion). A cost-effective route to high-temperature superconducting wires would provide substantial national energy benefits in the technology of electric power production, storage, and distribution. Superconducting transmission lines alone would enable 2-3 times the power transfer into urban areas without the need for additional rights-of-way and without significant losses to resistance. Other applications, such as power transformers, motors, current limiters, and magnetic energy storage, are projected to produce markets of tens of billions of dollars per year. This project will help DOE's Office of Utility Technologies, Energy Efficiency and Renewable Energy program to develop high-temperature superconductors.

8. Title: Bioactive and Porous Metal Coatings for Improved Tissue Regeneration

ID: PNL95-23

PI: Allison Campbell
Materials and Chemical Sciences Division

Phone: 509 375-2180

Partner: Health Tech Development
Dallas, TX

FY 97 Funding: \$191K **Total Project Funds:** \$597K

The goal of this project is to further develop technologies (void metal composites and surface induced mineralization) derived from DOE materials research programs and conduct an experimental program. This project satisfies DOE research interests in fundamental applications of novel materials. The project will aid in the development of a formal manufacturing program for an improved medical device. Nearly twenty million Americans and more than fifteen million others afflicted with degenerative bone and joint diseases, as well as those who have suffered bone fractures, need devices which anchor to the unaffected bony tissue around the defect site. Although significant advances have been made in the field of metallurgy to provide strong, non-toxic metals and alloys, biological integration of devices into natural tissues remains a problem. Thus, many effective devices become loose over time and necessitate subsequent surgery to replace the old device, a process fraught with high morbidity and mortality. Efforts to solve the problems associated with device anchoring have been highly fragmented among the biological, mechanical, and surgical disciplines. The Surface Induced Mineralization (SIM) and Void Metal Composite (VMC) processes produce a bioactive porous metal implant coating/device which may address many of the problems associated with conventional processing methods. The VMC process produces materials that have cylindrical pores of uniform diameter, which can completely penetrate the structure of the material. Pore diameter, orientation, and interconnectivity are easily controlled. The SIM process uses the idea of nature's template-mediated mineralization by chemically modifying the implant to produce a surface which induces heterogeneous nucleation from aqueous solution. SIM produced bioactive coatings provide (1) control of the thickness and density of the mineral phase, (2) a way to coat porous metals, complex shapes and large objects, (3) the ability to coat a wide variety of materials, and (4) a potential choice for the phase of the mineral formed. To date, the ability to coat a VMC material with bioactive materials has been successfully demonstrated. The ability to incorporate biological agents within this bioactive matrix has also been demonstrated. In addition, these coatings signal natural cellular response, *in vivo*. Currently, almost all synthetic bioactive coatings do not signal the desired biological response.

9. Title: Interfacial Interactions of Biological Polymers with Model Surfaces

ID: PNL97-21

PI: Allison Campbell
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Phone: 509 375-2180

Partner: Ross Products Division
Abbott Laboratories
Columbus, OH

FY 97 Funding: \$124K

Total Project Funds: \$749K

The adsorption of biological polymers onto surfaces affects many different industrial processes. However, the controlling mechanisms and the interfacial structure are not well understood for most systems. This project will develop and apply state of the art methods to design, synthesize, and characterize systems for adsorption experiments. Specifically, molecular beam epitaxy, chemical vapor deposition, and self-assembling monolayers will be used to construct surfaces with controlled properties such as chemistry, topography, and heterogeneity. For the first time, chemical vapor deposition methods for producing controlled surfaces of the biologically relevant calcium oxalate, carbonate, and phosphate systems will be developed. Biological polymers of human serum albumin, Protein G, and fibrinogen will be used in the adsorption experiments. These provide excellent models since they exhibit a range of structures, sizes, and chemistries. State of the art techniques of neutron scattering and reflectometry, quartz crystal microbalance, liquid chromatography/ mass spectroscopy, and atomic force microscopy will be employed to study adsorption *in-situ*. Information on adsorption kinetics, isotherms, and protein conformation will be obtained in real time. Finally, solid state nuclear magnetic resonance experiments will be conducted to identify the specific protein residues that are interacting with the surface. This investigation will provide molecular level information on specific interactions that has not yet been obtained. The project will contribute to achieving DOE's mission in fundamental science, while also providing knowledge and technology to potentially enable the development of improved materials for use in health care.

10. Title: Processing Property Relationships in Centrifugally Cast Aluminum Metal Matrix Composites

ID: PNL94-02

PI: Edward L. Courtright
Material Sciences Division

Phone: 509 375-6926

Partner: General Motors Corporation Gear Center
Romulus, MI

FY 97 Funding: \$245K**Total Project Funds: \$830K**

The goal of this project is to develop a cost-effective selectively reinforced metal matrix processing technology that can be used to manufacture automobile components. Light alloy metal matrix composites (MMCs) reinforced with silicon carbide or alumina particulates can replace steel in many automobile applications. The corresponding reduction in vehicle weight translates to a proportional increase in gas mileage, which will assist in fulfilling DOE's energy mission. This project concentrates on understanding the microstructure of centrifugally cast MMCs. This process offers the unique capability to distribute the particulate phase in regions or zones where the reinforcements will have the greatest benefit. Emphasis is being placed on understanding processing/property relationships and in determining how these can be controlled to optimize selectively reinforced composite structures. In the past year, several Al-SiC composite test gears have been centrifugally cast. Analytical studies have shown that the centrifugal casting process selectively distributes the hard ceramic particles in the tooth and root region of the gear where good mechanical properties and high wear resistance are needed. Single tooth bending studies have shown that these materials exhibit promising high-cycle fatigue properties. The effects of coining deformation on the fatigue behavior are also being investigated. Durability tests performed by GM have shown that Aluminum MMC gears out perform their steel counterparts. Engine dyno testing, under a variety of load conditions, is currently underway and will help provide information on service life.

11. Title: Innovative Multilayer Thermal Barrier Coatings for Gas Turbine Engines**ID:** PNL95-07**PI:** Edward L. Courtright
Material Sciences Division**Phone:** 509 375-6926**Partner:** Solar Turbines, Inc.
San Diego, CAHowmet Corporation
Whitehall, MI**FY 97 Funding: \$245K****Total Project Funds: \$835K**

The objective of this project is to demonstrate the feasibility of improving the performance of thermal barriers using nanoscale multilayer ceramic coatings. If improved thermal barriers can be developed,

stationary turbines and other types of processes can be operated at higher, more efficient temperatures, resulting in substantial energy savings, thus contributing to achieving DOE's energy mission. The coatings are designed to maximize infrared reflectivity while taking advantage of the principles of phonon scattering at the multiple interfaces. The thermal dynamic stability issues that affect durability, reliability and life-cycle performance are being investigated. To date, independent thermal conductivity measurements performed by the National Institute of Standards and Technology on PNNL coatings produced in the project have shown that the thermal conductivity of a zirconia coating can be reduced by a factor of two or more using a nanostructure multilayer design. The multilayer structure has also been shown to be tough and resistant to crack propagation. Current studies are now addressing adherence and long-term thermal cycle stability performance. In the past year, a variety of single crystal superalloy coupons have been coated and tested by Solar Turbines, Inc. and the Howmet Corporation for thermal cycle durability. In addition, studies are being performed at Ohio State University on the oxidation kinetics of the underlying metal substrate. Oxidation is expected to be significantly reduced by the insertion of alumina layers which are impermeable to oxygen. In the next year of the project, actual components will be coated and tested by industrial partners under simulated engine conditions, e.g., burner rigs or in actual land-based gas turbine engines.

Materials Design and Characterization**12. Title: Corrosion Resistance of New Alloys for Biomedical Applications**

ID: BNL94-20

PI: Hugh Isaacs
Department of Applied Science

Phone: 516 344-4516

Partner: Smith and Nephew Richards, Inc.
Memphis, TN

FY 97 Funding: \$140K **Total Project Funds:** \$450K

The objective of this project is to provide a detailed understanding of alloy corrosion in bio-systems and the role of the individual alloy additions. Ultimately, the project expects to obtain an understanding of the interactions between alloy composition and the electrochemical response of alloys with optimum mechanical properties and biocompatibility. The development of new materials for prosthetic devices and other biomedical applications is currently underway. In-situ X-ray absorption measurements in simulated bio-fluid (Ringer's solution) and under crevice conditions (concentrated chloride solution) will provide information on the chemical behavior of the alloys during corrosion. A detailed study of oxide formation will be carried out using X-ray absorption and surface analytical techniques. Corrosion resistance, growth kinetics, and composition of the oxide film on zirconium (Zr) is being studied to understand their contribution to increased corrosion resistance of titanium (Ti) alloyed with niobium (Nb) and Zr. BNL has performed extensive studies at the National Synchrotron Light Source to characterize the growth and composition of oxide film on Ti-Nb-Zr alloys. Future studies will include determination of any selective leaching of alloy components in crevice solutions, and variables relating to the adherence of deposits, the effects of substrates, deposit thickness, and hydriding. Research in this area supports DOE missions in materials science and medical applications.

13. Title: Nondestructive X-Ray Scattering Characterization of High Temperature Superconducting Wires

ID: BNL95-10

PI: Thomas Thurston
Physics Department

Phone: 516 344-5534

Partner: Intermagnetics General Corporation
Latham, NY

FY 97 Funding: \$160K

Total Project Funds: \$630K

The purpose of this project is to characterize the structure of the superconducting material within wires made at Intermagnetics General Corporation (IGC) in order to understand the cause of poor current carrying capacity, and to suggest alternative processing procedures which can minimize or eliminate the effects which cause poor wire performance. The methods which BNL personnel are using to characterize the wires utilize intense beams of X-rays generated at the National Synchrotron Light Source (NSLS) at BNL. Work performed in this project has shown that the current carrying capacity is affected by the presence of certain impurity phases, and by poor texturing of the superconducting material within the wires. Both of these deleterious effects can be readily measured only with the intense X-ray beams available at facilities like the NSLS. Work currently in progress involves direct X-ray monitoring of superconducting wire processing in a "mini-factory" which has been set up at BNL. This work has already suggested modifications to the processing procedures which have helped increase the current carrying capacity of IGC's wires. The project has also characterized wires made in the DOE's high-temperature superconductor electric power applications program at BNL. Finally, BNL has started to apply the techniques developed to characterize these wires on other problems of interest to the DOE, such as characterizing the properties of battery electrode materials and permanent magnets.

14. Title: Next Generation Corrosion Inhibitors for Steel in Concrete

ID: BNL95-12

PI: Hugh Isaacs
Applied Science

Phone: 516 344-4516

Partner: Grace Construction Products (W.R. Grace & Co.)
Cambridge, MA

FY 97 Funding: \$50K**Total Project Funds: \$636K**

Steel-reinforced concrete is the most widely used construction material in the world. This is almost an ideal composite, with the steel providing tensile strength and the alkaline concrete imparting passivity to the steel. However, passivity can be compromised by the ingress of chlorides from a marine environment or from de-icing salts. To address this problem, corrosion inhibitors are added to the concrete mixture, usually as simple inorganic anions (e.g. nitrite). Both the mechanism of corrosion in a concrete environment and the action of inhibition are not well understood. The goal of this project is to elucidate the action of corrosion and the behavior of inhibitors. The objective of the study will ultimately be to develop more effective inhibition, possibly by the use of mixed anodic/cathodic inhibitors or altering the form in which the inhibitors are added. Corrosion measurements are being made of the anodic and cathodic kinetics taking place in concrete, which describe the processes occurring with and without inhibitors. Nitrite inhibitors have been found to display different degrees of effectiveness at various stages during the development of corrosion. In sufficient quantities, the inhibitors maintain passivity. However, they apparently have little action on the very early stages of passivity breakdown. At a later stage, when corrosion is well developed, corrosion is again influenced by nitrite additions. Very small quantities distinctly reduce the corrosion rates, whereas large additions again act to produce passivity and no corrosion. Efforts are now underway to define more closely the critical factors determining the differences in behavior. X-ray absorption near edge measurements will also be performed to examine the effect (if any) of inhibitors on the structure and chemistry of the passive oxide. Research in this area supports the DOE mission in materials characterization and processing.

15. Title: Catalytic Production of Organic Chemicals Based on New Homogeneously Catalyzed Ionic Hydrogenation Technology**ID:** BNL97-05**PI:** Morris Bullock
Chemistry Division**Phone:** 516 344-4315**Partner:** DuPont Company
Wilmington, DE**FY 97 Funding: \$118K****Total Project Funds: \$706K**

This project will focus on the development of new technology for the production of organic chemicals

of commercial interest, based on fundamental research at BNL exploring the reactivity of transition metal hydride complexes. The scientific objectives are to explore the feasibility, scope, and selectivity of catalytic *ionic* hydrogenation technology. In these reactions, H₂ is added to an organic chemical sequentially, in the form of a proton (H⁺) followed by hydride (H⁻). The project plans to discover transition metal complexes that can carry out these functions catalytically, with hydrogen (H₂) being the ultimate source of both the proton and hydride. Homogeneously catalyzed ionic hydrogenations offer the possibility of enabling efficient and selective hydrogenation processes for organic transformations that are difficult to achieve by conventional methods. Initial work will focus on attempts to develop prototype metal systems capable of catalytic hydrogenation of ketones. Tungsten systems with weakly coordinating counterions will be investigated first, since preliminary results have indicated that such systems have the requisite ability to form cationic tungsten dihydride complexes upon reaction with H₂. A key issue to be addressed will be the relative binding strength of different ligands to the metal, and measurements of this type may require high pressure nuclear magnetic resonance experiments at DuPont. When a successfully functioning catalytic system is developed, optimization will be attempted by systematic variation of ligands and the metal. Further elaborations will later attempt to utilize these methods in asymmetric hydrogenations to produce commercially viable processes. This project supports the fundamental DOE mission in understanding the mechanisms for catalysis and the chemical conversion of materials from biomass.

16. Title: Novel Biocompatible "Smart" Contact Lens Material**ID:** LBL94-28**PI:** Carolyn Bertozzi
Materials Sciences Division**Phone:** 510 643-1682**Partner:** Sunsoft Corporation
Albuquerque, NM**FY 97 Funding:** \$211K**Total Project Funds:** \$510K

Vision is by far the most important of the human senses, and better ophthalmological care products are continuously being sought. For example, current synthetic contact lens materials have limited tolerance by the population. The goal of this project is to develop improved materials that will increase the quality of life not only for current wearers but also for those whose physiology cannot tolerate existing materials. In the design of new contact lens materials, LBNL has modified materials with favorable lens properties so that they more closely resemble biological tissue, and are therefore

tolerated well by the eye. The knowledge gained is expected to further the understanding of how materials behave in a physiological environment and benefit biomedical implant devices development in general. This work represents a significant advance in the development of new biocompatible materials. The first phase in the development of new contact lens materials was the design of biocompatible monomers for incorporation into hydrogel polymers. In order to create lenses that best mimic biological tissue, the project focused on carbohydrate molecules which comprise the coating of most living cells. The strategy was to synthesize polymerizable monomers possessing cell surface-like carbohydrates, and to incorporate them into lenses with better biocompatibility properties. So far, the project has developed new carbohydrate-based monomers; showed that carbohydrate composition is related to water content; developed synthetic methods for biomolecular monomers and incorporated them into contact lenses; characterized the bulk and surface properties of the polymers; uncovered an effect of mold composition on surface structure; developed new methods for biomolecular surface coating; and developed a protocol to quantify the adsorption of proteins from a synthetic complex tear solution onto lenses. A collaboration has been added with the School of Optometry at the University of California, Berkeley, for contact lens/corneal interactions. This project supports DOE's long standing mission in development of health related materials research.

17. Title: Microscopic Imaging of Magnetic Materials**ID:** LBL95-12**PI:** Howard Padmore and Neville Smith
Accelerator and Fusion Division**Phone:** 510 486-5787**Partner:** IBM Research Center
San Jose, CA**FY 97 Funding:** \$378K**Total Project Funds:** \$900K

This project is an IBM-LBNL partnership to produce a powerful and unique tool for microscopic imaging of magnetic materials - a tool which will take full advantage of the capabilities of the Advanced Light Source -and to use this tool to develop new magnetic materials for high-density information storage. The microscope is based on a full-field photoelectron emission technique, and magnetic information is extracted using a synchrotron radiation spectroscopy known as X-ray Magnetic Circular Dichroism (X-MCD). The microscope will have elemental and chemical selectivity, combined with surface sensitivity, and the ability to measure surface magnetic moments. This combination of features is unique in the array of tools currently used to study magnetic

materials. The project combines LBNL's expertise in design and operation of synchrotron instrumentation, beamlines, and experimental end stations with IBM's expertise in microscopy and in the production of artificially engineered magnetic microstructures. IBM will use the information from the studies to advance the technology of high-density information storage, thereby assisting the development of new products such as non-volatile magnetic random access memories, and keeping IBM competitive in a fast-moving industry. Progress has been achieved in four areas; (1) PEEM1 Microscope - the microscope has been fully commissioned, and its spatial resolution has been demonstrated at $0.3 \mu\text{m}$. Many new surface phenomena have been investigated, including segregation phenomena in co-polymer systems, formation of thin film diamond-like carbon crystallites and films, micro-tribological studies of the wear of magnetic disc surfaces and reactions with the lubricant layer, as well as the formation of titanium silicide in confined domains. (2) Beamline 7.3.1 - The beamline used to monochromatize and focus circularly-polarized soft x-rays has been completed and fully commissioned. Initial experiments in bulk X-MCD are about to commence as a prelude to installation of the microscope. (3) PEEM2 Microscope - A new high voltage version of PEEM1 has been constructed. This works at 30 KV, and has a more sophisticated electron optical system. Detailed theoretical analysis indicates that we should be able to achieve 20 nm resolution. All system components have been constructed, and we expect first operation in October 1997. (4) PEEM3 Microscope - A theoretical design for an aberration-corrected PEEM is in progress, and indicates that a resolution near 3 nm should be possible. Research in this area supports the DOE mission in improved materials.

18. Title: Light Emission Processes and Dopants in Solid State Light Sources**ID:** LBL97-13**PI:** Eugene E. Haller
Materials Sciences Division**Phone:** 510 486-5294**Partner:** Hewlett-Packard Company
Palo Alto, CA**FY 97 Funding:** \$125K**Total Project Funds:** \$750K

Light emitting diodes (LEDs) functioning in the red and infrared have been manufactured in large quantities since the 1960s. However, until very recently, only very inefficient and dim LEDs were available in the green and, especially, in the blue. Although there are a handful of semiconducting materials with sufficiently wide bandgaps to function in principle in the blue region of the spectrum,

fundamental material properties and limitations have prevented bright and efficient diodes from being made. Recently, breakthroughs in the heteroepitaxial growth of gallium nitride (GaN) and its alloys with indium and aluminum have changed the blue and green LED technology outlook. Formerly, it was believed that III-V nitride layers had too high a defect density to function as LEDs. Nevertheless, a Japanese company (Nichia) has developed a family of blue and green LEDs based on GaN that are bright and efficient. For the last two years, Japanese companies have been manufacturing and selling blue GaN LEDs in bulk quantities. This project is a collaboration with Hewlett-Packard Company (HP), the leading producer of LEDs, to investigate the fundamental light-emitting mechanism. Epitaxial thin film growth, including specialized structures and doping series and basic parametric characterization, will be performed in the industrial research laboratories of HP. Highly homogenous, reliably reproducible, and stable metal organic vapor phase epitaxial growth processes have been established at HP laboratories. Highly specific spectroscopic characterization and analysis aimed at revealing the basic principles underlying doping and recombination will be performed by LBNL's Materials Sciences Division, supporting key DOE missions in materials research.

19. Title: Alloy Design of Neodymium (Nd₂Fe₁₄B) Permanent Magnets**ID:** ORL94-15**PI:** Joseph A. Horton
Metals and Ceramics Division**Phone:** 423 574-5575**Partner:** Magnequench International, Inc.
Anderson, IN**FY 97 Funding:** \$155K**Total Project Funds:** \$590K

The objective of this project is to improve the room-temperature fracture toughness and fracture strength of neodymium-based permanent magnets. Demanding motor applications (such as a prototype electric-vehicle drive motor with a 150 mm diameter neodymium magnet) need improvements in these properties to achieve acceptable failure rates for commercial use. This project is one of the first studies emphasizing mechanical properties of rare earth permanent magnets. Improvements in these properties should result from insights gained by correlation of microstructure with fracture properties, and by applying processing techniques similar to those applied to improve the properties of structural ceramics and brittle intermetallics. Based on a survey of (1) commercially available material produced by two different processes, melt spinning and powder metallurgy, and (2) experimental alloys produced by Magnequench, it has been determined that toughness is a

function of both grain size, and the composition and distribution of certain grain boundary phases. Processing variables were studied to optimize magnets with Magnequench's current composition and processing methods. A correlation of crystallographic texture, magnetic anisotropy, press direction, microstructure, and fracture toughness is underway in order to understand the fracture process and the preferred directionality of some of the failures. In collaboration with another Magnequench-DOE CRADA (with INEEL) and through the Division of Materials Sciences, Office of Basic Energy Sciences, Center for Synthesis and Processing of Advanced Materials project on Tailored Microstructures, a third processing route that utilizes gas atomized magnet powder was investigated, and bulk magnets were successfully produced. Insights from this work support DOE's basic science programs in alloy and ceramic science.

20. Title: Manufacturing of Ni-Base Superalloys with Improved High-Temperature Performance

ID: ORL95-05

PI: E. P. George
Metals and Ceramics Division

Phone: 423 574-5085

Partner: Allvac, an Allegheny Teledyne Company
Monroe, NC

FY 97 Funding: \$137K

Total Project Funds: \$540K

Nickel-base superalloys are state-of-the-art materials for high-temperature structural applications. The objective of this project is to enhance their manufacturing and performance through control of trace elements in the parts-per-million range without significantly increasing production costs. IN-718 alloy rods and plates doped with microalloying additions were fabricated at Teledyne, where it was shown that alloys doped with Phosphorus (P) and Boron (B) significantly improve creep rupture life. Using creep tests, the project has demonstrated that doping with optimum levels of B and P reduced the minimum creep rate by a factor of 28 and increased the activation energy for creep from 100 to 156 kcal/mole. Additionally, weldability tests demonstrated that the threshold-cracking stress increased from 14 ksi in commercial IN-718 to 18 ksi in the alloy doped with optimum B and P, indicating that these dopants rather than compromising weldability actually improved it. Using atom-probe analyses, the project has shown that B and P segregated strongly to the γ - γ grain boundaries in IN-718, but not to the γ - γ or γ - γ' interfaces. This suggests that the dopants were not likely to reduce the interfacial energies and slow down the coarsening kinetics of the strengthening particles.

Consistent with this, transmission electron microscopy measurements have revealed that B and P do not change significantly the size distribution of γ' particles in IN-718 during creep. Finally, aging studies have demonstrated that doping with B and P makes IN-718 more resistant to softening at elevated temperatures suggesting that their beneficial role may be related to pinning of dislocations or vacancies. Studies are planned to identify the detailed pinning mechanisms. Based on these results, Allvac has initiated efforts to prepare the first large heat (15000 lb.) of IN-718 and has held discussions with major aircraft engine manufacturers who expressed interest in establishing test programs critical to the commercialization of microalloyed IN-718. The knowledge gained through this project will enhance the Basic Energy Sciences (BES) Materials Sciences program's effort to understand the effect of minor-alloying elements on the mechanical behavior of high-temperature, ordered intermetallic alloys. It also strengthens ORNL's material processing capabilities that are part of DOE's BES, Energy Efficiency, and Fossil Materials R&D programs.

21. Title: Ion Implantation Processing Technologies**ID:** ORL94-72**PI:** Tony E. Haynes
Solid State Division**Phone:** 423 576-2858**Partner:** Lucent Technologies - Bell Laboratories
Murray Hill, NJ**FY 97 Funding:** \$136K**Total Project Funds:** \$395K

The objective of this project is to improve ion-implantation processing technologies in order to accelerate future development cycles for the manufacture of silicon integrated circuits. The approach involves experimental investigations of the behavior of point defects injected during the ion-implantation process and their interactions with impurities, with particular emphasis on transient-enhanced diffusion (TED) of dopants and gettering of contaminants. This project has produced (1) a physical model, based on boron-iron pairing, that quantitatively accounts for the gettering of iron in boron-implanted layers in silicon; (2) the tracking of the evolution of extended defects and dopant diffusion during annealing of implanted silicon; and (3) modeled and measured modifications of defect evolution and dopant diffusion as a function of implant conditions and specimen impurity. The need for ultra-shallow junctions with dopant profiles less than 50 nm deep fueled the thorough investigation of the dependence on implant energy down to 0.5 keV. It was found that the same physical processes dominate transient-enhanced diffusion at low energies, but with kinetics altered

by the larger concentrations of both point defects and dopants, as well as by increased interaction with the surface. These results have demonstrated that reducing implant energy can lead to elimination of TED through surface annihilation of excess interstitials. However, a chemically-induced diffusion enhancement has been detected for the first time and is the subject of a continuing investigation. Related studies of the clustering of implanted hydrogen in silicon have led to a patent application for a more efficient method for fabrication of thin silicon-on-insulator films. Since the microelectronics industry is dependent upon ion implantation to control location and concentration of dopants in integrated circuits, refinement of these processes and development of reliable, physics-based models for implantation-related processes are critical for continuing advances in this industry. This project provides experimental data that will enable the continued development of ion implantation processes in microelectronics manufacturing for at least the next 10 years. Since microelectronics underpins much of the nation's critical infrastructure in communications and transportation, this project provides essential support to the DOE national security mission as well as the science and technology mission through DOE's Basic Energy Sciences programmatic goals.

22. Title: The Role of Yttrium in Improving the Oxidation Resistance in Advanced Single Crystal Nickel-based Superalloys for Turbine Applications

ID: ORL95-07

PI: Kathleen B. Alexander
Metals and Ceramics Division

Phone: 423 574-0631

Partner: Pratt and Whitney
East Hartford, CT

FY 97 Funding: \$149K

Total Project Funds: \$320K

The focus of this project is the examination of the role of yttrium and other alloying elements on the microstructure and oxidation performance of improved single crystal nickel-based superalloys for advanced turbine applications. The microstructure and microchemistry of these alloys and their surface oxides are being measured with state-of-the-art microanalytical techniques (atom-probe field-ion microscopy and electron microscopy) and then correlated with burner rig and engine-test oxidation performance. Recent results indicate that cobalt, chromium, and rhenium are rejected from the γ' particles during growth with rhenium exhibiting significant compositional variations in the matrix phase near the γ' particles. These observations are relevant to the attainment of high-temperature creep strength in these alloys, since the sluggish diffusion of rhenium away from the

growing γ' particles will result in retarded precipitate growth rates. The overall technical goals include: (1) identifying the partitioning behavior of the elemental additions in these superalloys before and after burner rig and engine tests and the effect on the misfit energy between the phases in the alloys; (2) examining the oxidation performance of these newly-developed alloys; and (3) relating the microstructural observations to the observed performance. Anticipated improvements from these modified alloys include enhanced durability in the operating environments at the high temperatures required to improve energy efficiency. In addition, the availability of alloys capable of higher temperature operation will minimize the need for expensive coatings in some applications. These alloys are primarily used for the turbine components in engines which are exposed to the most extreme temperatures. These studies are relevant to both commercial land-based (energy-production) and advanced aircraft turbines used by a wide range of U.S. industries. This project supports DOE missions through increased energy efficiency and reduced fuel consumption. It also strengthens DOE's basic materials programs.

23. Title: New Thermoelectric Materials**ID:** ORL95-10**PI:** Brian C. Sales
Solid State Division**Phone:** 423 576-7646**Partner:** Marlow Industries
Dallas, TX**FY 97 Funding:** \$150K**Total Project Funds:** \$700K

The goal of this project is to develop materials that will improve the performance of thermoelectric devices for solid-state refrigeration and power generation. Thermoelectric devices have no moving parts, use no greenhouse gases, and are extremely reliable. The poor efficiency of commercial thermoelectric devices, however, has restricted their use to applications in which reliability or convenience are more important than economy. ORNL has evaluated several classes of compounds as possible advanced thermoelectric materials, including materials with the filled-skutterudite crystal structure, and unusual "Kondo-like" metals and semiconductors. Marlow Industries has participated in the material selection process, has provided technical guidance on thermoelectric materials and concepts, and will fabricate thermoelectric-refrigeration devices for a more complete evaluation of promising materials. Based on the current experimental data, the most promising approach to designing improved thermoelectric materials is the synthesis of new materials with crystal structures

that contain atomic-size cages into which "rattling" atoms or molecules can be inserted. The project has demonstrated that for the filled skutterudite antimonides, this approach results in materials with thermoelectric efficiencies at elevated temperatures (700-1000K) that are as good or better than all previously investigated materials. Current efforts are aimed at using this same approach to create improved thermoelectric materials that operate at and below room temperature. In addition to enhancing the DOE's basic materials sciences programs through an improved understanding of these unusual materials, a specific area of interest to DOE is in the automobile and truck industry where thermoelectric generators could be used to convert exhaust and engine heat to useful electrical power. Recent developments in DOE-sponsored research in new superconductors and superconducting electronics would also benefit from a thermoelectric refrigerant that could cool the electronics below the superconducting transition temperature using only the flow of electrons or holes through the device.

24. Title: Atomic Scale Structure of Ultrathin Magnetic Multilayers and Correlation with Resistance, Giant Magnetoresistance, and Spin-Dependent Tunneling

ID: ORL97-03

PI: William H. Butler
Metals and Ceramics Division

Phone: 423 574-4845

Partners: Honeywell, Inc.
Plymouth, MN

Nonvolatile Electronics, Inc
Eden Praire, MN

FY 97 Funding: \$100K

Total Project Funds: \$750K

Giant Magnetoresistance (GMR) and Spin-Dependent Tunneling (SDT) are two recently discovered phenomena that are providing important new insights into how spin affects the transport of electrons in materials. These phenomena have the potential to spark revolutionary advances in several important technologies and both require the controlled deposition of ultrathin films. In order to realize the scientific and technological potential of these phenomena, it is necessary to relate the spin-dependent transport properties to the spin-dependent electronic structure of the deposited structures. Since spin dependent transport is very sensitive to structure at that scale, an understanding of the deposited structures at the atomic scale is required to accomplish that goal. Recent advances in electronic structure theory allow the calculation of spin-dependent transport. The missing key, however, is atomic-scale characterization of the deposited films. Through a close collaboration

between theory and experiment, the objective of this project is to determine the physical, chemical, and magnetic structure of GMR and SDT films and to relate their structure to their magnetic and transport properties. This will be achieved by combining a uniquely powerful set of characterization tools, (X-ray Reflection and Diffraction, Atom- Probe Microscopy, Z-Contrast Electron Microscopy with Electron Energy Loss Spectroscopy, and Electron Holography) with first-principles computer codes that are capable of calculating the spin-dependent conductivity for realistic systems. The industrial partners (Honeywell Solid State Electronics Center and Nonvolatile Electronics Inc.) are uniquely qualified to optimize their deposition processes and to relate the structures they deposit to the observed spin-dependent transport. Success in this project should lead to better read sensors for magnetic disk drives, a new type of non-volatile radiation resistant magnetic random access memory device, and better position and motion sensors for numerous industrial, transportation, and consumer product applications. Additionally, this work enhances DOE's basic materials sciences programs in magnetic structures and advanced characterization methods.

INTELLIGENT PROCESSING/MANUFACTURING RESEARCH**Sensors, Instrumentation, and Processing****25. Title: A Microspectroscopy Facility for New Infrared Imaging Materials****ID:** BNL94-60**PI:** Gwyn Williams
National Synchrotron Light Source**Phone:** 516 344-7529**Partner:** Grumman Corporation
Bethpage, NY**FY 97 Funding:** \$91K**Total Project Funds:** \$300K

In this project, a synchrotron beamline at the National Synchrotron Light Source (NSLS) is being dedicated to infrared (IR) microspectroscopy. Grumman will use the facility to study materials for better (IR)-sensing and imaging systems for both civilian and defense markets. A cooperatively built research facility will permit microanalysis of materials for better IR sensors and will add an important capability for research on other materials and processes. Synchrotron IR light is needed for such studies because it gives a spatial resolution of a few micrometers, which enables it to image the structures used in most new materials for IR imaging (10 - 100 micrometers). It also matches the spectral range in which such materials operate. With the new equipment, researchers will be able to observe even the smallest micro-structures within these materials; with such data, they can improve the structures for better performance. The new beamline is the fifth IR beamline at the NSLS, a facility known for pioneering work with IR synchrotron radiation. Staff at the NSLS designed and fabricated the hardware to produce the beam, while staff at Grumman's Advanced Technology Development Center made the instrumentation necessary to perform IR microspectroscopy. The heart of the new facility is an infrared microspectrometer, a conventional instrument modified by Grumman to use the synchrotron light source. Specialized instrumentation and methods developed under this project will benefit ongoing materials research at the NSLS and support the DOE's mission in materials research. In particular, the new facility offers novel time-resolved spectroscopic capabilities for solid-state physics. It also has great potential for unique microanalysis in other areas, notably biology, polymers, pharmaceuticals, laminate and composite structures, and semiconductors.

26. Title: Development of a 10-Bit 500 Msample/sec Waveform Digitizer**ID:** BNL95-05**PI:** Maged Atiya
Physics Department**Phone:** 516 344-3067**Partner:** LeCroy Research Systems
Chestnut Ridge, NY**FY 97 Funding:** \$143K**Total Project Funds:** \$526K

The goal of this project is to combine technological advances in analog-to-digital conversion, recently introduced by LeCroy Research Systems (LRS), with the proven long term experience of BNL in high speed digital technology. LRS has been a leader in ultra-high speed test instruments for the past 10 years. The BNL group has built and operated (since 1988) one of the first high speed (8-bits 500-MSample), high density, fast read-out waveform digitizer systems. LRS and BNL have constructed a prototype single MCM (multi-chip module) containing the digitizer and memory which is currently under test and will become the main component of a standard commercial product. The technology of high speed analog-to-digital conversion (and the associated technology of digital-to-analog conversion) is at the heart of many of the current civilian-sector innovations, such as high definition television, multi-media, cellular communication, and high speed networks. The U.S. has been an undisputed leader in this area, holding a dominant position in both R&D and manufacturing. While there is increasing competition from foreign sources at the low-end of performance, an emphasis on the high performance devices should help retain this leadership. DOE laboratories, such as BNL, as well as industrial partners such as LRS, have long been active in this technology. This activity has been largely used to support the construction of instrumentation for basic research. The transfer of this technology to the industrial sector would represent a logical and beneficial step in the evolution of this technology. Additionally, by embedding high speed analog-to-digital conversion technology in systems capable of interfacing to standard high-speed computer buses, the benefits of this project will reach further into the industrial sector. This project supports the DOE mission in development of infrastructure for high speed instrumentation coupled with scientific computing.

27. Title: Development of Multi-Channel ASICs for CdZnTe Gamma Detector Arrays**ID:** BNL97-06**PI:** Paul O'Connor
Instrumentation Division**Phone:** 516 344-7577**Partner:** eV Products Division of II-VI, Inc.
Saxonbury, PA**FY 97 Funding:** \$82K**Total Project Funds:** \$651K

The objective of this project is to develop an X-ray imaging module consisting of a multi-element Cadmium Zinc Telluride (CZT) detector and a CMOS application-specific integrated circuit (ASIC). The module will detect x and gamma rays in the energy range from 20 - 150 keV in the photon counting mode. The electronics must be compatible with the CZT detector characteristics and at a minimum preamplify and shape the pulse signals from the detector elements. There is currently a large need for solid state gamma and X-ray imaging capability for both medical and industrial applications. In industry, a need exists for imaging food products for foreign matter, non-contact, high speed weighing of consumer products to ensure minimum weight compliance and multi-energy high speed imaging of manufactured products to detect subtle defects. Solid state CZT arrays offer the possibility of reducing the weight and bulkiness of existing nuclear medicine cameras based on NaI scintillators and photomultiplier angular camera technology. Small hand held imaging devices show promise for locating cancer tissue during surgery through the use of monoclonal antibodies tagged with radioactive tracers. For security screening, CZT arrays when used with multi-energy X-rays can detect explosives and other contraband. The technical approach consists of the evaluation of the performance of various existing BNL circuits with CZT arrays, adapting the existing design so that the front end and shaping parameters are ideally matched to the CZT detectors, producing a prototype array of a certain size by tiling arrays and ASIC circuits, developing a 64 or greater channel ASIC for larger substrates (or finer pitch), and configuring existing building blocks to build a multiplexing and image processing ASIC. The development of these solid-state detectors will benefit DOE mission areas in time-resolved X-ray crystallography, nuclear medicine, and extended X-ray absorption.

28. Title: Development of an In-Situ Scanning Surface Profiler**ID:** BNL95-07**PI:** Peter Z. Takacs
Instrumentation Division**Phone:** 516 344-2824**Partner:** Continental Optical
Hauppauge, NY**FY 97 Funding:** \$60K**Total Project Funds:** \$357K

The goal of this project is to extend the capabilities of the Long Trace Profiler (LTP) to develop a new surface profiling instrument that can be used to measure the distortion produced in X-ray mirrors exposed to high power synchrotron radiation (SR) beams. The standard LTP is used to measure the shape of mirrors in a controlled, stable laboratory environment. The new system will be able to measure mirrors in situ as they are exposed to the high heat loads from powerful X-ray beams generated by third-generation SR sources, such as the Advanced Photon Source (APS) at Argonne National Laboratory (ANL) and ELETTRA at Sincrotrone in Trieste, Italy. The In-Situ LTP will provide valuable information to SR beam line scientists to enable them to utilize the powerful X-ray beams in the most efficient manner. A prototype system has been built and is currently being installed on a beam line at the APS at ANL. Initial results are expected from tests to be conducted in September 1997. The development of the LTP support instrumentation program for high energy physics is a critical DOE mission area.

29. Title: Microcircuits and Sensors for Portable, Low-Power Data Collection and Transmission**ID:** BNL97-07**PI:** Paul O'Connor
Instrumentation Division**Phone:** 516 344-7577**Partner:** Symbol Technologies, Inc.
Holtsville, NY

FY 97 Funding: \$125K**Total Project Funds: \$750K**

The objective of this project is to design, fabricate and test two novel devices for data collection and transmission: an optical photosensor array and a 2.4 GHz, single-chip, frequency-agile radio transceiver. Both devices can be processed in a standard CMOS integrated circuit process. CMOS technology has advanced to the point where many conventional electronics systems can be fully integrated on a single chip. Up to now the vast majority of these chips perform purely electronic functions. In this project we propose to investigate two integrated circuits with sensors which can process information in the form of radio-frequency waves and optical images. Our project goal is to develop an inexpensive single-chip frequency agile RF transceiver operating at the 2.4 GHz range - a universally accepted unlicensed band - with data rates up to 250 Kbps and an approximate range of 50 feet. We have previously demonstrated successful circuit blocks and will be seeking to use a higher performance CMOS 0.35 micron process to achieve lower power consumption and higher integration density. For the optoelectronic imaging portion of this project, we will investigate the "active pixel" architecture. This architecture uses photodiode sensors which can be made in a native CMOS process. The imaging array requirements (pixel size and count, spectral responsivity, speed, signal-to-noise ratio, dynamic range, linearity, crosstalk, and power consumption) will first be specified based on a knowledge of the 2D bar code reader system. We plan to model alternative photodetectors using the semiconductor device simulation codes currently used at BNL for silicon radiation detector development. This project supports the DOE mission in advanced semiconductor research for development of crosscutting sensor technologies.

30. Title: Ion Source and Beam Control Technologies for Lithography**ID:** LBL95-16**PI:** Ka-Ngo Leung
Accelerator and Fusion Division**Phone:** 510 486-7918**Partner:** Advanced Lithography Group
Columbia, MD**FY 97 Funding: \$51K****Total Project Funding: \$1,050K**

The goal of this project is to develop technologies for a hydrogen or helium ion beam projection lithography machine. Hydrogen or helium ion beam projection lithography can be used in creating extremely small (< 0.18 um) features in the next generation of integrated circuits. In order to

maintain the chromatic aberrations below 25 nm, an ion source which delivers a beam with an energy spread of less than 3 eV is required. In the past two years, the Ion Beam Technology (IBT) Group at the LBNL has been collaborating with the Advanced Lithography Group (ALG) in developing an ion source that can meet this requirement. This project has met or exceeded project goals. Work has progressed on this project with the goal of reducing the axial energy spread to less than 1 ev. This was approached by two methods. First, more work was done optimizing the ALG style multicusp/filter ion source. This provided energy spreads of below 2 ev. The second approach was to design and fabricate a new ion source with a coaxial filter arrangement. Preliminary tests are very promising with axial energy spreads measured in the 1 ev range. It is believed that with optimization the energy spread can be maintained below 1 ev. The research collaboration supports the DOE mission in advanced electronic processing methods.

31. Title: Development and Commercialization of a Carbon Monoxide Occupational Dosimeter

ID: LBL95-30

PI: Lara Gundel
Environmental Energy Technologies Division

Phone: 510 486-7276

Partner: Quantum Group, Inc.
San Diego, CA

FY 97 Funding: \$101K

Total Project Funds: \$300K

In this project, Quantum Group, Inc. (QGI) is working with LBNL to develop an inexpensive passive carbon monoxide (CO) occupational dosimeter. CO is an unwanted by-product of energy production by fossil-fuel combustion and is one of the most deadly environmental pollutants encountered in indoor and occupational settings. Occupations such as forklift operators, automobile mechanics, bus drivers, toll booth operators, and parking garage attendants are all exposed to elevated levels of carbon monoxide. Many deaths occur every year from workers using gasoline-powered tools (e.g., cement cutters, power washers) in poorly-ventilated spaces. One obstacle in the way of identifying and mitigating high carbon monoxide environments is the lack of an inexpensive but sensitive and accurate CO monitoring device such as a passive carbon monoxide dosimeter. By the start of this project, the first proof-of-concept passive dosimeter had been designed. During the project, three new passive dosimeters have been designed and tested. The final design has achieved control of sensor reversibility, humidity dependence and sensor variability. Preliminary field studies have produced

positive results. A prototype dosimeter has been developed to the stage where hundreds of units can be assembled, so that extensive interference testing and field validation can be conducted.

32. Title: Prevention/Elimination of Metal-Water Explosions in Aluminum Casting Pits

ID: ORL92-05

PI: Rusi P. Taleyarkhan
Engineering Technology Division

Phone: 423 576-4735

Partner: Aluminum Association
Washington, DC

FY 97 Funding: \$174K

Total Project Funds: \$440K

Metal-water or steam explosions in aluminum industry casting pits have caused numerous injuries and fatalities, and significant damage and destruction of infrastructure over the past fifty years. Traditionally, industry has attempted to prevent explosions by using an empirical-based approach involving the coating of sensitive surfaces with materials like Tarset Standard (TS). However, due to environmental concerns and other reasons, TS is being discontinued from production, leaving industry with the task of evaluating and finding alternate materials. As part of this project with the Aluminum Association (AA), ORNL is investigating how steam explosions initiate over specific surfaces, and what other coatings and novel methods may be appropriate as alternatives to TS. Work completed has resulted in the development and validation of a unique apparatus that, at significant cost reduction, accurately and rapidly simulates in a laboratory environment the interaction of molten aluminum contacting various submerged surfaces without attendant safety problems associated with field experiments involving molten aluminum pours in water-filled containers. While further testing and theoretical model development still need to be done, unprecedented insights have been obtained on key phenomenological issues. This has enabled the development of a novel approach for conclusive explosion prevention. A patent has been filed and granted for this novel, environmentally-friendly, cost-effective approach based on knowledge of fundamentals of the physics of explosion initiation. Field demonstrations are planned. This project supports DOE's energy-related mission to develop a more energy-efficient metal-casting operation. Additionally, this work is expected to provide a better physical understanding of entrapment-boiling heat transfer that could be applied to safety improvements in DOE and commercial nuclear reactors.

33. Title: Ultra-Precision Automated Measurement for Manufacturing**ID:** ORL95-08**PI:** C.E. Thomas
Instrumentation and Controls Division**Phone:** 423 574-1155**Partner:** InterScience, Inc.
Troy, NY**FY 97 Funding:** \$75K**Total Project Funds:** \$255K

The objective of this project is to demonstrate a new level of automated process control, non-contact measurement technology for the United States manufacturing sector. The immediate goal is proof of concept for intelligent, automated-electronic inspection of wafers for digital microchips with a resolution better than the lithographic mask resolution (e.g., a transverse resolution of 200 nanometers and a longitudinal resolution of 3 nanometers). This will allow automated three-dimensional inspection of the chips between processing steps to ensure success of the processing at each step; immediately identify process failures; save time, money, and energy; improve quality and yield by identifying wafers for re-work early in the processing (before etch); and immediately identify process failures. The intent is to provide a totally automated, rapid, on-line inspection capability to detect and sort defective wafers or call for human intervention. Technological developments in the optical, laser, electronics, and computer sciences have made it possible to realize a new generation of intelligent automated-measurement process control. High-coherence single-mode lasers, diffraction-limited optics, and high-resolution CCD cameras, combined with state-of-the-art computer control, data acquisition, and data analysis, will be utilized to demonstrate proof of concept for automated ultra-precision measurements. The present approach is to acquire purely digital holograms. Extended Fourier transform algorithms, invented at ORNL, allow complete analysis for phase and amplitude of the object wave for every pixel (i.e., the object is recorded in three dimensions). Pure digital-hologram acquisition has been demonstrated, and preparation is underway to perform proof of concept experiments. The technology is very near the critical break-through point for exponential growth as an inspection technology. In fact, several organizations have expressed strong interest in the final proof of concept. Rapid, ultra-precision measurement technologies enhance DOE programs in (1) energy efficiency through the development of high-tolerance, high-performance parts; (2) national defense through intelligent manufacturing and more accurate, in-situ monitoring of dimensional changes; and (3) science and technology through the development of ultra-precision measurements and technologies for a wide range of research programs.

34. Title: An Implantable Drill Bit Monitor for Use in Oilfield Drill Bits**ID:** ORL95-13**PI:** David E. Holcomb
Instrumentation and Controls Division**Phone:** 423 576-7889**Partner:** Hughes Christensen
The Woodlands, TXHouston Applied Research Center
Houston, TX**FY 97 Funding:** \$192K**Total Project Funds:** \$680K

The purpose of this project is to develop an incipient failure prediction system to be incorporated into production roller-cone, oilfield drill bits. The cost of catastrophic bit failure can exceed \$1,000,000 per well and up to a worst case of \$5,500,000 per well. Drill bit failure can also leave chunks of metal at the bottom of the well bore that must be extracted before drilling can continue. To avoid these incidences, drill bits are often replaced with significant remaining life. The time costs associated with early bit replacement (drill rigs cost up to \$10,000/hr and bit replacement times can exceed a day) are a significant portion of the cost of well drilling. The space available within the drill bit, harsh downhole environment, power available, and cost of implementation all restrict monitor development. Because of these challenges, electronics have never before been directly placed within a rock bit. However, drill bit journal bearing temperature was selected as the primary impending failure indicator. The parameter selection was confirmed in the field by monitoring bit journal bearing temperature while drilling with known good and near failure bits. A novel temperature-measurement scheme (patent pending) has been developed that does not require high-gain amplification or a stable current source, requires very little power, and directly produces a digital result. The new measurement scheme has been implemented in the form of electronic modules incorporating two custom application-specific integrated circuits. These modules have been successfully used while drilling. Effort continues to develop a definitive impending failure temperature signature and the up-hole impending failure signal technique (patent in process). In addition to ensuring the reasonable cost availability of petroleum to the US market, the temperature monitor developed in this project will have wide applicability to improving energy efficiency including: truck brake overheating, food processing quality assurance, remote environmental sensing, heat-treatment furnace monitoring, and reentry vehicle thermal-shield monitoring.

35. Title: Microfabricated Instrumentation for Chemical Sensing in Industrial Process Control**ID:** ORL97-08**PI:** J. Michael Ramsey
Chemical and Analytical Sciences Division**Phone:** 423 574-5662**Partner:** Waters Corporation
Milford, MA**FY 97 Funding:** \$99K**Total Project Funds:** \$747K

The monitoring of chemical constituents in manufacturing processes is of economic importance to most industries. The monitoring and control of chemical constituents may also be of importance for product quality control or, in the case of process effluents, of environmental concern. The most common approach now employed for chemical process control is to collect samples which are returned to a conventional chemical analysis laboratory. The objective of this project is to demonstrate the use of microfabricated structures, referred to as "lab-on-a-chip" devices, that accomplish chemical measurement tasks that emulate those performed in the conventional laboratory. The devices envisioned could be used as hand portable chemical-analysis instruments where samples are analyzed in the field or as emplaced sensors for continuous "real-time" monitoring. This project will focus on the development of filtration elements and solid phase extraction elements that can be monolithically integrated onto electrophoresis and chromatographic structures pioneered at ORNL. Successful demonstration of these additional functional elements on integrated microfabricated devices will allow lab-on-a-chip technologies to address real world samples that would be encountered in process-control environments. The resultant technology will have broad application to industrial environmental monitoring problems such as monitoring municipal water supplies, wastewater effluent from industrial facilities, or monitoring of run-off from agricultural activities. The technology will also be adaptable to manufacturing process control scenarios. This project supports DOE missions in environmental quality and energy efficiency.

36. Title: High Sensitivity Electrospray Ion Source Development**ID:** PNL94-15

PI: Richard D. Smith
Environmental and Molecular Sciences Division

Phone: 509 376-0723

Partner: Finnigan MAT
San Jose, CA

FY 97 Funding: \$110K

Total Project Funds: \$495K

The purpose of this project is to improve the understanding, and the actual performance, of the transport and focusing of charged particles that are formed at atmospheric pressure through the interface to the high vacuum region of a mass spectrometer. This improved understanding should lead to the development and commercialization of an improved electrospray ion (ESI) source for use in conjunction with mass spectrometers. In the later stages of this project a totally new concept for ion focusing was developed that promises to greatly exceed programmatic goals. The "ion funnel" concept provides the basic approach for focusing ions dispersed at high gas pressures, and should result in a breakthrough in ESI ion source technology, as well as in most other modern ion sources. It should also have a major impact on the operation and performance of other mass spectrometers and devices that depend on the measurement of ion currents. The ion funnel provides additional benefits that, for the case of mass spectrometry, include allowing better differential pumping due to the improved focusing through apertures (thus allowing smaller, less expensive pumps to be used) and better control of ion introduction (providing better resolution performance). Implementation of this novel "ion funnel" approach is the focus of current efforts. Initial results have shown that an approximate two orders of magnitude increase in sensitivity is achieved using the ion funnel in a direct comparison with the best conventional technology. The application of this technology is projected to benefit biological, chemical, and environmental research conducted by the DOE laboratories and others, as well as the rapidly increasing medical and clinical applications of mass spectrometry.

37. Title: Development of a Near-Field Optical Microscope and Spectrometer

ID: PNL94-20

PI: Sunney Xie
Environmental and Molecular Sciences Division

Phone: 509 375-6882

Partner: Digital Instruments, Inc.
Santa Barbara, CA

FY 97 Funding: \$117K

Total Project Funds: \$595K

Optical trapping by highly focused laser beams has been extensively used for the manipulation of submicron-size particles such as biological structures. Such manipulation is useful for fundamental biological research supported by DOE and in the development of new biotechnology. Conventional optical tweezers rely on the field gradients near the focus of a laser beam, which give rise to a trapping force toward the focus. The trapping volume of these tweezers is diffraction limited. Near-field optical microscopy enables optical measurements at a dimension beyond the diffraction limit. This project has started to extend the near-field approach to high-resolution optical tweezers that can potentially be applied in biological and other types of research. The nanometric optical tweezers being developed in this project offer the following potential advantages: (1) highly confined evanescent fields significantly reduce trapping volume, (2) large field gradients result in a larger trapping force, and (3) field enhancement reduces illumination power and radiation damage to the sample. In 1997, a rigorous electromagnetic analysis of the field enhancement was performed. To solve Maxwell's equations in the specific geometry of the tip, the multiple multipole method was used. The trapping force at different center positions of the particle was also calculated. The results of this analysis indicate the feasibility for trapping on a nanometer scale at a moderate power level. A practical concern is heating of the tip, which could damage the sample and induce convection. Results of a finite-difference time-domain study indicated that the temperature rise induced by laser heating is minimal for the intensity level required for stable trapping. Experimental realization of the near-field optical tweezers is underway. It has the potential for nanometric manipulation of individual biomolecules in an aqueous environment and control of their chemical activities, which could contribute to the development of new bioremediation methods. In related project work, high-quality two-photon fluorescence images of immobilized single fluorophores on polymethyl methacrylate films and photosynthetic membrane fragments were obtained. The signal-to-background ratio was as high as 30:1, and the full width at half maximum of a single-molecule peak was significantly shorter than that for one-photon excitation. In process is the combination of two photon fluorescence imaging with a near-field approach using the metal tip in order to achieve a 10 nm spatial resolution.

Fabrication**38. Title: Microfabrication of a Multi-Axis Micro-Accelerometer Using High Aspect Ratio Microfabrication and Silicon Micromachining****ID:** BNL94-02**PI:** John B. Warren
Instrumentation Division**Phone:** 516 344-4203**Partner:** Loral Control Systems
Archbald, PA**FY 97 Funding:** \$100K**Total Project Funds:** \$600K

The goal of this project is to use microfabrication methods developed at BNL to improve the performance and reduce production costs of a multi-axis accelerometer originally constructed and tested by Loral Control Systems. In its original configuration, the accelerometer can simultaneously detect accelerations along three translational and three rotational axes, a capability that is unique for an instrumental package that has a volume of about 8 in³. Loral's original prototype consisted of a precision-machined cubical enclosure that contains 18 levitation coils and 36 capacitive sensors. The primary goal of this project is to use high aspect ratio microfabrication to fabricate in bulk all of these components, thus minimizing precision machining steps and greatly reducing production cost. In addition, the inherent accuracy of the microfabrication (based on lithography) should lead to additional improvements in performance. The completed micro-accelerometer will have many applications in commercial aviation, auto navigation, active automotive suspension system control, drillbit navigation, and airbag deployment. The high aspect ratio microfabrication process has many scientific applications and is currently being used at BNL to construct prototype position-sensitive X-ray detector arrays that have applications in high energy physics. Knowledge gained from this project concerning microfabrication methods is directly applicable to these on-going efforts. In the past year, research has concentrated on the development of high aspect ratio resists that can be adapted for levitation coil fabrication, the most cost-critical component of the sensor. It has been conclusively demonstrated that a new epoxy-based resist (SU-8, manufactured by Dupont) enables the fabrication of microstructures with aspect ratios of 15 to 1. This is an improvement of almost a factor of three over traditional ultraviolet based resists and is directly competitive with the traditional LIGA process (X-ray based lithography requiring synchrotron exposure). Further development of this resist system will lead to significant cost reductions in the fabrication of this sensor, and many

new microfabrication applications as well. This project supports DOE missions in material processes and electronics.

39. Title: Development of an Aluminum Bridge Deck System**ID:** ORL94-56**PI:** H. Wayne Hayden
Metals and Ceramics Division**Phone:** 423 574-6936**Partner:** Reynolds Metals Company
Richmond, VA**FY 97 Funding:** \$105K **Total Project Funds:** \$800K

The purpose of this project is to develop and eventually commercialize an aluminum bridge-deck panel system based on the use of aluminum multi-void extrusions which are welded to make panel sections and coated with an aggregate/epoxy composite as the pavement wear surface. The panels are then bolted in place to existing bridge sub-structure. Work at ORNL has concentrated on critical issues affecting the performance life of decks under projected conditions of loading and exposure temperature. ORNL studies during the first year demonstrated acceptable fatigue lifetimes of bolted joints; no undue residual-stress development in the vicinity of welds; non-destructive evaluation (NDE) procedures for weld and pavement surface bonding; and the role of monotonic and fatigue loading on delamination of the wear surface. ORNL studies are now concentrating on establishing tensile stress and interfacial shear-stress limits for the wear surface/aluminum bond and fatigue loading behavior of welded sections. The desired results are to begin the upgrade of deficient bridges throughout the U.S. with the use of aluminum bridge decks, and to use aluminum decks on new bridges. Two replacement bridge decks, one in Pennsylvania and one in Virginia, using the Reynolds decks, are now in operation and are generating interest from many state highway departments as well as many non-US concerns. This project clearly enhances DOE missions in transportation technology, infrastructure, energy conservation, and environmental quality.

40. Title: Rapid Prototyping of Ceramics**ID:** ORL94-95**PI:** Robert J. Lauf
Metals and Ceramics Division

Phone: 423 574-5176

Partner: Conway
Charlotte, NC

Argonne National Laboratory
Argonne, IL

FY 97 Funding: \$153K

Total Project Funds: \$634K

The goal of this project is to develop a fundamental understanding of computer data file conversion, resin mold fabrication using a stereolithography apparatus (SLA), and ultraviolet (UV) curing of gelcasting-type ceramic slurries so that these tools can be used in place of the neat resin to directly produce sinterable ceramic green bodies. Conversion programs will be identified or developed to convert a variety of object description files, such as engineering CAD/CAM files and medical Computed Tomography (CT) or Magnetic Resonance Imaging (MRI) files, to the appropriate SLA layered structure files. The ultimate goal is the ability to rapidly convert a computer file to a finished, net-shaped ceramic component. Critical tasks include: successful file conversion; development of compatible SLA mold materials and gelcasting slurries; and development of precisely controllable UV-cured gelcasting-type ceramic slurries. Accomplishing these goals will provide the ability to rapidly fabricate complex-shaped ceramic components. The potential applications for this technology are numerous and varied, such as biomedical implants and numerous automotive components. For example, biomedical components can be custom fit to the individual patient during the fabrication process, assuring precise fit while eliminating the need for "final finishing" during the surgical procedure. Additionally, applications are anticipated in the electronics industry and solid oxide fuel cells. Progress during the past year has included (1) the identification of software that can convert MRI files to SLA files; (2) the use of two different resin materials for molds fabrication with a 3-D Systems SLA-250 machine; (3) the evaluation of these resin molds for compatibility with the gelcasting chemical systems and processes presently used; and (4) the selection of UV sensitive initiators that were shown to successfully form gels with neat monomer solutions and with ceramic-loaded gelcasting-type slurries. Optimization of the amount of initiator and UV irradiation will be determined for the various material systems, fabrication techniques, and component configurations that are required for producing different ceramic products. This project enhances DOE's effort in advanced manufacturing and an ongoing Office of Transportation Technologies ceramics program. It also creates a broad capability to make single or low-volume ceramic components that will speed the development of better optimized gas turbines and other energy-saving machinery.

41. Title: Novel Methods for Fabrication Cost Reduction of Pressure Infiltration Cast Metal Matrix Composite Components**ID:** ORL95-01**PI:** James G. Hansen
Engineering Technology Division**Phone:** 423 241-2102**Partner:** Metal Matrix Composite Castings, Inc.
Waltham, MA**FY 97 Funding:** \$192K **Total Project Funds:** \$760K

The goal of this project is to produce low cost, high quality metal-matrix composite components via a high production rate process. The approach is to pressure cast near-absolute net-shape components by using inexpensive, nonstructural tooling. Metal matrix composite components are manufactured by first forming a ceramic preform and then infiltrating the preform with molten aluminum using Metal Matrix Cast Composites (MMCC), Inc. patented pressure-infiltration casting technology. The overall objective is to demonstrate cost-effective manufacturing technology on composite material with the strength and stiffness of cast iron and the weight of aluminum. Critical tasks are to tailor gelcasting process parameters and slurry rheology to enable the fabrication of defect-free preforms from MMCC specified powders for a low-cost manufacturing process, demonstrate inexpensive tooling, characterize the mechanical behavior of metal-matrix composites, and evaluate the performance of a brake caliper. This project supports DOE programs by developing technology to manufacture lightweight automobile parts, thereby reducing vehicle energy consumption. The high-production rate of pressure cast composites can dramatically increase introduction of metal matrix composites into the automotive market. The immediate application of the technology is to automobile brake components. For example, by replacing a Lincoln Town Car's cast-iron brake calipers with aluminum matrix composite, the total brake caliper weight is reduced by 60% with no loss of stiffness. Project accomplishments include (1) tailoring ORNL's patented gelcasting technology to produce preforms packed to about 50% by volume with inexpensive alumina particles; (2) developing slurry rheology to produce stable gelcasting slurries with pseudo-plastic behavior that avoids particle settling problems; (3) developing mold releases that eliminate adherence of gelcast preform to mold walls; (4) demonstrating techniques for filling simple molds with a patented hydraulic-casting apparatus; (5) designing nonstructural tooling and using it to manufacture calipers with excellent surface finish and dimensional control; (6) evaluating numerous batches of cast composite material by modulus of rupture, tensile, and fracture toughness tests and modeling with constitutive theories; and (7) fabricating test fixture and testing a cast-iron caliper as a reference.

42. Title: Polymer Multilayer (PML) Film Applications in Optics, Electrolytes, and Glazings**ID:** PNL94-06**PI:** John Affinito
Materials and Chemical Sciences Division**Phone:** 509 375-6942**Partner:** E.I. du Pont de Nemours and Company
Wilmington, DESigma Laboratories
Tucson, AZUnited Solar Technologies, Inc.
Olympia, WA**FY 97 Funding:** \$200K**Total Project Funds:** \$716K

This project addresses the requirements of a number of industries for a much higher rate, and much lower cost, process for vacuum deposition of dielectric and/or electrolyte films, where the process is also compatible with conventional evaporation or sputtering vacuum deposition techniques. The **Polymer MultiLayer (PML)** deposition technology, being developed at PNNL, can meet these requirements and provide great competitive advantage to manufacturers of: multilayer optical filters; energy conserving window films; dyed window films; electrochromic, switchable windows and mirrors; rechargeable lithium polymer batteries; non-linear optical devices; EMF shielding; and solar concentrator technology. While the technology being developed in this project will benefit many industries, three different applications relevant to achieving DOE missions are under investigation: (1) multilayer, PML-only reflectors; (2) PML/silver solar thermal control glazings; and (3) advanced PML/silver and PML/aluminum solar reflector materials. In roll-to-roll vacuum deposition onto a flexible polyester substrate, the project has demonstrated: all PML reflectors with greater than 97% reflectivity; PML/metal/PML reflectors with (1-3)% higher reflectivity than metal films alone; excellent PML-to-silver and silver-to-PML adhesion; and PML/oxide optical interference filters. This project has also contributed to the development of PML/oxide barrier coatings with three orders of magnitude lower permeation than current food packaging industry goals. All of these can be fabricated in a continuous, in-line, roll-to-roll vacuum deposition process, at very high rate, using low cost acrylic monomers as starting materials. Additionally, as a result of this work, there have been: 12 publications, two licenses, an R&D 100 Award, and a Federal Laboratory Consortium Award for Technology Transfer. This project supports the DOE mission for improved material applications.

43. Title: Development of Tape Calendering Technology for Separation Membranes**ID:** PNL95-04**PI:** Timothy Armstrong
Materials and Chemical Sciences Division**Phone:** 509 375-3938**Partner:** Air Products
Allentown, PA**FY 97 Funding:** \$257K**Total Project Funds:** \$750K

The purpose of this project is to develop tape calendering technology to produce passive oxygen separation membranes from mixed conducting ceramics, and to transfer that technology to Air Products. In support of DOE missions, this project can contribute to major energy conservation and cost savings in two ways. First, successful development of passive oxygen separation membranes will directly reduce the amount of energy used in producing purified oxygen. Second, indirect savings will be achieved by reducing the energy used in other industrial processes, as a result of reducing the cost for purified oxygen that may be used in these processes. In order to successfully develop these membranes, methods are needed to produce membrane structures that balance high flux requirements and robust mechanical properties. Tape calendering combines oxide powders, binder, and plasticizer in a high-intensity mixer. The binder-plasticizer system can be softened by externally heating the mixing chamber, using only internal heating resulting from frictional forces generated within the mixing chamber, or combinations of the two. The softened binder system mixes with the ceramic powder to form a plastic-like mass. The mass is calendered into a thin, flat tape using a two-roll mill with counter rotating rolls. Tape thickness is controlled by the spacing of the two rolls. Tape calendering technology shows exceptional promise as a means to manufacture complex ceramic structures on a large scale and at low cost. If successful, this project could provide key technology that would help Air Products produce large quantities of oxygen at a significantly lower cost (40-50%) than current cryogenic methods. To date, PNNL has developed a calendering technique that utilizes powders manufactured by Air Products. Test results of thin membranes (<50 microns) show high oxygen flux rates consistent with theoretical predictions, significantly higher than membranes made by other fabrication methods.

Modeling and Computing**44. Title: Application of High Performance Computing of Automotive Design and Manufacturing****ID:** ANL94-54**PI:** David P. Weber
Reactor Engineering Division**Phone:** 630 252-8175**Partner:** SCAAP Consortium
Dearborn, MI**FY 97 Funding:** \$175K**Total Project Funds:** \$1,425K

ANL, four other DOE national laboratories (LANL, LLNL, ORNL, SANL) and the "Big Three" automakers (GM, Ford, and Chrysler) are collaborating in the development of high-performance computer systems that will "leapfrog" the technology automakers now use. This collaboration — the Supercomputer Automotive Application Partnership (SCAAP) — is the 12th major R&D consortium formed under the auspices of the U.S. Council for Automotive Research (USCAR). ANL researchers are participating in the development of comprehensive numerical models for combustion, sprays, and aerodynamics in the design process. Specific partnership tasks include modeling in-cylinder fluid dynamics and conducting analyses of vehicle aerodynamics; under-hood cooling; and heating, ventilation, and air-conditioning (HVAC) systems. ANL has focused on model development for HVAC analyses. Specific models for important thermal, thermal-hydraulic and porous flow phenomena have been developed at ANL and implemented in the partnership code known as CHAD (Computational Hydrodynamics for Advanced Design), which was originally developed at Los Alamos National Laboratory. Testing and application are now being performed in collaboration with industry partners. In other areas partnership engineers are also developing efficient computational models of lightweight fiberglass composites, which automakers can use to design and manufacture lighter, crashworthy vehicles economically. Specific partnership tasks include developing new material and element models for composites, verifying and validating the models, and performing crash simulations on advanced computing architectures. The computational mechanics researchers at ANL have developed advanced element technology for use in large numerical (finite element) simulation of an all composite front structure during a crash process. The results of this project are expected to benefit DOE in both its advanced automotive technologies program and its high performance computing program. The Computational Fluid Dynamics and composite materials activities are directed toward advanced automotive designs that reduce energy consumption, which are being investigated by DOE as part of the Partnership for Next Generation Vehicle (PNGV) program. The computational technologies being developed for three dimensional flow and mechanics

analysis will lead to better understanding of how to use leading-edge massively parallel computers for engineering applications.

45. Title: Advanced Nuclear Power Plant Simulation

ID: ANL95-03

PI: David P. Weber
Reacton Engineering Division

Phone : 630 252-8175

Partner: Fauske and Associates, Inc.
Burr Ridge, IL

FY 97 Funding: \$200K

Total Project Funds: \$600K

The objective of this project is to develop an advanced nuclear power plant simulation capability. Nuclear power plants are complex engineering systems that are designed to operate safely, and the plant operators undergo extensive training for normal and off-normal conditions. The industry makes extensive use of computer-based simulators in training. The purpose of this project is to develop an advanced power plant simulation computer code that will aid and train the plant operator in off-normal and accident conditions. The project is taking the present serial version, 4.0, of the Electric Power Research Institute (EPRI) code Modular Accident Analysis Program (MAAP) and creating a new parallel software package by parallelizing the "driver" software and as many of the subroutine modules as possible. Further, additional phenomenological and system simulation capability are being added to the software. The software will be marketed by Fauske to the utility industry. The ANL project objectives of implementation of space-time reactor kinetics capability and the development of a parallel computing capability have been successfully completed. The project is directed toward developing tools that can further assure safe operation of nuclear power plants in the U.S. and around the world. This assurance of safe operation is part of the mission of DOE's nuclear energy program.

46. Title: A Neural Network Model for the Sheet Metal Forming Die Design Process

ID: ORL95-90

PI: Gerald M. Ludtka
Metals and Ceramics Division

Phone: 423 574-5098

Partner: Lear, Inc.
Morristown, TN

FY 97 Funding: \$248K

Total Project Funds: \$905K

The principal focus of this project is a significant reduction in the very high costs presently incurred by virtue of the highly iterative nature of the metal die design process. The objective is the development of a method or methods for generating the required die in one (or at most a few) iteration(s) in a manner sensitive both to material properties, and to material forming conditions and parameters (press, die, tooling, lubrication, etc.). Tasks critical to ultimate success are identification of a predictive technique capable of dealing with data representing surface conformation, and development of data compression methods by which to represent three-dimensional component data in a readily computable manner. Of the techniques potentially suited to die conformation prediction (statistical methods, fuzzy logic, neural networks, among the better known), only trainable neural networks satisfy the important condition on a priori "ignorance" of functional form. A neural network system incorporating a novel "Patch" compression method has been developed and demonstrated to be capable of the required performance. A significant advantage of the Patch method derives from its abilities both to capture with high fidelity the general relationships inherent in material forming operations and to apply these relationships effectively in the prediction of die designs for unfamiliar configurations. No other explored mechanism (2-D Fourier Transform, 2-D Wavelet Transform, etc.) has exhibited such a capability. To date the new method has resulted in the filing of one patent and the presentation of an invited conference paper. The developed system will be refined during FY 1998 and applied to the development, in collaboration with the industry partner, of a component of commercial utility. If, as expected, the combination of novel data compression and neural network codification of material forming relationships leads to the reduction of even one iteration of the die design process, the potential savings can amount to billions of dollars. The Patch compression/neural network method need not be limited in its application to the die design process. Any industrial process for which the ability to capture relationships in three-dimensional data and to make subsequent predictions based thereupon can benefit from the newly developed method. This project supports the DOE Technologies Enabling Agile Manufacturing (TEAM) program through the development of a real-time tool that can capture the predictive capability of the most sophisticated, validated computational tools for process control and tooling design.

47. Title: Modeling and Simulation of Advanced Sheet Metal Forming

ID: PNL94-38

PI: Mark Smith
Materials and Chemical Sciences Division

Phone: 509 376-2847

Partner: General Motors Research and Development Center
Warren, MI

FY 97 Funding: \$170K **Total Project Funds:** \$1,370K

This project will enhance numerical modeling and simulation of advanced sheet metal forming processes, allowing rapid processing of lightweight aluminum alloy sheet. In this project, improved material deformation models and predictive codes for advanced forming processes will be developed. Benefits of this research include development of analytical tools that allow the modeling and simulation of complex sheet forming processes, and the ability to optimize these processes without the usual trial-and-error approach. Development of the new capabilities will allow the vehicle manufacturing industry to optimize its component and tooling designs and improve its forming processes, leading to greater use of lightweight materials in its products and reduced energy consumption. The development of numerical modeling capabilities for advanced sheet forming requires integrated materials modeling, finite element code development and experimental verification in order to provide an accurate and efficient computational platform. The project team, which includes substantial contributions from MARC Analysis Research Corporation (finite element code) and two universities, has focused on developing a mechanistic and metallurgical based understanding of the deformation process which can be implemented in the commercial MARC finite element code. This finite element modeling platform has been designed to provide the Industry Partner with an efficient, work station-based analysis capability that allows complex sheet parts to be analyzed and optimized. Accomplishments to date for the project include the development of a new, more accurate material test method that provides significantly improved materials modeling data, the implementation of the material models into an enhanced and highly efficient MARC analysis code, and experimental forming and verification experiments that have allowed the forming process to be optimized. The application of the technology will directly benefit the future development of lightweight, fuel efficient vehicles by providing a cost effective forming technology for aluminum sheet materials. Remaining activities will focus on the implementation of a microstructurally-based cavitation/damage model that will minimize the development of cavitation in the formed part by controlling forming rates at larger superplastic strains. Research will also demonstrate the use of energy control methodologies for management of the gas pressure system during superplastic forming. Both modeling and control enhancements will be verified using tray and bulge forming experiments. This project supports the DOE mission for advanced processing methods to support more fuel efficient vehicles.

SUSTAINABLE ENVIRONMENTS

Energy Conversion and Storage**48. Title: Development of Lumeloid, A Solar Energy Conversion Material****ID:** ANL 94-42**P.I.** Michael R. Wasielewski
Chemistry Division**Phone:** 630 252-3538**Partner:** Advanced Research Development, Inc. (ARDI)
Athol, MA**FY 97 Funding:** \$200K**Total Project Funds:** \$900K

The objective of this project is to develop photoactive polymer composite materials to directly convert solar energy into electricity. This technology utilizes ANL expertise in developing photoactive materials, in combination with Advanced Research Development, Inc. (ARDI) film technology to generate new material composites that could have a significant impact on cheap and efficient power generation from solar energy. The more than 25% theoretical efficiency of Lumeloid compares advantageously with the silicon solar cell which is near 20%. Using Lumeloid as a light-to-power device could be at least as efficient, but considerably cheaper to produce than silicon solar cells, thus allowing efficient, economical operation of solar power plants in regions of the U.S. not previously considered. A new class of perilune dye-based donor-acceptor molecules has been developed to function as photodopants for conductive polymers based on polyacetylene and poly(phenylenevinylene). These dopant molecules are among the most efficient molecules known for photon to stored charge conversion. These molecules strongly absorb visible light and are reasonably soluble in organic solvents to aid processing. The chemical compatibility of the dopants has been tested with polymer films developed both at ARDI and ANL. Photovoltaic characteristics of these films have been measured. Film processing at ARDI has focused on using stretch orientation of the dopant-conductive polymer composites with simultaneous stretching and electrical poling of the films to produce the ordered arrays of molecules necessary for the resulting film to function properly. This process and the associated engineering tests and controls are similar to those used in the commercial manufacture of polarized film. Conductor bus connections on a 100 micrometer scale have been developed in a process similar to that employed in the semiconductor or photolithography industry to access the charge in the films. Construction of solar cells based on these component technologies

is proceeding. This project supports the DOE mission in energy efficiency and renewable energy sources.

49. Title: Synthesis and Crystal Chemistry of Technologically Important Ceramic Membranes

ID: ANL97-06

PI: James D. Jorgensen
Materials Science Division

Phone: 630 252-5513

Partner: Amoco Corporation
Naperville, IL

FY 97 Funding: \$125K

Total Project Funds: \$510K

Achieving the conversion of natural gas to synthesis gas (syngas) using oxygen-permeable ceramic membranes would bring vast resources of natural gas within our economic reach. This new technology depends on the development of suitable ceramic membrane materials whose performance is then demonstrated in prototype reactors. This project includes the development of suitable membrane materials at ANL, and the construction of a prototype reactor to evaluate the materials performance and demonstrate the viability of the process at Amoco. A suitable ceramic membrane material, that demonstrates the potential for the desired performance, has been developed in previous work. However, the exact chemical composition and crystal structure of this material is not known. Neutron and x-ray diffraction techniques will be used to determine this information. This will allow the synthesis and processing of the membrane material to be optimized to produce the best performance. In-situ neutron diffraction at elevated temperature in conditions that simulate the environment in a working syngas reactor will be used to study aspects of the materials related to achieving the longest possible working lifetime. Existing laboratory and pilot plant facilities will be upgraded and modified to facilitate testing of the ceramic membranes under increasingly rigorous conditions. This will provide a valid test of the suitability of the ceramic materials for use in large-scale reactors that convert natural gas into syngas and, at the same time, a useful test of the overall process.

50. Title: Low Temperature Liquid Phase Catalytic Synthesis of Methanol from Synthesis Gas

ID: BNL95-09

PI: Devinder Mahajan
Department of Applied Science

Phone: 516 344-4985

Partner: Amoco Corporation
Naperville, IL

FY 97 Funding: \$210K

Total Project Funds: \$726K

Natural gas conversion into liquid fuels is a viable option to transport remote gas. Methanol is manufactured by conversion of synthesis gas, a mixture of mainly CO and H₂, in the following reaction: CO + 2H₂ → CH₃OH_(l), (ΔH°_R = -128.6 kJ. mol⁻¹). This exothermic reaction is accelerated by utilizing a supported Cu/ZnO heterogeneous catalyst at T ~ 250°C that limits the equilibrium conversion to less than 20%. A novel homogeneous nickel catalyst, designed and developed at BNL, operates in a liquid phase and allows unprecedented high conversion (>90%) per pass at high reaction rates (without optimization, up to 9 g-mol MeOH/g-mol Cat. h) under thermodynamically allowed low temperature (<150°C) and low pressure (< 5 Mpa). These operating conditions, unique to this catalyst system, eliminate the need for gas recycle, attain excellent heat management, and allow air partial oxidation of natural gas (catalyst being inert to N₂) eliminating the cost of an air separation plant during production of synthesis gas feed. This project is aimed at developing a modified catalyst that incorporates these features and addresses the following tasks: (1) develop a non-Ni(CO)₄ catalyst precursor for safety considerations, (2) establish catalyst tolerance to CO₂, (3) establish catalyst tolerance to H₂O, and (4) transfer and install the Amoco mini pilot unit at BNL, for evaluation of the catalyst in continuous mode. In FY 1997, the following tasks were completed: (1) A series of new Ni complexes were synthesized and characterized using infrared and ultraviolet/visible spectroscopy. After evaluation in batch mode, a catalyst of the general formula Ni(N-N)(CO)₂, where N-N refers to N-containing bidentate ligands that satisfies preset criteria, has been selected for further evaluation in the continuous unit. (2) The ongoing batch evaluation was completed, and a cocatalyst was selected that minimizes the CO₂/base interaction, a side reaction that competes with the methanol forming step in the catalytic cycle. (3) The catalyst/ H₂O interaction was studied from the water-gas-shift-reaction perspective, and a cocatalyst was selected that is compatible with methanol synthesis conditions. (4) The unit transfer from Amoco to BNL was completed, and the unit was ready for shake-down. The remaining tasks to be undertaken in FY 1998 are: (1) selection of an optimum Modified Catalyst formulation, and (2) evaluation in the Amoco Continuous Unit that is now set up at BNL. A successful incorporation of this catalyst in the Methanol Synthesis process could result in up to 40% cost reduction. This is extremely attractive to U.S. companies with large reserves of remote natural gas for transportation by conversion into methanol liquid fuel that competes with well-established liquefied natural gas and Fischer-Tropsch liquids routes.

51. Title: Thin Film Lithium Batteries**ID:** BNL95-11**PI:** James McBreen
Department of Applied Science**Phone:** 516 344-4513**Partner:** Power Conversion, Inc.
Elmwood Park, NJ**FY 97 Funding:** \$80K**Total Project Funding:** \$531K

This project is focused on low cost cathode materials for thin film lithium ion batteries. Present commercial batteries use LiCoO_2 which gives the most consistent performance and life. However, it is expensive. Lower cost materials would broaden applications beyond the present high end applications such as laptop computers and camcorders. Possible lower cost substitutes are LiMn_2O_4 or LiNiO_2 . Reproducibility and cycle life of these materials is a problem. BNL has evaluated several types of LiMn_2O_4 and LiNiO_2 as well as mixed transition metal lithium oxides. Extensive in-situ X-ray absorption (XAS) studies were done on these materials at the National Synchrotron Light Source (NSLS). This was supplemented by both in-situ and ex-situ X-ray diffraction (XRD) studies. The results of these studies were correlated with material stability on cycling. In FY 1998, the best of the cathode materials will be combined with electrolytes developed in FY 1996 and will be tested in small lithium cells. The cells will be examined at the NSLS using both in-situ XAS and XRD. This project supports DOE's mission in energy efficiency and alternative energy sources.

52. Title: New Catalysts for Direct Methanol Oxidation Fuel Cells**ID:** BNL95-14**PI:** Radoslav Adzic
Department of Applied Science**Phone:** 203 727-2278**Partner:** International Fuel Cells Corporation
Windsor, CT

FY 97 Funding: \$80K**Total Project Funds: \$510K**

The goal of this project is the synthesis and characterization of new efficient electrocatalysts for direct methanol oxidation fuel cells and development of a basic procedure for catalyst fabrication. This will assist International Fuel Cells Corporation in their efforts to develop an efficient methanol oxidation fuel cell for electric vehicles. The new electrocatalysts are expected to provide the oxygen-containing species to oxidize the strongly bound intermediates (poisons) in methanol oxidation, without affecting the intrinsic activity of Pt for methanol adsorption. The latter appears to be one of the problems with the best existing Pt-Ru alloy catalysts whose activity is still insufficient. Oxide and mixed oxides, RuO_2 , CoWO_4 , NiWO_4 , and chalcogenide $\text{Mo}_4\text{Ru}_6\text{Se}_8\text{O}_2$, were synthesized and characterized using X-ray diffraction, and used as a support for the Pt electrocatalyst. Also, RuO_2 doped with tungstates was used. Upon activation with Pt, the activity for methanol oxidation of electrodes prepared from these electrocatalysts was measured by electrochemical techniques. Two samples were characterized by in-situ X-ray absorption techniques with synchrotron radiation at the National Synchrotron Light Source. The activity was investigated as a function of several parameters that affect the properties of these systems. Some samples showed activity similar to that of the best available electrocatalysts based on Pt-Ru alloys. Further work will involve some modification and optimization of the best catalysts and their long-term testing. Further characterization of these systems by in-situ infrared spectroscopy and synchrotron radiation techniques will provide data on the factors governing the activity of electrocatalysts for methanol oxidation and help their optimization. The best catalysts will be delivered to International Fuel Cells Corporation for evaluation in large-area electrodes. These investigations will determine future efforts with these metal-metal oxides electrocatalysts. Fuel cell research is a key component of the DOE mission in exploring alternative energy resources.

53. Title: High-Rate Zinc/Air Batteries for Consumer Applications**ID:** LBL94-43**PI:** Elton Cairns
Environmental and Energy Technologies Division**Phone:** 510 486-5028**Partner:** Rayovac
Madison, WI**FY 97 Funding: \$62K****Total Project Funds: \$285K**

The Zn/air battery is an especially appealing technology for use in consumer batteries because of its

high specific energy, low cost, and environmentally benign components. The zinc-air technology is greatly under-utilized because of the generally low power available from the cell. The power limitations stem primarily from the air electrode as a result of the slow kinetics of the electrochemical reduction of oxygen from air. Complete utilization of the zinc loading can also be a problem at high power drains. The focus of this project has been to address these two limitations in order to extend the possible markets for the zinc/air primary battery technology. The first part of this project has been concerned with the application of novel electrocatalysts to the air electrode structure to improve the high-power performance of this electrode. The second part will focus on the study and modification of the zinc electrode formulation in order to optimize zinc utilization at high power. Five electrocatalyst systems are under study at LBNL. The electrocatalysts are added to the state-of-the-art Rayovac air electrode, and performance is being evaluated in the three-electrode configuration in the absence of zinc. Methods to apply electrocatalysts to carbon electrodes have been tested and improved. A wet-proofing method for catalyzed electrodes has been developed: airbrushing to add a porous polytetrafluoroethylene layer. Five electrocatalysts have been tested, and three have demonstrated higher current densities for oxygen reduction than the uncatalyzed control electrodes. Palladium-catalyzed carbons have been found to be especially promising. This project contributes to DOE's mission in alternative energy sources.

54. Title: Development of Zinc/Nickel Oxide Batteries for Electric Vehicle Applications**ID:** LBL95-27**PI:** Elton Cairns and Frank McLarnon
Energy Conversion and Storage Division**Phone:** 510 486-5028**Partner:** Energy Research Corporation
Danbury, CT**FY 97 Funding:** \$42K**Total Project Funds:** \$260K

In this project, Energy Research Corporation (ERC) and LBNL are developing a zinc/nickel oxide battery for electric vehicles, which is light-weight, rechargeable, sealed, and maintenance-free. This battery uses an alkaline electrolyte, a zinc negative electrode, and a nickel oxide positive electrode. It has two major advantages over competing types such as cadmium/nickel oxide (nickel-cadmium) and metal-hydride/nickel oxide (nickel-metal hydride): it delivers more energy per unit battery mass and costs less to produce. LBNL has developed a novel electrolyte for the zinc/nickel oxide battery that extends its useful life to several hundred charge-discharge cycles. ERC has scaled up the LBNL

laboratory model by a factor of ten and has demonstrated a similarly long lifetime. Together, ERC and LBNL are working on additional improvements to lower the battery mass and to increase the ability of the electrolyte to wet the electrodes. To achieve these goals, the project is incorporating lighter wicking and current-collector materials in the electrode structures. If these efforts are successful, ERC will produce full-size electric vehicle batteries for testing. A superior zinc/nickel oxide battery could be the key to inexpensive and durable electric vehicles which will reduce air pollution and petroleum imports while creating a new growth industry. Two new problems were identified that were compromising cell performance: electrode dehydration (particularly the nickel oxide electrode) and excessive weight of the nickel oxide electrode. Modifications to the battery were developed to correct these problems. Cell designs have been simplified and made less costly. This project supports DOE's mission in alternative energy sources and conservation.

55. Title: Development of a Thin-Film Battery Powered Hazard Card and Other Micro-electronics Devices

ID: ORL94-39

PI: John B. Bates
Solid State Division

Phone: 423 574-6280

Partner: Research International
Woodinville, WA

FY 97 Funding: \$74K

Total Project Funds: \$470K

The goal of this project is to explore the development of micro-electronic devices that can take advantage of the unique properties of ORNL's thin-film rechargeable lithium batteries. The first product is a personal hazardous gas monitor, the "Hazard Card", which is designed to detect the presence of aromatic hydrocarbons. This credit-card sized device indicates four hazard levels with an LED display and sounds an alarm when the highest level is detected. The Li-V₂O₅ battery designed and fabricated for this application had an active area of 9 cm² and exceeded Research International's requirements. Moreover, the battery occupied less than one-half of the area allocated for it on the reverse side of the device substrate. Other value-added applications of thin-film rechargeable lithium batteries include backup of Personal Computer Memory Cards (PCMCs). While the areas of these devices are about the same as that of a credit card, they are much thicker because of the size of the (non-rechargeable) coin cell batteries presently used to maintain static random access memory. A single Li-V₂O₅ battery with the same footprint as the Hazard Card battery maintained the memory of

a 1 MB PCMC active for over 50 days before recharging was necessary. If the area available inside the card package were used for a battery having two cells fabricated on the opposite sides of a thin polymer substrate, memory could be retained in 1 MB and 2 MB PCMCs for over 400 and 200 days, respectively, before recharge. With these batteries, the PCMCs could be made as thin as a credit card. A new oxynitride lithium-ion anode material recently discovered at ORNL increases the service temperature of thin-film batteries to over 250 °C. This will allow these batteries to be incorporated into micro-electronics devices using reflow techniques. The work performed in this project supports DOE's Basic Energy Sciences programs in advanced battery technology and advanced ceramics.

56. Title: Development of a Thin Film Battery Powered Transdermal Medical Device**ID:** ORL95-11**PI:** John B. Bates
Solid State Division**Phone:** 423 574-6280**Partner:** Teledyne Electronic Technologies
Los Angeles, CA**FY 97 Funding:** \$187K**Total Project Funds:** \$650K

Heart and brain activity are monitored by measuring microvolt signals developed on the surface of the skin using transdermal electrodes. The first objective of this project was to develop a thin-film battery powered preamplifier that would attach directly to these electrodes so that the small electrocardiogram (EKG) and electroencephalogram (ECG) signals could be amplified before transmission to the recording unit. These "active" electrodes will eliminate the effect of interference from ac pickup in the long cables from the recording unit and improve the reliability in diagnosing heart or brain malfunctions. By incorporating batteries into the circuit to power the amplifiers, no change to existing EKG or ECG recording equipment is required. A thin-film lithium battery was developed that exceeds the requirements of Teledyne's transdermal-electrode application. The battery, which is based on a LiCoO_2 cathode, was fabricated directly onto the backside of the multi-chip modules developed by Teledyne as a prototype electrode preamplifier. This was the first demonstration of integration of thin-film batteries into electronic devices. When developed, the active electrodes will significantly improve the reliability of EKG and ECG diagnostic measurements and thereby help to improve the quality of patient care at a lower cost. The second objective of this project is to demonstrate manufacturing of thin-film batteries in a pilot scale facility at Teledyne. The cathode and electrolyte films deposited at Teledyne are being shipped to ORNL for a comparison of their properties with those grown at ORNL. To date, Teledyne has fabricated excellent LiCoO_2

cathode films over areas nearly 40 times larger than possible at ORNL. Batteries fabricated at Teledyne will be evaluated at ORNL. If they meet the rigid requirements of the medical device, full-scale manufacturing will follow. Teledyne has licensed ORNL's thin-film battery technology for application in medical devices. The work performed in this project supports DOE's Basic Energy Sciences programs in advanced battery technology and advanced ceramics.

Energy Technologies

57. Title: Evaluation of Fluid Catalytic Cracking Feed Nozzles

ID: ANL95-09

PI: Rajesh Ahluwalia

Phone: 630 655-0238

Partners: Amoco Oil Company
Naperville, IL

Chevron Research and Technology Company
Richmond, CA

Phillips Petroleum Company
Bartlesville, OK

UOP Research Center
Des Plaines, IL

FY 97 Funding: \$150K

Total Project Funds: \$750K

Although the importance of feed nozzle atomization to the yield of Fluid Catalytic Cracking (FCC) units is widely recognized, quantitative data on nozzle performance is not available. This project between ANL, a consortium of oil refiners, and an FCC nozzle vendor will fill the gap in knowledge by characterizing the performance of commercial feed nozzles using state-of-the-art laser-optics instrumentation. The performance data will allow the participants to select commercial nozzles that are optimal for their feeds and operating conditions. In a complementary task, a hydrodynamics model is being developed to determine the impact of atomization parameters on the overall yield of FCC units. The participants will use the information generated in this project to design and fabricate advanced feed nozzles which will also be tested at ANL. To date, a unique, closed water loop has been assembled and commissioned for testing air-assisted commercial feed nozzles of 300 gpm or larger capacity. A Phase Doppler Particle Analyzer was upgraded to a fiber optic system to separate the 5-W multiline Argon laser and the fiber drive from the transmitter and the receiver modules mounted on the rails of a computer controlled traverse. Extensive data were obtained on two commercial nozzles of different configurations to determine the operating map, droplet size distribution, number density, three-dimensional velocity components, fluctuating velocity components, and spray pattern as a function of radial and axial distances, water-flow rate, air-water

ratio, and nozzle rotation. Work has been initiated to model oil droplet lifetimes and trajectories taking into consideration droplet composition, convective heat transfer, radiative heating from catalyst particles, contact heat transfer due to droplet-catalyst collisions, and heat and mass transport within and around the droplets. Research in these areas supports the DOE mission in energy conservation and environmental quality.

58. Title: In-Line Sensors for Electrolytic Aluminum Cells**ID:** ORL95-04**PI:** Jack P. Young
Chemical and Analytical Sciences Division**Phone:** 423 574-4922**Partner:** Alumax Corporation
Ferndale, WAKaiser Aluminum and Chemical Company
Pleasanton, CAReynolds Metals Company
Muscle Shoals, AL**FY 97 Funding:** \$113K**Total Project Funds:** \$370K

The objective of this project is to develop in-line sensors for commercial aluminum electrolytic cell operation. The sensors to be developed will be of a Raman spectral type. The research goal is to develop technology which will allow measurement of soluble alumina, bath ratio, and bath temperature. These in-line measurements will be inputs to new process control algorithms that can then be developed to improve the efficiency of aluminum electrolysis operations thereby reducing energy consumption. Such energy savings are in line with DOE goals. The improved control algorithm will also lead to a reduction in the anode effect frequency which results in wasted energy and fluorocarbon emission. Reduction of potentially hazardous environmental gases is also a DOE goal. Progress has been made in achieving these goals. Short-term compatibility tests of several candidate sheath materials have been carried out. Longer term tests are being conducted by the industry partners. By a modified Raman spectral approach, the concentration of soluble Al_2O_3 in cryolite mixtures has been measured in molten or solid samples. Raman studies of fluor-aluminum species in cryolite melts are also underway using this approach.

59. Title: Moving Advanced Desiccant Material into Mainstream non-CFC Cooling Products**ID:** ORL95-06**PI:** Phillip D. Fairchild
Energy Division**Phone:** 423 574-2020**Partner:** Engelhard/ICC
Philadelphia, PA**FY 97 Funding:** \$178K**Total Project Funds:** \$640K

This project focuses on advancing desiccant-based systems for cooling buildings. The goal is to develop the enabling technologies that will foster the acceptance of desiccant-based equipment into the mainstream of the U.S. commercial air-conditioning market. Barriers to the broad acceptance of desiccant technology are: (1) a perception of inefficiency from earlier research on desiccant space-conditioning systems; (2) lack of suitable metrics to evaluate desiccant-based system performance against conventional systems; and (3) absence of computerized algorithms that allow conventional incorporation of desiccant modules in heating, ventilation, and air conditioning (HVAC) simulation programs used by evaluation and application engineers. The critical tasks to be accomplished include (1) identifying two target HVAC applications for desiccant-based products; (2) developing computerized desiccant-component performance-evaluation and system-simulation tools for building-specification design engineers and architects; (3) preparing a draft performance-evaluation methodology, product rating, and system comparison standard for the desiccant air-conditioning industry; and (4) completing a cost/performance versus market penetration analysis for desiccant products. Desiccant systems can potentially reduce building energy consumption, decrease emissions of greenhouse gases, reduce the use of halogenated refrigerants, reduce electrical peak demand, improve indoor air quality and comfort control, and create jobs in the manufacturing and service sectors in support of DOE missions. To date, a 2,600 cubic feet/ minute desiccant system has been installed in the laboratory for testing over a range of different ambient conditions. Instrumentation, including humidity sensors, gas and water-flow meters, differential pressure transducers, air-flow meters, and temperature sensors, is being installed to determine the performance of the system, while varying operating parameters such as wheel speed, air flow rate, and desiccant material. This installation is about 75% complete. The installation of the chilled mirror system for humidity measurement and the data acquisition system will be completed in September 1997. Testing will begin in early October.

60. Title: An Air Conditioning System with Improved Efficiency for Hybrid/Electric Vehicles**ID:** ORL95-09**PI:** Donald J. Adams
Engineering Technology Division**Phone:** 423 576-0260**Partners:** Nartron Corporation
Reed City, MIAdvanced Vehicle Systems, Inc.
Chattanooga, TNChattanooga Area Regional Transit Authority
Chattanooga, TNElectric Transit Vehicle Institute
Chattanooga, TN**FY 97 Funding:** \$138K**Total Project Funds:** \$670K

The primary technical goal of this project is to develop an advanced, highly efficient, electric motor and controller for a mobile air-conditioning compressor for use in an electric bus air-conditioner system. The objective is to improve the air-conditioner system efficiency so that the useful bus range is extended. To achieve this goal, the efficiency of the Nartron Corporation's technically-advanced, high-efficiency, microprocessor-controlled, turbine-driven, modular air conditioner will be further improved, and mass and size of the compressor motor will be reduced by incorporating advanced, electric-machinery-systems technology that is only available at ORNL. The advanced system will then be installed in an Advanced Vehicle System's (AVS) electric bus that will be operated by the Chattanooga Area Regional Transportation Authority (CARTA) with Electric Transit Vehicle Institute liaison. In parallel, AVS and Nartron may install an air-conditioning system with existing motor technology in another bus for comparison. The electrical and mechanical design of an axial-gap, permanent-magnet motor with challenging size and power constraints has been completed. The first prototype is nearly complete, and comparative lab testing is beginning in late FY 1997. A self-sensing controller for the permanent-magnet machine has been developed that will operate in conjunction with ORNL's Resonant Snubber Inverter. Although Nartron had considerable difficulty in procuring a suitable alternate-technology radial-gap motor, they did succeed in delivering such a system to AVS. All simulations have predicted much improved efficiencies for both the motor and

controller, and the reliability should be much improved. In addition to the development of potentially new technology that could provide significant energy savings for electric-vehicle air conditioners, this work also enhances DOE's Uranium Enrichment programs in power electronics and electric machinery.

Environmental Technologies**61. Title: Bioremediation of Contaminated Soils by Enhanced Plant Accumulation****ID:** ANL95-06**PI:** Ray R. Hinchman and M. Cristina Negri
Energy Systems Division**Phone:** 630 252-3391**Partner:** Applied Natural Sciences, Inc.
Fairfield, Ohio**FY 97 Funding:** \$200K**Total Project Funds:** \$700K

The remediation of contaminated soils and wastewaters at DOE and other sites is a major environmental activity that consumes billions of dollars a year. The emerging technology of phytoremediation uses green plants and their associated rhizospheres in low cost, low technology, and environmentally friendly remediation processes that absorb, accumulate, sequester, metabolize, or otherwise render harmless multiple environmental contaminants (radionuclides, organics, and heavy metals) in soils, groundwater, and wastewater. This project includes greenhouse experiments on the uptake, bioaccumulation, and fate of several heavy metals, as well as the degradation, transformation, and transpiration of chlorinated organic compounds in several woody and herbaceous species. Several of these experiments were conducted using field samples and data from operating plant-based cleanup systems installed by Applied Natural Sciences, Inc. In one greenhouse experiment, zinc concentrations of 800 ppm in the soil water were totally sequestered by hybrid poplar trees in about four hours during a single pass through the root system. The data also showed very high concentrations of sequestered metal ($>38,000 \mu\text{g/g}$) in the root tissue. Because plant roots sequester most of the contaminant taken up, a major objective of this project is to determine the feasibility of root harvesting to maximize the removal of contaminants from soils. Other ongoing studies are evaluating the mobilization, uptake by plants, and fate of several heavy metals in soil using chelating agents, the development of rapid and simple analytical protocols for tracking the uptake and fate of chlorinated organics in plants, the phytoremediation of lead and arsenic, the development and maintenance of a computerized phytoremediation database, and the successful field demonstration of an ANL-developed plant bioreactor for processing the salty wastewater (produced water) from natural gas wells. This project has resulted in new, cost-effective plant-based cleanup systems for contaminated soil, groundwater, and wastewater for use at DOE and other sites.

62. Title: Application of Oxygen-Enrichment Technology for Locomotive Diesel Engines**ID:** ANL 95-10

PI: Raj Sekar
Energy Systems Division

Phone: 630 252-5101

Partners: Association of American Railroads
Washington, D.C.

Electro-Motive Division of General Motors
Dearborn, MI

FY 97 Funding: \$200K

Total Project Funds: \$900K

The Environmental Protection Agency (EPA) has proposed regulatory requirements for the control of emissions from locomotives and engines used in locomotives, as required by Federal Clean Air Act Amendments of 1990. On the basis of the date (from 1973 through 2005 and later) when a locomotive is first manufactured, three separate sets of standards are proposed. As a result, both railroad operators and engine manufacturers face a major challenge to meet the EPA's standards for emissions of smoke, particulates, unburned hydrocarbons, and oxides of nitrogen (NO_x). Railroads also face an increasing demand for higher motive power because of recent developments in a/c traction, so payloads in freight locomotives (revenue) can be increased. The concept of oxygen-enrichment aims at limited substitution of the nitrogen in air by oxygen to achieve low emission levels. Because of the increased oxygen content, additional fuel is burned resulting in increased power output. The reduction in ignition delay period with oxygen-enrichment provides an opportunity to control NO_x emissions by retarding the injection timing. A novel method is being pursued of reducing NO_x emissions by using monatomic nitrogen induced by a pulsed plasma arc. Air delivered to the engines can be oxygen-enriched by selective permeation through non-porous, polymer membranes. A synergistic use of an air separation membrane that provides oxygen-rich air for intake and nitrogen-rich air generating monatomic nitrogen for the NO_x control device may lead to improved combustion and cleaner locomotive diesel engines. This will enable the railroads and engine manufacturers to meet the goals of lower exhaust emissions and higher motive power from locomotives in a cost-effective manner. The project has developed appropriate bench test facilities to characterize the air separation membrane for engine applications. With prototype air separation membranes, various membrane intrinsic properties (permeability, selectivity), coating thickness, hollow-fiber size, and mode of operation (vacuum/mixed) were investigated. Membrane requirements for low parasitic power consumption and compact module size were established for locomotive diesel engine application. For a given constraint on engine structure, the additional power benefits of an engine operating under different levels of oxygen-enrichment were evaluated through an appropriately modified thermodynamic engine performance simulation code. Laboratory engine

tests are presently being conducted on a two-cylinder locomotive research engine to obtain the performance and emissions data at different levels of oxygen-enriched air by using bottled oxygen. An air separation membrane supplying oxygen-enriched air in the engine intake and with solutions to control NO_x emissions will be demonstrated on a research engine (two-cylinder EMD 567B) in the laboratory. This project supports the DOE mission in development of technologies for improved environmental quality.

63. Title: Condensing Economizers for Improved Efficiency and Reduced Pollution**ID:** BNL94-22**PI:** Thomas Butcher
Department of Applied Science**Phone:** 516 344-7916**Partner:** Babcock and Wilcox
Barberton, OHConsolidated Edison Company
New York, NY**FY 97 Funding:** \$136K**Total Project Funds:** \$825K

Condensing economizers recover sensible and latent heat from boiler flue gas, leading to marked improvements in thermal efficiency. These economizers also have the potential to serve as pollution control devices, capturing SO₂, particulates, and air toxics. Configured for pollution control, these systems have been named Integrated Flue Gas Treatment Systems (IFGT). The main objective of this project is to develop IFGT technology so that it will be strongly attractive commercially, providing a viable method of both improving energy efficiency and reducing pollutant emissions from boilers. Expected new products resulting from this work include a variety of IFGT systems. These will reduce operating costs of power generation and process plants through increased energy efficiency. In addition, these will offer attractive options for sites that need to reduce particulates, air toxics, and SO_x emissions. The target market includes existing gas, oil, coal, wood, and waste-fired plants ranging in size from light industrial to utility. At BNL, small-scale pilot research is being done with an emphasis on mechanisms of particulate capture. Babcock and Wilcox (B&W) is doing larger scale, application specific pilot tests with an emphasis on SO₂ and air toxics capture. B&W is also actively marketing these systems both as simple economizers and as integrated pollution control systems. At Con Edison, a full scale, 30 MW unit has been constructed and a comprehensive

demonstration/test program is in progress. This project supports DOE's mission in environmental quality.

64. Title: A Pilot Scale Demonstration of Citric Acid Technology

ID: BNL95-13

PI: A.J. Francis
Department of Applied Science

Phone: 516 344-4534

Partner: Forrester Environmental Services, Inc.
Hampton, NH

FY 97 Funding: \$140K

Total Project Funds: \$438K

The overall objective of this project is to remove toxic metals such as Pb and Cd from incinerator ash using the BNL Citric Acid Process. In this process toxic metals in bottom ash from the incineration of municipal solid waste were first extracted with citric acid followed by biodegradation of the citric acid-metal extract for metals recovery. The ash contained the following metals: Al, Ba, Ca, Cd, Cr, Cu, Mg, Mn, Ni, Pb and Zn. The extraction efficiency of the metals by citric acid was affected by the mineralogical association of the metals in the ash. Biodegradation of the citric acid extract resulted in the precipitation of metals along with the biomass. The project also investigated (1) the impact of mixing aggressiveness during citric acid extraction, (2) the use of low molarity citrate, (3) citric acid recovery and recycle, and (4) speciation of Pb and other metals associated with the biomass after biodegradation of citric acid extract by X-ray absorption spectroscopy techniques at the National Synchrotron Light Source. Optimization of the citric acid process removed greater than 97% of Pb and Cd from incinerator ash allowing to pass Toxicity Characteristic Leachability Procedure. The chemical treatment process resulted in the complete (>99%) recovery of spent citric acid. The extraction efficiency of recycled (recovered) citric acid is similar to the fresh citric acid. Speciation of Pb associated with the biomass by extended X-ray absorption spectroscopy show that precipitated lead is predominantly associated with the phosphate and carboxyl groups. These results suggest the potential application of this technology to remove and recover the metal contaminants from incinerator ash and from other waste forms. Information developed from this project is being applied by Forrester Environmental Service, Inc. to remediate Pb paint bearing soils and the removal of Pb and other toxic metals from electric arc furnace dust waste from steel processing. This citric acid project supports DOE's mission in environmental quality and pollution prevention.

65. Title: Development of Low NO_x Natural Gas Furnaces and Boilers**ID:** LBL94-45**PI:** Robert Cheng
Environmental Energy Technologies Division**Phone:** 510 486-5438**Partner:** Teledyne Laars
Moorpark, CA**FY 97 Funding:** \$48K**Total Project Funds:** \$435K

The goal of this project is to develop LBNL's lean pre-mixed weak swirl burner (WSB) for use in low-emission commercial water heaters, supporting DOE's mission in energy conservation for environmental quality. A lean combination of fuel and air, mixed upstream of the flame and set into weakly swirling motion, burns stably at a low temperature which reduces NO_x emissions. The LBNL burner uses a radically different operating principle from its commercial counterparts and appeals to Teledyne Laars' design engineers because of its simplicity. A patent application on this novel burner has been granted by the U.S. Patent Office. This project has enabled LBNL scientists to offer their fundamental combustion knowledge to adapt the burner for practical use. The Teledyne Laars engineers, in turn, contribute practical skills to develop a new commercial product. To date, a compact burner has been designed to operate under the requirements of a typical Teledyne Laars product. Laboratory tests performed with a 13 kW (50,000 Btu/hr) Teledyne Laars Telstar Spa heater fitted with a WSB have reduced NO_x emissions by more than 20 times (from 90 to 4 ppm) without sacrificing efficiency. The WSB concept has been further refined to sharply reduce manufacturing costs; a specialized vane swirler has been invented and studied in the laboratory at LBNL (patent pending). Teledyne Laars and LBNL have successfully tested the vane swirler in a prototype burner of 104 kW (400,000 Btu/hr). These test results have prompted Teledyne Laars to begin developing WSB-based pool heaters for market introduction in 1998.

66. Title: Catalytic Conversion of Chloro-fluorocarbons Over Palladium-Carbon Catalysts**ID:** LBL95-45**PI:** Gabor A. Somorjai
Materials Sciences Division

Phone: 510 486-4831

Partner: DuPont Company
Wilmington, DE

FY 97 Funding: \$257K **Total Project Funds:** \$714K

Chlorofluorocarbons must be replaced by fluorocarbons as refrigerants and chemicals because of their adverse health effects (ozone depletion and other effects). The hydrodechlorination (HDCI) of $C_2F_4C_{12}$ to $C_2F_4H_2$ using palladium catalyst supported on carbon achieves this goal. This project investigates the kinetics and mechanism of this reaction using palladium foils and single crystals as catalysts. From reaction rate studies, the kinetic parameters for the reaction have been determined along with many of the elementary reaction steps. The structure and bonding of reactants and products on various single-crystal palladium surfaces have also been determined. The causes for catalyst deactivation have been studied along with the roles of the carbon support to influence catalytic reaction rate and selectivity. The desired $C_2F_4H_2$ molecules are formed in parallel with other competing reaction products, and the inhibition of these side products was investigated. The adsorption of the molecular $C_2F_4C_{12}$ reactant is the rate determining step for the process. The reaction is surface structure insensitive; sulfur causes deactivation, and the carbon support plays a minimal role during the catalytic reaction. Early results indicate that hydrogenation of chlorofluorocarbons with solid state hydrogen is much more selective and occurs at a higher rate than hydrogenation using gaseous hydrogen. The project team will explore the palladium-catalyzed HDCI of chlorofluorocarbons to produce fluorinated olefins. These are important reaction intermediates that can be used to produce new fluorocarbons in addition to tetrafluoroethane. The hydrogenation ability of solid state hydrogen that is stored in the bulk palladium will be studied. This project supports DOE's mission in environmental quality and advanced materials processing

67. Title: Advanced Quadrupole Ion Trap Instrumentation for Low Level Vehicle Emissions Measurements

ID: ORL94-62

PI: Michelle V. Buchanan
Chemical and Analytical Sciences Division

Phone: 423 574-4868

Partner: Environmental Research Consortium (ERC)
Dearborn, MI

FY 97 Funding: \$75K **Total Project Funds:** \$685K

Rapid, rugged, and cost-effective instrumentation for emissions monitoring is required both in the development of ultra-low emission vehicles and in meeting regulatory requirements. The primary goal of this project is to develop an advanced quadrupole ion-trap mass spectrometer (QIT) (a device for separating and sorting ionized molecules by their molecular weight) for the real-time measurement of trace components in automobile exhausts. The critical tasks are to develop means for ionizing hydrocarbons and oxygenated hydrocarbons in a selective fashion and to develop tailored analysis protocols using ion-trap tandem mass spectrometry to give highly specific detection. This work should result in a new tool for vehicle-emissions measurements for use in vehicle test facilities. The successful development of these devices will provide the U.S. automobile manufacturers with a competitive advantage over their non-domestic rivals in bringing ultra-low emission vehicles (ULEVs) to market. This will also have obvious benefits to the air quality of our cities. The technology may also find use in air-quality measurements of interest to other government agencies, such as the Environmental Protection Agency. The work on this project was completed this fiscal year with the successful development of glow-discharge NO₂ ionization coupled with an ion trap that employs tailored wave-forms for tandem mass spectrometry. The ability to measure targeted hydrocarbons to low parts-per-billion levels was demonstrated. This work provides the ERC with the option of using this powerful methodology for other measurement needs. A paper for publication in a peer-reviewed journal that describes this work has been drafted and is undergoing clearance review in the respective partners' organizations. This project supports DOE's mission in energy production and use. It also contributes to the minimization of hydrocarbon emissions from anthropogenic sources.

68. Title: Development of Environmentally Conscious Machining Fluids

ID: ORL94-91

PI: Michael Sigman
Chemical and Analytical Sciences Division

Phone: 423 576-2173

Partner: Cincinnati Milacron
Cincinnati, OH

FY 97 Funding: \$204K **Total Project Funds:** \$895K

The objective of this project is to develop a method to degrade synthetic cutting fluids and reduce their total organic carbon (TOC) content to allow for disposal of used fluids in municipal sewage treatment facilities. Photochemical-based advanced oxidation technologies (AOT) have been successfully utilized to remove greater than 95% of the TOC from synthetic cutting fluids possessing excessively high initial TOC levels (ca. 5,000-15,000 parts-per-million [ppm]). Optimal treatment conditions for fluids with an initial TOC level of 15K ppm involve photolysis of an ozone (~2.5% in air) purged fluid following addition of sufficient hydrogen peroxide to give a 3% (volume/volume) peroxide concentration. Energy and chemical costs for these processes have been determined in a laboratory reactor and found to be comparable to projected costs from commercial manufacturers of industrial scale AOT equipment. New and used cutting fluids have been successfully treated by this methodology. High TOC containing aqueous fluids containing cutting fluid constituents have also been studied, and a structure/treatability relationship has been established that will allow for a new fluid formulation optimized for post-use treatment. Current investigations are focused on understanding the fluid component degradation at a molecular level to further enhance the utilization of AOT in treating high TOC aqueous fluids. Research into the reduction of pollutants supports DOE Environmental Management as well as basic chemical science programs and the DOE mission in environmental quality.

69. Title: Hybrid Quantum Mechanical/Molecular Mechanical Methods for the Development of Pesticides, Dyes, and Polymers

ID: PNL94-10

PI: Richard Kendall
Environmental and Molecular Sciences Division

Phone: 509 375-6734

Partner: E.I. du Pont de Nemours & Company
Wilmington, DE

FY 97 Funding: \$150K

Total Project Funds: \$367K

The purpose of this project is to develop Hybrid Quantum Mechanical/Molecular Mechanical (QM/MM) methods to study general solution photochemistry and apply this to the development of new pesticides, dyes, and polymer processing technologies. This project targets the chemical manufacturing industry's need to develop and employ advanced computational chemistry tools. Further development and application of state-of-the-art codes to specific problems will also increase the usefulness of codes in addressing DOE-related problems, thus contributing to the fulfillment of DOE missions in science and technology and environmental quality. Employing its extensive expertise in computational chemistry and chemical structure modeling, PNNL is collaborating with

DuPont to perfect the parameterization of the QM/MM methods and development of leading edge computational algorithms. Hybrid QM/MM methods are theories that use quantum mechanics (i.e. solve the molecular Schroedinger equation) for part of a complex chemical system, such as a dye molecule embedded in a solvent bath. The remainder of the system (e.g. the solvent) is treated with nonquantum mechanical methods that model bonded and nonbonded interactions with simpler potentials. These methods are useful for studying inherently quantum mechanical phenomena in complex environments, without using QM on the entire system. This project will further develop and apply these methods to enable DuPont to develop more environmentally benign products, while also enhancing understanding of important chemical reaction mechanisms. To date, the project has achieved the first use of a theoretical approach to demonstrate the role of the protein in understanding the origins of the Stark effect of the primary electron donor in photosynthetic reaction centers. It has also produced the first demonstration of the existence of quinoid structures in the solvent-induced rehybridization of triphenylmethane cation dyes in polar solvents. The first use of a QM/MM density functional theory to study a large dye molecule in aqueous solution has also been demonstrated. Use of the QM/MM method for molecular dynamics simulations of chromophores in bulk solvent has been demonstrated. Finally, QM/MM methods have been partially incorporated into PNNL's NWChem suite of codes being developed for massively parallel computing platforms. DuPont has used some of this technology to study the photochemistry of polymeric systems and agrochemicals and to develop new dyes (three patents have been applied for.)

70. Title: Vehicle Exhaust Treatment Using Electrical Discharge and Materials Chemistry**ID:** PNL95-10**PI:** Thomas Orlando
Environmental and Molecular Sciences Division**Phone:** 509 376-2847**Partner:** USCAR
Dearborn, MI**FY 97 Funding:** \$210K**Total Project Funds:** \$750K

This project will help determine the feasibility of using non-thermal plasmas in conjunction with catalytic materials to mediate exhaust gas emissions from an internal combustion engine. The project is directed toward developing an understanding of plasma activated processes which can be exploited to reduce NOx emissions under lean burn (highly oxidative) conditions. Work is focused on the plasma and materials issues that will ultimately allow the development of a device for reductive elimination of NOx from lean burn exhaust streams. The device should also simultaneously oxidize

hydrocarbons while reducing NOx. Technologies will be evaluated with a goal of supplementing or replacing existing automobile catalytic converters. This work is also being evaluated with respect to its applicability to compression ignition (diesel) emissions. A systematic study has been of the destruction of low concentrations (<600 ppm) of NO and NOx in synthetic lean-burn exhaust mixtures using a dielectric barrier/packed-bed corona reactor. It has been the NO and NOx removal efficiency using several packing materials and demonstrated that the packing (and the subsequent surface/material chemistry) can alter the overall discharge-driven chemistry and energy requirements. Materials which are inefficient in removing NO via thermally activated processes become "plasma-activated" when used as packing materials in the corona reactor. A two stage reactor which utilizes corona in the first stage and a thermally activated catalyst in the second has been developed.. Using this reactor, up to 65% overall NOx removal at relatively modest input energies has been achieved. The use of a pulsed power source in the reactor improves the efficiency, particularly at higher temperatures. Although many technical issues remain to be addressed, these results provide a strong indication of the potential for non-thermal plasmas (in conjunction with appropriate catalytic materials) to provide a means for the elimination of NOx (and possibly other) emissions from lean burn vehicle exhaust streams. As a result of these promising results, the industry partner has substantially increased its resource commitments to the project. This increased commitment has been matched by an increase in DOE funding from the DOE Office of Transportation Technologies, which is now co-funding the project with the Laboratory Technology Research program. The results of the work have been presented in several technical meetings and publications. This project directly supports DOE missions in environmental quality and energy efficient vehicle design.

71. Title: Solid Acid Environmental Catalysis**ID:** PNL95-27**PI:** John Nicholas
Environmental and Molecular Sciences Division**Phone:** 509 375-6559**Partner:** Catalytica Advanced Technologies
Mountain View, CA**FY 97 Funding:** \$102K**Total Project Funds:** \$291K

The purpose of this project is to develop new catalytic materials and processes that have little or no impact on the environment. Most industrial catalytic processes involve toxic liquid acids and bases, which are dangerous to work with and difficult to dispose of. In collaboration with Catalytica Advanced Technologies (a small business), PNNL is working to understand the atomic-level details of catalysis, with the eventual goal of developing new catalysts that are environmentally benign.

PNNL will develop theoretical predictions about the function of the new catalysts, which will be complemented by experimental evaluation and verification by Catalytica. In this way, the unique computational capabilities of the Environmental and Molecular Sciences Laboratory will be used to elucidate the fundamental aspects of catalysis, which are needed in order to develop new environmentally-benign products. Because many large-scale processes used in the petroleum and chemicals industries rely heavily on catalysis, the development of a new catalytic process could have a significant economic impact. In addition, catalytic processes that reduce energy consumption, or lead to alternative fuel sources, could reduce U.S. dependence on foreign oil supplies. Current work is focused largely on the validation of new theoretical methods that can be applied to catalyst design. The model systems required to obtain an accurate picture of the catalytic system are often large, and calculations on them require extensive computational resources. The NWChem computer code recently developed by PNNL for massively parallel computers is being evaluated for catalyst research. NWChem includes a highly efficient density functional theory (DFT) module. DFT gives geometries and energies that agree well with experimental data. In addition, DFT is able to treat transition metal systems, important to catalysis, that can be problematic with other methods. The mechanisms of two new catalysts developed by Catalytica are also being studied. These catalysts convert methane, which commonly must be burned at remote oil drilling sites, to methanol, a liquid that can be safely transported and processed into an alternative energy source.

72. Title: Highly Dispersed Solid Acid Catalysts on Mesoporous Silica**ID:** PNL97-28**PI:** Yong Wang**Phone:** 509 376-5117**Partner:** UOP Research Center
Des Plaines, IL**FY97 Funding:** \$125K**Total Project Funds:** \$750K

This project will develop new materials optimized for use as solid acid catalysts by coupling the advanced characteristics of mesoporous silica with the superacidic properties of tungstophosphoric acid and sulfated zirconia. The surface of mesoporous silica will be functionalized to accommodate the dispersion of tungstophosphoric acid and sulfated zirconia. This approach should produce a new class of highly active, shape selective, and robust solid superacid materials. The novel catalysts will be tested with the alkylation and isomerization reactions in the bench and pilot scale testing unit. The goal is to exceed the performance characteristics of existing solid superacid catalysts, thereby enabling the chemical and petrochemical industries to replace homogeneous acid catalysts. This will contribute to DOE's mission to reduce environmental impacts in the energy sector. Homogeneous

acid catalysts such as sulfuric acid and aluminum chloride are currently used to catalyze many of industrially important reactions. Although these homogeneous acid catalysts are efficient, they are not environmentally benign and create many operational problems. These problems can be mitigated with solid acid catalysts. Tungstophosphoric acid and sulfated zirconia are two solid acid catalysts with super acidity. Low catalytic efficiency is the common problem with these two catalysts. In addition, it is difficult to disperse tungstophosphoric acid on supports due to its large cluster size and sulfated zirconia generally suffers rapid deactivation. These problems can be minimized with the superior characteristics of mesoporous silica. This work will enhance understanding of how the mesoporous support properties and acid grafting strategy influence reactivity, yields, selectivity, thermal stability, coking, and regeneration of the solid acid catalysts. Research under this project was initiated in August 1997. To date, efforts have been conducted to define the specific catalyst properties of interest. Initial synthesis and functionalization of the mesoporous silica supports has also been initiated.

Biotechnology**73. Title: Development of Rapid Prototyping Technology for Bioceramic Applications****ID:** ANL 95-08**PI:** William A. Ellingson
Energy Technology Division**Phone:** 630 252-5068**Partners:** Zimmer, Inc
Warsaw, IN**FY 97 Funding:** \$276K**Total Project Funds:** \$750K

This project addresses the need to reduce medical costs associated with orthopaedic implant design, fabrication, and implantation, including medical costs for injury recovery time, as many situations require special implant configurations and designs. The approach is to reduce the cost of producing these complex implants using FDA approved bio-ceramic materials through two activities: (1) development of a new fabrication technology called "Rapid Prototyping" (also called Solid Freeform Fabrication) through use of FDA approved bioceramic materials, and (2) development of reverse engineering technology using 3-Dimensional X-ray Computed Tomographic Imaging (often called CAT scans in the press) and necessary advanced digital imaging methods. Tasks in the project include development of: (1) appropriate bioceramic feed stock for the rapid prototyping machine, (2) binder burn out and sintering schedules for the bioceramic materials, (3) machine parameters for proper fabrication of these materials, (4) digital image methods to allow digital files to be extracted from the "CAT" scan images to allow use by the rapid prototyping machine, (5) algorithms to allow digital image files as input to CAD software packages for design modifications, and (6) surgical implant procedures for these new implants. To date, selected bones (chosen by the industrial partners as being of importance), hand and forearm, have been used for high resolution X-ray imaging, and digital images have been obtained. The files have been extracted and modified to allow input to the rapid prototyping machine. (Feedstock materials for the rapid prototyping machine, using aluminum oxide, have been developed including binder burn out and sintering schedules.) The first hand bone and forearm bone have been fabricated using the feedstock material and the rapid prototyping machine. New bioceramic materials including hydroxyapatite/tricalciumphosphate are now under development. Machine parameters, including thickness of layer, filament temperature, and cross-head speed, have been established for using the new feedstock material. The reverse engineering research is currently under study by the industrial partner for application. This project supports the DOE mission in materials research and medical applications.

74. Title: DNA Repair Enzyme-Liposomes: Human Skin Cancer Prevention**ID:** BNL95-03**PI:** Betsy Sutherland
Biology Department**Phone:** 516 344-3380**Partner:** Applied Genetics, Inc.
Freeport, NY**FY 97 Funding:** \$200K**Total Project Funds:** \$756K

Sunlight-induced skin cancer results from the induction of damage in DNA by ultraviolet (UV) light in the solar spectrum, and from unsuccessful repair of that damage. Applied Genetics, Inc. (AGI) has devised and holds the U.S. patent on liposome products which deliver DNA repair enzymes to the skin, thus increasing its ability to repair UV-induced lesions. As part of its program to test and validate the efficacy of these repair enzyme-containing liposomes, AGI wants to test their action at low levels of DNA damage which might be met in normal human exposure to sunlight. In this project, BNL has developed sensitive methods for measuring DNA damage at low levels in non-radioactive DNA, including skin. In addition to carrying out measurements of DNA damages at low levels in human skin and cells, BNL will transfer to AGI the technology for such high sensitivity quantitation, including training of AGI personnel and providing written methodologies. The partnership of AGI and BNL will contribute to the development of methods for reduction of cancer, new scientific knowledge, and the economy of the U.S. This project supports DOE missions in molecular biology and health applications.

75. Title: Neurochemical Imaging of Gene Therapy**ID:** LBL94-09**PI:** William Jagust
Life Sciences Division**Phone:** 510 486-6241**Partner:** Somatix Therapy Corporation
Alameda, CA**FY 97 Funding:** \$382K**Total Project Funds:** \$1,047K

Parkinson's disease is an extremely common neurodegenerative disease which results in loss of mobility, tremor, and eventually death. The disease is caused by a loss of dopamine-producing cells in the substantia nigra. Although some benefit is produced by treatment with L-DOPA, this treatment eventually fails in most patients. This project is designed to develop primate models of Parkinson's disease and apply this model to a novel therapy of Parkinson's disease using genetic engineering, thus supporting DOE's mission in health applications and molecular biology. The primate model used is MPTP-parkinsonism, in which the neurotoxin MPTP is used to destroy dopaminergic neurons, producing animals with parkinsonism. Positron Emission Tomography (PET) scanning using the tracer 6-[18F] fluoro-L-m-tyrosine (FMT) is used to document and stage the severity of the dopaminergic lesion. Following this, animals are treated with genetically engineered fibroblasts which express one of several enzymes involved in dopamine synthesis, or with fibroblasts expressing brain trophic factors which produce neuronal growth and sprouting. PET studies with FMT are used to monitor the effects of these genetically engineered brain grafts to determine efficacy and time course of the therapeutic benefit. In this project, efforts at development of the MPTP model have been highly successful. Lesioned animals have been produced, and PET studies of these lesions clearly demonstrate reductions in tracer uptake in the caudate and putamen (striatum) which are the dopamine-containing structures in the lesioned hemisphere. A separate series of experiments evaluated blood and brain metabolism, supporting the use of the three-compartment model which was applied for quantitation. In a third study, the time course of the lesion was examined and, surprisingly, an upregulation of tracer uptake in the cells of origin of dopamine in the substantia nigra was found. This suggests that the nigra retains functional plasticity, raising the possibility that appropriate treatment could result in recovery of the dopamine system. Furthermore, the clinical symptoms of the animals have been related, in terms of the degree of parkinsonism, to the amount of tracer uptake seen with PET. The project has found that more parkinsonian animals have less striatal uptake of the tracer. Finally, the first series of grafted animals has now been completed. Imaging evidence has been found of focal areas of tracer uptake in the animals grafted with active tyrosine hydroxylase or dopamine decarboxylase-producing grafts, as compared with sham-grafted animals. Although quantitation of uptake has not been completed, these results are promising for detection of the physiological and biochemical results of the grafts.

76. Title: Cloning Genes for Atherosclerosis and Respiratory Therapeutics**ID:** LBL95-05**PI:** Edward Rubin
Life Sciences Division**Phone:** 510 486-5072

Partner: Rhone-Poulenc Rorer
Collegeville, PA

FY 97 Funding: \$202K **Total Project Funds:** \$583K

This project involves the use of transgenic mice to identify genes involved in pulmonary and arterial disorders (i.e. atherosclerosis). These studies utilize the unique ability to model genetically engineered mice after the human condition, and thus identify genes which contribute to the human condition. These studies are, in large part, dependent on LBNL's ability to create libraries of the human genome in transgenic mice. The creation of a 1.5 Mb in vivo library of chromosome 5q31 in a panel of transgenic mice has been completed. These animals have been analyzed with regard to distinct phenotypes associated with the presence of human DNA. The project has demonstrated specific developmental abnormalities in certain of these mice due to expression of the human DNA. Some of the genes discovered in this project have potential functions in tumor suppression, double-strand DNA repair, and transcription factor. The industry partner has greatly increased their projected funding; their total commitment, including in-kind contributions and a follow-on grant, exceeds \$14 million. This project contributes to DOE's mission in genome research and enhances laboratory core capabilities in molecular techniques.

77. Title: Enzymatic Remediation of Waste Streams

ID: LBL95-37

PI: Alexander Glazer
Material Sciences Division

Phone: 510 642-3126

Partner: Enzymol International, Inc.
Columbus, OH

FY 97 Funding: \$69K **Total Project Funds:** \$335K

This project is designed to develop the enzyme soybean peroxidase (SBP) for use in the degradation of toxic waste chemicals such as polychlorobiphenyls (PCBs), nitroaromatics, and chlorophenols (CPCs). These highly toxic materials, of which several hundred million pounds have been released into the environment as a result of agricultural, industrial and munitions uses, are extremely persistent. Despite claims to the contrary, there is presently no generally applicable technology for

their remediation. They resist bioremediation and give rise to other toxic compounds on incineration. Horseradish peroxidase can catalyze the reaction between hydrogen peroxide and phenols and aromatic amines, to remove them from waste streams, but this is not economically practical because of the cost of the enzyme. SBP is much less expensive and promising. To date in this project, several compounds have been tested to see if they can be removed by SBP-mediated peroxide reactions. Nitro-aromatic and nitro-aliphatic compounds were not found to react, but several important compounds were found to react well, including phenol, p-alkyl phenols, 4-chlorophenol, and highly substituted chlorophenols such as pentachlorophenol (PCP). Reaction products have been investigated; these are typically polymers and oligomers. Surfactants have been tested to improve the PCP reaction. The SBP-peroxide process has also been tested on an authentic waste stream containing phenol at moderate concentration, resulting in 95% phenol removal. Waste streams with high concentrations of phenol may present a complication (inactivation of SBP by higher concentrations of peroxide), but a reactor system to circumvent this problem has been conceptualized. This project directly supports DOE's program in environmental quality and remediation.

78. Title: Distributed Health Care Imaging on the National Information Infrastructure (NII)**ID:** LBL95-48**PI:** William Johnston
Information and Computing Services Division**Phone:** 510 486-5014**Partner:** Kaiser Foundation
The Permanente Medical Group
Oakland, CA**FY 97 Funding:** \$177K**Total Project Funds:** \$426K

Health care systems are increasingly collecting high-volume image data used for diagnostic purposes such as X-ray, CT, MRI, and cardio-angiography at central facilities remote from the clinical physicians. The goal of our project was to set up a prototype system to allow access by the referring physicians at several Kaiser Permanente sites to the raw image data taken at the centralized facilities. Software was written to digitally capture cardio-angiography data from the Phillips instrument at Kaiser's San Francisco Medical Center Catheterization Laboratory, to transmit it over a metropolitan ATM network to a large distributed parallel storage system at LBNL. A Web-based image library system was used to catalogue the images graphically and textually and to return URLs that could be used to access the original data. A video player was written that could be launched by a Web browser

and would display both the compressed and non-compressed video data across the network. This system was available on a daily basis to Kaiser doctors for a period of about 4 months for evaluation. The doctors found the functionality of the system to be very promising as it gave them access to the raw data within a few hours of when it was taken, replacing the current choices of driving to San Francisco, having the film sent to them, or only reading the cardiologist's report. The image library system has been recently extended to deal with additional types of medical images, specifically DICOM format MRI and NM data. This allows any platform that can run a Web browser access to an uniform interface to a large variety of images. Our goal is to replace the modality-specific, location-specific access to images that is currently required. The Web interface to the cardio-angiography data was accessed from the internal Web-based patient record system being developed by Kaiser Division of Research. The interface to DICOM images could also be easily integrated. this projects supports the DOE mission in health applications and advanced computing.

79. Title: Structure of the Erythropoietin Receptor**ID:** LBL95-49**PI:** Thomas Earnest
Structural Biology Division**Phone:** 510 486-4603**Partner:** Amgen, Inc.
Thousand Oaks, CA**FY 97 Funding:** \$324K**Total Project Funds:** \$844K

This project has a goal of determining the three-dimensional structure of the erythropoietin receptor (EPO-R) by x-ray and electron crystallographic methods. EPO-R is a transmembrane protein which binds to EPO (produced in the kidneys) and leads to the production of red blood cells, needed for oxygen transport. Recombinant human EPO-R is expressed in eukaryotic cells for purification, crystallization, and subsequent structure determination. Expression, purification, and crystallization of transmembrane proteins are all exceedingly difficult tasks compared to the case of water-soluble proteins, as is evidenced from the small numbers of structures of membrane proteins available. Once the structure is obtained, it will be analyzed to elucidate the structural mechanism of this signal transduction pathway, and to determine if small molecules can act as activators. EPO-R is homologous to a large number of other members of the cytosine receptor superfamily and thus can serve as a basis for attempts to understand the function of these molecules as well. EPO-R expression was tested in cell suspensions from yeast and hamster ovaries; a very low level of expression was

found. An insect-cell approach gives better yield but results are variable. Cell lysis requires the presence of detergents; the best yield was with alpha-lyso-phosphocholine. Purification is based on an EPO-affinity column, low-pH elution, and anion exchange chromatography. It has been shown that EPO-R eluted at low pH can still bind EPO. An EPO cold displacement assay indicates that purified, full-length EPO-R binds EPO with the same affinity as the well-characterized extracellular domain. Recently, large amounts of EPO-R have been expressed in *E. coli*. Inclusion bodies are formed from which EPO-R is solubilized and refolded. Initial crystallization trials yielded positive results and conditions for growing large crystals are being explored. Research in this project area supports the DOE mission in medical applications and biotechnology.

80. Title: Identification of Genes Affecting the Immune System, using Chromosomal Rearrangements in Mice

ID: ORL95-02

PI: Lisa J. Stubbs
Life Sciences Division

Phone: 423 574-0864

Partner: Darwin Molecular Corporation
Bothell, WA

FY 97 Funding: \$197K

Total Project Funds: \$635K

The biotechnology segment of the pharmaceutical industry, which combines the use of cloned genes and genetic information with advances in chemistry, is emerging as an important producer of novel drugs, therapeutics, and diagnostics. Mouse mutants can represent invaluable raw materials to this industry, aiding in the identification of health-related genes and serving as model systems in which disease progression and the efficiency of new treatments can be assessed. This project explores the utility of a specific class of mouse mutations - those carrying chromosomal rearrangements - as a means of identifying genes required for proper development and function of the immune system. The project has involved application of specific screens that are aimed at identification of immune defects to a collection of over 200 mutant mice; basic pathology, weight gain/loss, and other characteristics that would permit efficient detection of health-related disorders have also been examined. These studies have led to the identification of several new mutations exhibiting clinically-relevant immune system defects, as well as several mutant stocks expressing disorders of the kidney, liver, and other organ systems. Additional novel phenotypes are expected to be uncovered as immune assays, pathology studies, and chromosome analyses are completed and collated. These mutant-screening efforts are yielding a valuable series of new mouse models that will aid in the discovery and testing of treatments for human immune-related disease. This project directly supports the DOE Human

Genome program through its increasing emphasis on functional genomics, since readily clonable mutations with specific phenotypes facilitate efforts to relate DNA sequence to function. Many of the mutations already produced through this project are being used in DOE-supported gene-mapping and positional-cloning research.

81. Title: Rapid Prototyping of Bioceramics for Implants**ID:** ORL95-12**PI:** April D. McMillan
Metals and Ceramics Division**Phone:** 423 241-4554**Partner:** Smith and Nephew Richards
Memphis, TN**FY 97 Funding:** \$64K**Total Project Funds:** \$275K

The purpose of this project is to develop net-shape forming methods for directly creating dense Hydroxyapatite (HA) ceramic otologic implants for commercial applications. HA, in both dense and porous forms, is being used in bone-replacement surgery because of its well established biocompatibility. Otologic implants, because of their small size, are costly to manufacture by the traditional approach of machining from dense HA ceramic billets. Using ORNL's gelcasting process, these parts may be cast as near net-shape with very little finish machining, dramatically increasing product yields and reducing costs. The technological hurdles that have been overcome to demonstrate gelcasting as a viable technique for this application include: development of a biocompatible monomer/dispersant system suitable for HA powder; maintenance of dimensional tolerances in small components; and attainment of adequate green strength, green density, and economical-process yields. ORNL is developing an improved understanding of the gelcasting process, and is demonstrating the necessary proof-of-principle to stimulate the transfer of this technology to the biomedical sector. This project also enhances DOE programs in advanced ceramics and manufacturing.

82. Title: Subsurface Microbial Culture Collection: Characterization and Screening**ID:** PNL94-36

PI: F. Blaine Metting
Earth Systems Sciences Division

Phone: 509 372-0317

Partner: ZymoGenetics, Inc.
Seattle, WA

FY 97 Funding: \$170K

Total Project Funds: \$450K

The purpose of this project is to use molecular and physiological methods to characterize the DOE Subsurface Microbial Culture Collection (SMCC). This should result in the identification of the systematic relationships, metabolic diversity, and culturing requirements of individual strains so that subsets of strains can be screened using molecular, cellular, and whole animal protocols to reveal potential pharmacological activities. The pharmaceutical and biotechnology industries have traditionally screened microorganisms and other natural products for novel bioactivities leading to drug development. Recent scientific advances in molecular biology have resulted in new, highly specific methods for screening natural products for useful pharmacokinetic activities, thus significantly increasing the chances for identifying new drugs. The limitation to industry is access to novel genetic resources. Preliminary research suggests that the SMCC may be a unique genetic resource. The benefits to industry and society from the research would derive from the discovery of novel biological activity in one or more of the bacteria in the collection that could form the basis for development of a new drug(s). Targets for this project include cancer, AIDS, diabetes, and other diseases. Results to date include the successful sequencing and analysis of a ~1000 base pair region of the 16S rRNA gene from subsets of isolates in six phylogenetic groups of bacteria: the high G + C and low G + C Gram-positive bacteria; the α , β , and γ subdivisions of the Proteobacteria; and the Flexibacter/Cytophaga/Bacteroides group. Most are new species, some of which have been described in the scientific literature. Some represent new genera. In addition, the diversity of subsurface Arthrobacter and Sphingomonas isolates, both known to harbor broad biodegradation potential, was assessed. Cluster analysis comparisons of metabolic traits revealed distinct physiologic profiles, suggesting a greater overall diversity than indicated by the 16S rRNA gene sequencing. Screening isolates for aromatic hydrocarbon degradation provided additional evidence that subsurface microbial communities are capable of catabolizing a wide range of compounds. This project supports DOE missions in environmental and remediation research.

83. Title: Peptide Nucleic Acid Arrays and Fluorescence Resonance Energy Transfer to Identify and Enumerate Microorganisms in Environmental Samples

ID: PNL97-11

PI: Darrell P. Chandler
Environmental Microbiology Department

Phone: 509 375-2543

Partner: PerSeptive Biosystems
Framingham, MA

FY 97 Funding: \$125K **Total Project Funds:** \$750K

This project will contribute to DOE's mission in environmental quality by enabling the detection of specific bioremediative and/or pathogenic microorganisms in environmental (soil, sediment, groundwater) samples with greater speed, ease, and sensitivity. This will be accomplished by developing and coupling automated sample processing with hybridization of the sample extract using peptide nucleic acid (PNA) probes. The transfer of polymerase chain reaction (PCR) and genosensor microarray technology to environmental samples (e.g. air, soil, sediment, sludge, water, food) is problematic, because environmental nucleic acid extracts contain copurified inhibitors that interfere with enzymatic processes such as PCR. Manual nucleic acid extractions are also too slow, time consuming, or expensive to find routine use in applied environmental settings. In this project, these limitations will be addressed by (1) developing an automated flow injection system for nucleic acid purification from environmental samples, and (2) using PNA hybridization coupled to fluorescence resonance energy transfer (FRET) to amplify the hybridization signal. A novel, prototype sample processing device has been constructed, and research into the optimal solution hybridization conditions for PNAs (which are products uniquely available from PerSeptive Biosystems) and low-copy nucleic acids (i.e. ≤ 1 ng or 10^6 cell equivalents) in complex environmental matrices has been initiated. The automated extraction device will greatly simplify and speed nucleic acid isolation and purification from environmental matrices and provide an up-front processing capability for nucleic acid delivery to genosensor arrays. PNA technology will facilitate the recovery of nucleic acids from environmental matrices and simplify hybridizations on genosensor microarrays. Solid surface FRET detection of nucleic acid hybridization should minimize the requirement for PCR amplification labeling and/or detection of target sequences and provide rapid and quantitative detection of microorganisms in environmental samples with sensitivity approaching that of PCR (ca. 100 cells). Collaboration with PerSeptive Biosystems and a future genosensor array company will ensure meaningful technology transfer to the industrial collaborator(s) for possible application in environmental microbiology, biotechnology, food science, and agricultural markets while maintaining the DOE's and PNNL's long-standing leadership and support of environmental research.

Nuclear Medicine**84. Title: High Beam Current, Low Energy Targetry for Production of Radioisotopes for Positron Emission Tomography****ID:** BNL95-04**PI:** David Schlyer
Chemistry Department**Phone:** 516 344-4587**Partner:** AccSys Technology Inc.
Pleasanton, CA**FY 97 Funding:** \$220K**Total Project Funds:** \$813K

The purpose of this project is to develop targetry for the low energy (7 MeV), high beam current (>100pA) accelerator being marketed by AccSys Technology. These targets will enable such an accelerator to provide a reliable source of positron emitting short-lived isotopes for use in Positron Emission Tomography (PET). The project team's extensive experience in designing cyclotron targets is an advantageous starting point for these new, more demanding targets. A 4 MeV prototype accelerator supplied by AccSys Technology Inc. is being utilized to carry out these design tests. The critical tasks which must be accornplished are the design of very thin vacuum isolation windows which will withstand the very high pressures generated inside the targets, and of very efficient heat removal systems for use with these targets as a result of the high power density. Without these targets, the accelerator can not be used for production of PET isotopes and, therefore, a large potential market is closed to the accelerator manufacturer. There has been significant progress during the past year. The accelerator has been installed at BNL, and the first beam has been extracted. The full energy has not yet been achieved, but that milestone is imminent. The targets have been designed, built, and tested on the cyclotron and are now on the accelerator beam line awaiting achievement of full energy. The work done in developing these targets has been published in two articles and has been presented at three international meetings. This project directly supports the DOE mission in development of instruments for medical applications.

85. Title: A Radiopharmaceutical for the Treatment of Cancer-Related Bone Pain**ID:** BNL95-01

PI: Suresh Srivastava
Medical Department

Phone: 516 344-4459

Partner: Diatide, Inc.
Londonderry, NH

FY 97 Funding: \$255K

Total Project Funds: \$770K

The goal of this project is to develop tin-117m Diethylenetriamine-pentaacetic acid (DTPA) as a commercial radiopharmaceutical for the treatment of cancer-related bone pain. In approximately one-half million patients every year in the U.S. the cancer spreads to the bone. Most of these patients experience severe chronic pain which causes immobility and requires the use of major narcotic analgesics. In contrast to competitive technologies, Sn-117m DTPA provides substantial relief of bone pain without causing intolerable bone marrow toxicity or the adverse effects associated with the use of narcotic treatments. Considerable progress has been achieved both at BNL and by the industrial partner. A phase II clinical trial in 47 patients with advanced osseous metastatic involvement (26 at BNL and the rest at other collaborating institutions) showed substantial or complete pain relief in about 75% of the cases. At the recommendation of the Food and Drug Administration, extended phase II/III clinical trials (sponsored by Diatide) in about 500 prostate and breast cancer patients are expected to begin in September 1997. If these trials are successful, tin-117m DTPA is expected to benefit this segment of patient population through the commercialization by Diatide, Inc. of this technology. Research on and development of radiopharmaceuticals is a long standing DOE mission.

86. Title: Medical Accelerator Technology

ID: LBL94-36

PI: William Chu
Accelerator and Fusion Division

Phone: 510 486-7735

Partner: General Atomics
San Diego, CA

FY 97 Funding: \$126.9K**Total Project Funds: \$1,500K**

For more than four decades, under the auspices of DOE's Medical Applications program, LBNL has developed technologies of treating human cancer using accelerated heavy charged particle (proton and heavier ion) beams. In 1991 the first hospital-based medically dedicated proton accelerator facility was built in Southern California, and now another hospital-based facility is being built in Boston by commercial firms. Although the first facility was built by a national laboratory, the second facility is being built as a turn-key system by the private sector, and the latter trend will continue. In this project, LBNL and the industry partner are cooperatively developing technologies to channel the extracted proton beam from the accelerator to the treatment room, and then deliver it accurately into the treatment volume in a patient. Specifically, the project has developed beam transport systems to bring the protons to the treatment rooms; rotating gantries to aim the treatment beams precisely into patients from any angle; and patient positioners to align the patient accurately relative to the treatment beams. A patient treatment delivery system will be developed that controls the radiation dose in the patient, and hardware will be developed to improve accelerator performance. The majority of the system's final design and fabrication is complete. The accelerator has been installed and is being tested. Other components are being fabricated and installed. Commissioning of the facility is scheduled for 1998. Development of an integrated ion source-low energy beam transport system and an advanced patient treatment nozzle are scheduled for 1998.

Advanced Computational Technology Initiative**87. Title: Development of a New Generation Framework for Parallel Reservoir Simulation****ID:** ANL ACTI 95-95**PI:** Bill Gropp
Mathematics and Computer Science**Phone:** 630 252-5218**Partners:** ARCO, BP Exploration, Chevron, Conoco, Cray Research, IBM, Landmark Graphics, Mobil, Scientific Software-Intercomp, Schlumberger-GeoQuest, Texaco, Unocal
Plano, TX**FY 97 Funding:** \$150K **Total Project Funds:** \$1,800K

The simulation of petroleum reservoirs is an important component in the development of more efficient techniques in oil recovery. Current simulators used routinely in industry are fundamentally limited in the size and complexity of the problems that they can handle. Only through the use of parallel computing will the industry be able to tackle problems of current interest. Increasingly, science and engineering activities are becoming more dependent on computational modeling and simulation. This project provides much underlying research and development (the physics, mathematics, and computer science) for the next generation of simulator codes. The results of this project also closely relate and apply to the simulation, and therefore understanding, of underground pollution plumes. Industry involvement is as advisors to the project specifying the industry's needs (and where possible as contributors). The main work is performed by ANL and the University of Texas at Austin. At any appropriate point in time when resultant research elements are ready for application, the industry participants, whether petroleum companies or service companies, are able to employ/adapt this work into their own proprietary simulators. Code development for the simulator framework has proceeded with the successful development and testing of the 2-D fully implicit equation-of-state compositional model. A 3-D extension of the EOS compositional model is in progress. A 3-D black oil simulator with multiblock geometry and mortar interfaces is also being developed under the framework. A prototype has been developed and is being tested for a two phase problem. The multiblock data structure is based upon the Distributed Adaptive Grid Hierarchy, which is now fully integrated into the framework. In addition to the many presentations concerning various aspects of the project, the publications include twenty-five journal articles, technical reports, and conference proceedings. Advanced methods in computer simulation for development of oil and gas exploration supports a major DOE mission in energy security.

88. Title: A High-Resolution Subsurface Electromagnetic Imaging Tool**ID:** LBL94-14**PI:** Ki Ha Lee
Earth Sciences Division**Phone:** 510 486-7468**Partner:** Western Atlas Logging Services (WALS)
Houston, TX**FY 97 Funding:** \$112K**Total Project Funds:** \$545K

The goal of this project is to develop and apply a new survey method and instrumentation for high-resolution subsurface electromagnetic (EM) imaging. The technology is critically important in assisting improved management of petroleum reservoirs for increased oil production. This project supports DOE's mission in enhanced U.S. energy security. The basis of the technology is the new tomographic imaging technique via wavefield transform developed at LBNL. The theoretical basis of the wavefield transform has been known for some time, but Lee et al (1989) generalized it to include EM fields and demonstrated the usefulness of such a transform using a forward model study. In that study, wavefields were first obtained by numerical modeling, and corresponding EM fields were calculated by simple integration of these wavefields. Since then, effort has been focused on solving the inverse problem in which a diffusive EM field is transformed to a wavefield. The velocity of the wavefield is inversely proportional to the square root of the electrical conductivity; therefore, velocity mapping directly leads to conductivity imaging. Once fully developed, the technique could produce electrical conductivity images with a spatial resolution equivalent to that of the seismic imaging for the elastic parameters. This technology will enable WALS to bring the current state-of-the-art knowledge in underground imaging for increased production to the U.S. oil industry. This will directly help reduce U.S. energy dependence on foreign oil. To date, wavefield transform software has been prepared and delivered to WALS. The wavefield transform concept has been verified in a laboratory model study (1-dimensional). WALS is investigating integration of LBNL's prototype transmitter for subsurface measurement. For high accuracy measurements, extensive noise suppression and application of state-of-the-art electronics are required. Although this was time-consuming, progress has been good.

89. Title: Subsalt Imaging with Marine Magnetotellurics**ID:** LBL ACTI 95-90

PI: H. Frank Morrison
Earth Sciences Division

Phone: 510 486-5080

Partner: Scripps Institute of Oceanography, Amoco, BP, Chevron, Exxon, Geotools Corporation, NWG Associates, Texaco, Unocal, Conoco, and University of California at San Diego

FY 97 Funding: \$295K **Total Project Funds:** \$1,050K

Marine magnetotellurics (MT) is a new technique to augment seismic imaging in geological surveys. MT can reveal the size and thickness of underwater salt structures using differences in natural electromagnetic radiation in rock structures. This information can help researchers gauge the prospects for the sediment underlying the salt to be rich in oil or gas. LBNL is specifically assisting the partners in the development of software algorithms and computer simulation models used in the analysis of the MT data. Scientists at LBNL are initially developing inversion code for the two dimensional computer depiction of MT field data. In follow-on tasks LBNL will extend the code development to integrate seismic, gravity, and MT data for two and three dimensional depictions of survey areas. Initial work is on potential oil and gas fields in the Gulf of Mexico. A series of numerical simulations of 2D and 3D salt structures showed that MT should be able to map the base of salt structures. For field testing, a hardware package to acquire MT data from the sea floor was developed at Scripps, and data processing software was developed at LBNL and UC Berkeley. Initial field surveys have been conducted at two sites in the Gulf of Mexico. The first survey, conducted in August 1996, went well but the data were too noisy to be useful. The second survey, conducted in July 1997 after instrument problems were corrected, went extremely well, providing impressive imaging of salt shapes using new numerical codes developed for the project. Oil exploration companies are very interested in commercializing the results of this project.

90. Title: Optimal Fluid Injection Policy and Producibility in Fractured, Low Permeability Reservoirs

ID: LBL ACTI 95-92

PI: Tadeusz W. Patzek
Earth Sciences Division

Phone: 510 643-5834

Partners: Aera Energy (formerly CalResources, now merged with Mobil), Chevron CTPC, and Crutcher-Tufts

FY 97 Funding: \$202K

Total Project Funds: \$750K

In low-permeability oil reservoirs (diatomites, chalks and carbonates), primary production yields only 2 to 6% of the oil-in-place. Fluids such as water, steam or carbon dioxide are injected into these reservoirs to increase or sustain oil production and prevent reservoir damage. An optimal injection policy minimizes formation damage while maximizing oil production per unit volume of injectant. The purpose of this project is to provide the oil industry with tools to optimize injection policies in the low-permeability reservoirs. Fluid injection into low-permeability reservoirs either for pressure maintenance or secondary oil recovery is very difficult. On one hand, injection rates must be low enough to prevent reservoir damage from overpressuring and inducing unwanted fractures. On the other hand, these rates must be high enough to make the costly fluid injection process economic. Historically, the conflict between prudent reservoir management and meeting the injection targets has resulted in significant reservoir and well damage, injectant recirculation and irreversibly lost oil production. Current engineering practice is to develop injection policy from past experience, partial knowledge of the state of reservoir stress, production history, and limited predictions of future reservoir performance from numerical simulation. Although an oil field is a complex and highly coupled system, injectors are usually controlled individually, with constant set points, and without feedback among neighboring patterns. In this project, LBNL is developing, with the help of oil producers, an innovative set of Computer-Assisted-Operations (CAO) tools to promote higher and cheaper recovery from fractured, low-permeability diatomite fields in Kern County, CA. The tools function by making optimal, or "expert," decisions that balance fluid injection rate and fluid injection pressure goals versus the capacity of the reservoir to sustain hydrofracture extension and formation damage, with the subsequent decrease of oil production, injectant recirculation and well failure. Features of this project include: (1) real-time, well-by-well passive monitoring of injectors and their associated fractures, and the progress of injected fluid; (2) software tools to compile and evaluate the injector performance; (3) active monitoring of hydrofracture growth and well coupling; and (4) optimal model-based wellhead controllers of fluid injection. We have demonstrated that neural networks are capable of predicting injection rate as a function of wellhead injection pressure and vice-versa. The first generation of neural network controllers has been developed and deployed on Aera Energy's server. We have also observed that neural network predictions and, therefore, neural network controllers may become unstable if the injector dynamics changes sufficiently. To quantify better the injector dynamics, we have developed a software package, I.D.E.A., (Interactive Data Evaluation and Analysis Tool). We have found that at constant injection pressure, injection rate is remarkably constant. Therefore, either the injection hydrofractures grow with time, or the formation permeability increases with time, or both. We have expressed a simple theory of hydrofracture growth in terms of two easily measured field parameters, the early injection slope in linear transient flow, and the average injection rate. We then compared the prediction of our theory with the growth rate of hydrofracture area calculated independently (in our ORTP project) for two steam injectors. There is

remarkable agreement between our predictions and the independently estimated rates of growth of these two hydrofractures. The latter finding has a significant impact on the design of adaptive controllers of fluid injection into a low permeability rock. We have shown that pressure control may be stable, but rate control is not. A semi-continuous measurement of wellbore and hydrofracture acoustics may be required to update the controller parameters. We have constructed, and are evaluating for possible patent, an optimal controller to stabilize the injection rate near a prescribed value. The stability of an iterative procedure producing the controller has been studied. The time interval over which the controller is stable has been estimated. The controller can be extended in step-by-step mode over an arbitrary large time interval. The feedback from the real data can be input into the controller through the measurement and/or modeling of the growth rate of hydrofracture area. Research in well simulation supports the DOE mission in energy security.

91. Title: Advanced Computational Analysis of Drill Cuttings for Real-Time Well Site Decisions

ID: LBL ACTI 95-94

PI: Larry R. Myer
Earth Sciences Division

Phone: 510 486-6456

Partner: Meridian Oil, BP-America, and ARCO E&P Technology

FY 97 Funding: \$294K **Total Project Funds:** \$2,955K

The information available to the engineer on a drill rig platform is very limited about the properties of the subsurface being drilled thousands of feet below. Nonetheless, critical decisions must be made "on-the-spot" with regard to the stability of the well and the location and quality of reservoir rock. This project focuses on development of a new technology utilizing rock fragments produced during drilling to provide the information on rock properties needed to make these rig-site decisions. The successful implementation of this technology is estimated to yield at least 300 million barrels of oil equivalent, as bypassed producing zones are developed, and missed opportunities are reduced. This project combines a unique mathematical modeling capability developed at LBNL with oil company experience and expertise to solve the problem. The key to success of this technology is the development of efficient computational models which will be used to calculate flow properties including permeability, capillary pressure, relative permeability and mechanical properties based on images of the rock pore space derived from cuttings. This direct calculation takes into account the effect of microscale heterogeneity, thereby mitigating one of the major sources of uncertainty in currently available cuttings analysis methods. The needed highly efficient computational models for

flow are made possible by innovative application of graph theory, an established branch of topology. Other simple, inexpensive, direct, and indirect measurements of drill cuttings properties, which are available or under development, will be combined with the algorithms developed in this project. A three-year program is underway, bringing the technology to the point needed for commercialization. In this time period, development and testing will be completed for: graph-theory based computer models; well-site computer imaging and 3-D pore reconstruction techniques; a methodology for integration of model calculations with other direct and indirect measurements; and a methodology to ensure that results are representative of bulk rock behavior. The original code utilizing graph theory, for calculations of flow properties in a 3-D pre network, has been significantly updated, including use of elements more representative of the observed pore space shapes. A new technique for measurement of capillary pressure using Wood's metal has been implemented. Object-oriented program design is a contribution to network simulation methods. New algorithms and simulations provide new understanding of the importance of pore space connectivity to flow properties. A new technique for measurement of capillary pressure using Wood metal was developed and used to obtain data on Nugget sandstone. This data was used as a basis for extensive simulations to validate the graph theory code. A comparison of three types of advanced imaging capabilities was complete: Microfocus X-Ray CT, Laser Scanning Confocal Microscopy, and Nuclear Magnetic Resonance. Microfocus CT is now being tested to obtain 3D images of epoxy pore space casts. Research in well simulation supports the DOE mission in energy security.

92. Title: Subsidence, Analysis, and Control of Oil and Gas Reservoirs**ID:** LBL ACTI 95-91**PI:** Don Vasco
Earth Sciences Division**Phone:** 510 486-5206**Partner:** Long Beach Department of Oil Properties (LBDOP), Tidelands Oil Production Company (TOPKO), THUMS Long Beach Company, and California State Lands Commission**FY 97 Funding:** \$41K**Total Project Funds:** \$223K

Subsidence is a drastic change in surface elevations usually caused when fluids are extracted from the subsurface causing vertical shifts in the rock formations. This project will develop computational tools to predict the surface subsidence associated with the development of oil and gas reservoirs, supporting DOE's mission in energy security. The approach is to couple a multiphase fluid flow simulation program to a routine for calculating surface displacements. The flow simulator chosen is the TOUGH2 package developed at LBNL. The pressure field produced by the TOUGH2 program

is input to the surface displacement program. A simple homogeneous poroelastic half-space model was used to propagate the pressure changes to the surface. Testing was initiated for both a simple five-spot well pattern undergoing both water and steam flooding as well as a configuration of 46 wells conducting a waterflood patterned after the North Robertson field in Texas. The influence of reservoir heterogeneity on the calculated surface displacements was notable. LBNL scientists have completed coding and debugging a finite difference routine to compute surface displacements in an arbitrary three-dimensional poroelastic medium. The routine is quite efficient and may serve as a component of a planned inversion routine. LBNL is currently using the finite difference routine to predict surface displacements based upon volume injection data. Additional work has concentrated on incorporating the effects of temperature changes on surface displacements. For a steam flood, reservoir and overburden temperature may have an effect on surface displacements, in addition to reservoir pressure, through the thermal expansivity of the poroelastic medium. This will allow reservoir simulations in which conductive heat transfer occurs from the reservoir to the overburden without requiring any more than the reservoir itself to be discretized. LBNL researchers are developing a code for determining variations in poroelastic parameters from volume injection and surface displacement measurements. This will allow researchers to calibrate the material properties using the waterflood data from the Wilmington, CA field. Based on this approach, investigators can then apply an existing inversion routine to determine volume changes in the reservoir from observed surface displacement data. In addition, the calibrated model, coupled with the reservoir simulator, may be used to predict surface displacement based upon various production scenarios. The subsidence software module has been delivered and demonstrated to the industry partners. Modeling of a steam flood operation showed qualitative agreement with observed reservoir behavior. A preliminary inversion of 20 years of surface displacement data has been completed..

93. Title: Advanced Flux Visualization and Virtual Reality for Reservoir Engineering**ID:** LBL ACTI 95-93**PI:** Edward W. Bethel
Computing Sciences Division**Phone:** 510 486-7353**Partner:** Western Atlas Logging Services (WALS)
Houston, TX**FY 97 Funding:** \$22K**Total Project Funds:** \$180K

The accurate determination of the mechanisms of recovery in an oil or gas reservoir will directly affect the recovery efficiency of a given reservoir. A number of tools are used by engineers to

understand the characteristics of these reservoirs, with flux visualization a key parameter. Unfortunately, current technology limits the engineer's ability to easily interpret the flux data. The objective of this project is to provide new computer visualization tools to enhance the ability of engineers to rapidly determine oil and gas recovery mechanisms. This is a core DOE/Laboratory mission in enhancing U.S. energy security. A multidisciplinary team of computer and earth scientists at LBNL is exploring the use of advanced user visual interfaces, commonly called "Virtual Reality" (VR), coupled simulation, and modeling software. Working closely with industry, these efforts have resulted in an environment in which VR technology is coupled with existing visualization and computational tools. Two specific geoscience application areas are under development. In the first, LBNL scientists have developed VR technology to manipulate three-dimensional input parameters, such as the spatial location of injection or production wells in a reservoir simulator. In the second, LBNL scientists have demonstrated how VR technology can be used to manipulate visualization tools. The information is presented to the user in the form of a virtual injection well and is then applied to computing streamlines through fluxes. LBNL's project focus is on creation of a software infrastructure to support VR. This infrastructure is a collection of software "building blocks" and a visual programming interface for constructing a complete "program" for visualization. LBNL scientists can rapidly and easily interface several different VR input devices, such as a Spaceball or magnetic trackers, to a variety of computational or visualization tools. To date, software deliverables have been made available in source code form via the Internet. These deliverables include audio/visual modules for visualization of flux data, and manipulation and processing of VR device events.

94. Title: Advanced Computational Tools for 3-D Seismic Analysis**ID:** ORL ACTI 95-09**PI:** Jacob Barhen
Computer Science and Mathematics Division**Phone:** 423 574-7131**Partner:** Society of Exploration Geophysicists
Tulsa, OK**FY 97 Funding:** \$107K**Total Project Funds:** \$400K

The development of 3-D structural and stratigraphical models of hydrocarbon reservoirs is crucial for the future ability of the exploration industry to economically discover and produce oil and gas. It requires extensive use of 3-D seismic data. The goal of this project is to develop advanced-computational tools for 3-D seismic analysis, and test these products using a model data set developed under the joint aegis of the United States Society of Exploration Geophysicists (SEG) and the

European Association of Exploration Geophysicists (EAEG). Since these computational tasks are extremely expensive and new ideas and methods are necessary to render them timely, efficient, and cost-effective, ORNL's aim is to develop leading edge U.S. capabilities in this application area of vital importance to DOE and the national economy by leap-frogging state-of-the-art seismic computation methods currently dominated by European technology. A major degradation of seismic signals of the subsurface structure usually arises from near-surface geologic irregularities due to misalignment of signals caused by unpredictable delays in recorded travel times of seismic waves in the source and receiver. The occurrence of severe residual statics, and the significant noise contamination, render the automatic identification of large static shifts extraordinarily difficult. The first objective of this project is to investigate the applicability of the TRUST global-optimization methodology (invented at ORNL) to residual statics problems, enhance the algorithms (by developing a new code, TRUST_RS), and carry out demonstrations using the SEG/EAEG dataset. The second objective is to demonstrate the viability of Artificial Neural Network (ANN) algorithms in estimating oil field reservoir parameters from remotely sensed seismic data. The specific objectives of this task are to (1) demonstrate the use of the k-fold cross validation technique to obtain confidence bounds on an ANN's accuracy statistic from a finite sample set; (2) demonstrate that the classification accuracy of an ANN can be dramatically improved by transforming data into an appropriately-defined feature space; and (3) develop faster learning algorithms. There have been two major technical accomplishments in this work: the methodology development and demonstration of TRUST_RS, and the successful proof-of-concept application of neural networks to reservoir parameter estimation. These accomplishments are documented in a journal article (Science, 276, 1094-1097, May 1997), and in two refereed conference papers (NEURAP'97, Marseille, France). There has also been one patent disclosure with an additional one forthcoming.

Selected Quick Response Projects

Personnel Exchanges

95. Title: A Solarblind Radiation Detector

Brookhaven National Laboratory
E. K. Souw (516) 344-5407

Industrial Partner:

Northrop Grumman Corporation
T. Donnellan (516) 575-5474

Total Project Funding: \$40K

BNL and Northrop Grumman have completed a personnel exchange project to investigate the development of a robust and solarblind radiation detector. This design allows for wider field applications since measurements of samples can be taken in the field rather than returned to a laboratory for more costly analysis. In addition, the solarblind radiation detector is not affected by ambient light, it is suited for use in nuclear waste management sites. This personnel exchange builds on a previous collaboration to develop a chemical-vapor-deposited (CVD) diamond radiation detector, and will utilize the CVD wafer in combination with a detector made from Cadmium Zinc Telluride. Northrop Grumman plans to expand its ongoing efforts to grow CVD diamond wafers with consistent radiation detector properties. BNL researchers will study and improve detector material qualities, and to test the detector system. BNL and Northrop Grumman have performed preliminary tests to determine diamond detector limits (since the current design detects only alpha and low level beta particles), detector calibration procedures, trap states, and polarization and quenching techniques. This project supports DOE's mission in development of radiation detection methods and instrumentation.

96. Title: Optimization of Selective Oxidation of Olefins

Brookhaven National Laboratory
J. Hrbek (516) 344-4344

Industrial Partner:

Eastman Chemical Company
E. Middlemas (423) 229-6427

Total Project Funding: \$20K

BNL and Eastman Chemical Company are participating in a personnel exchange project to provide a molecular level understanding of chemical reactions that take place at the surface of the model silver catalyst during the selective oxidation of olefins. This objective will be achieved by examining how the interplay of the electronic and structural properties of a material affects the chemical reactivity of the catalytic surface. Adsorption, desorption, and heterogeneous catalytic reactions will be explored on a model Ag(111) catalyst. The personnel exchange project will combine physical characterization of the catalyst with investigations of its chemical reactivity using state-of-the-art ultra-high vacuum (UHV) surface science systems. Thermal desorption spectroscopy is a UHV technique that allows the study of desorption kinetics and energetics of molecules interacting with surfaces. The sample is covered with a chemisorbed layer of molecules under study and positioned in front of a mass spectrometer, and its temperature is increased linearly. Different desorbing species are monitored as a function of temperature; the data are analyzed to determine the activation energies, pre-exponential factors, and lateral interaction energies of species desorbing from the surface. BNL and Eastman will study desorption of 1,3-butadiene and 3,4-epoxy-1-butene from a clean, Cs-promoted, and O-modified Ag(111) surface. Cesium is a known promoter in selective oxidation reactions, and the project team will use both metallic cesium and cesium chloride to study the effect of promoters on the desorption. By combining several surface science techniques (thermal desorption, photoemission, and low energy electron diffraction), BNL and Eastman will study the chemical bonding, thermal stability, and electronic structure of the promoter and its possible effects on the single-crystal substrate. This project contributes to the DOE mission in the applications of research advances in materials and chemical sciences, and the use of advanced instrumentation techniques.

Technical Assistance

97. Title: Plasma Cleaning Methodology for Electron Microscopy

Argonne National Laboratory
Nestor J. Zaluzec (630) 252-5075

Industrial Partner:
South Bay Technology, Inc.
David Kenriks (800) 728-2233

Total Project Funding: \$4K

South Bay Technology (SBT) is a sub-licensee of ANL, Patent No. 5,510,624 (<http://www.amc.anl.gov/docs/anl/techtrans/plasmacleaning.html>). This patent describes the experimental methodology to remove the source of hydrocarbon contamination from specimens studied using any type of electron microscope. The technology involves subjecting the specimen and the specimen stage to a reactive plasma gas (either DC or RF excited) which efficiently removes a wide range of conditions in which the project team determined the optimal power levels, gas composition, gas pressure, and processing times. The results of these measurements were published as two papers in the scientific literature. These papers are available via the ANL web site (www.amc.anl.gov). As a result of this project, SBT was able to increase production and sales (now having scientifically sound data to prove the capabilities of their unit). In addition, SBT has purchased a small company which manufactures the plasma generators for the cleaning system to keep all the manufacturing in-house.

98. Title: New Methods for Semiconductor Photoresist Removal

Pacific Northwest National Laboratory
Max R. Phelps (509) 375-6678

Industrial Partner:
Enhanced Chemical Applications
William Mullee (503) 310-6460

Total Project Funding: \$5K

Water contamination from photoresist masking is an important environmental problem in the semiconductor manufacturing industry. Because the Pacific Northwest is both environmentally sensitive and a major center for semiconductor manufacturing, this problem has taken on heightened

importance. Enhanced Chemical Applications (ECA) is a small company that provides technical services to the industry. ECA asked PNNL to investigate the feasibility of using a unique processing method that utilizes environmentally benign solvents to remove photoresist masking in the semiconductor manufacturing process, that would greatly reduce the amount of water that becomes contaminated. PNNL utilized its substantial expertise to demonstrate the feasibility of the removal method, but only in combination with another unique technology that was also developed at PNNL. The combination of the two processing technologies that was achieved in this project has the potential to contribute substantially to DOE missions in environmental quality. PNNL and ECA are now collaborating (along with other firms) via private contracts to commercialize this photoresist removal approach.

Small CRADAs**99. Title: Use of Reactor-Produced Radioisotopes for Prevention of Restenosis after Coronary Angioplasty**

Oak Ridge National Laboratory
F. F. Knapp, Jr. (423) 574-6225

Industrial Partner:
Mallinckrodt Medical, Inc.
Edward Deutsch (314) 895-8925

Total Project Funding: \$50K

The purpose of this project is to develop and evaluate the effectiveness of various reactor-produced, beta-particle emitting radionuclides for the inhibition of restenosis (the reformation of plaque deposits in arteries), which is often observed following coronary artery angioplasty and stent implantation. Radioisotopes that decay by beta-particle emission are useful for therapy, and in particular, have been suggested as an attractive new approach to inhibit the rapid growth of arterial smooth-muscle cells following therapeutic balloon-inflation angioplasty for opening clogged coronary arteries. Coronary heart disease leads to myocardial infarction and is a major cause of death in the U.S. Myocardial infarctions result from atherosclerotic-plaque deposits in the coronary arteries, reducing blood flow through the arteries that supply oxygen and nutrients to the heart muscle. The biological response to such controlled vessel damage after angioplasty is the stimulation of accelerated growth of the smooth muscle cell layer that lines the arteries, leading to restenosis. Since restenosis following angioplasty is a major clinical problem (based on conservative estimates from the American Heart Association there are over a 120,000 incidents of coronary restenosis in the U.S.), a major current area of vascular-biology research is the development of methods to inhibit restenosis after angioplasty. Although a variety of pharmacological approaches (such as the use of corticosteroids, heparin, and other drugs) are being explored, ionizing radiation has been found to be one of the most efficient, easily performed procedures, and is expected to be very cost effective and useful. Since the radiation must pass through the plastic balloon and penetrate the smooth muscle layer, a growing body of experimental data suggests that high-energy beta particles (> 1 MeV energy) are effective and safe for localized irradiation. Based on the high incidence of coronary-artery disease in the U.S. and the significant costs associated with the treatment of restenosis after coronary angioplasty, this project has very high probability of identifying the best candidates for detailed clinical evaluation that would be expected to be further developed into important new clinical agents for therapy following the use of angioplasty for the coronary arteries and atherosclerosis of other arterial systems. This project presents a good opportunity to use DOE-sponsored advances in nuclear medicine to make a valuable contribution to a very important U.S. health care issue.

100. Title: Microwave Processing of Advanced Electro-optic Materials

Oak Ridge National Laboratory

Lynn A. Boatner (423) 574-5492

Industrial Partner:

Commercial Crystal Laboratories, Inc.

Michael Urbanik (941) 643-5959

Total Project Funding: \$70K

The purpose of this project is to exploit the unique aspects of microwave heating; develop new crystal-growth and post-growth microwave heating processes; and develop an improved level of understanding of the effects of microwave interactions. Electro-optic materials in the mixed-solid-solution system, potassium tantalate-potassium niobate (KTN), have electro-optic coefficients, refractive indices, and other physical properties that make these materials superior candidates for numerous advanced optical and opto-electronic applications. The devices in which single-crystal KTN can be utilized (e.g., optical mixers, phase-conjugate mirrors, optical modulators, switches, and couplers) are applicable to the monitoring and control of energy-related devices; to systems for environmental monitoring; and in optical sensing and communication systems for national security applications. However, the penetration of KTN into the mainstream electro-optic marketplace is limited due to the difficulty of growing large homogeneous single-crystals. Homogeneity is a central issue because the growth of KTN single crystals involves the solidification of a binary solid solution. Since such systems do not solidify congruently, minute temperature fluctuations at the growing solid/liquid interface create compositional fluctuations that are manifested in the form of deleterious optical striations throughout the boule. There are indications, however, that microwave-heating processes can create "non-thermal" effects, i.e., enhanced rates of diffusion, which can be exploited in the homogenization of KTN crystals to make this material a viable and important commercial product. Accordingly, a project in which compositionally striated KTN crystals are treated under controlled conditions of microwave frequency, power, and temperature will not only provide a potential new processing technology and a new material for the electro-optics community, but will also result in an excellent model system for studying and understanding non-thermal diffusion in microwave-assisted heating processes in general. This work enhances DOE basic materials and national security programs.

APPENDIX A

CROSS REFERENCE TABLE FOR MULTI-YEAR PROJECTS*

Program Focus Area	National Laboratory				
	ANL	BNL	LBNL	ORNL	PNNL
Advanced Materials					
Synthesis and Processing	1, 2, 3, 4		5, 6	7	8, 9, 10, 11
Materials Design and Characterization		12, 13, 14, 15	16, 17, 18	19, 20, 21, 22, 23, 24	
Intelligent Processing/ Manufacturing Research					
Sensors, Instrumentation, and Processing		25, 26, 27, 28, 29	30, 31	32, 33, 34, 35	36, 37
Fabrication		38		39, 40, 41	42, 43
Modeling and Computing	44, 45			46	47
Sustainable Environments					
Energy Conversion and Storage	48, 49	50, 51, 52	53, 54	55, 56	
Energy Technologies	57			58, 59, 60	
Environmental Technologies	61, 62	63, 64	65, 66	67, 68	69, 70, 71, 72
Biotechnology	73	74	75, 76, 77, 78, 79	80, 81	82, 83
Nuclear Medicine		84, 85	86		
Advanced Computational Technology Initiative	87		88, 89, 90, 91, 92, 93	94	

* The numbers in this list refer to the project sequence numbers in this book.

APPENDIX B

ALPHABETICAL LIST OF INDUSTRIAL PARTNERS*

3M	7
AccSys Technology Inc.	84
Advanced Lighography Group	30
Advanced Research Development, Inc.	48
Advanced Vehicle Systems, Inc.	60
Aera Energy	90
Air Products	43
Allvac, an Allegheny teledyne Company	20
Alumax Corporation.	58
Aluminum Association	32
Amgen Corporation	79
Amoco Corporation	89, 49, 50
Amoco Oil Company.	57
Applied Genetics, Inc	74
Applied Natural Sciences, Inc.	61
ARCO	87, 91
Association of American Railroads	62
ASTeX	4
Babcock and Wilcox	63
BP America	91
BP Exploration	87, 89
California State Lands Commission	92
Catalytica	71
Chattanooga Area Regional Transit Authority	60
Chevron	57, 87, 89, 90
Cincinnati Milacron	68
Commercial Crystal Laboratories, Inc.	100
Continental Optical Corporation	28
Conoco.	87, 89
Consolidated Edision Company	63
Conway	40
Cray Research	87
Crutcher Tufts	90

*The numbers in this list refer to the project sequence numbers in this book.

Darwin Molecular Corporation	80
Diatech, Inc.	85
Digital Instruments, Inc	37
DuPont	15, 66
Durametallic Corporation	4
Eastman Chemical Company	96
Electric Transit Vehicle Institute	60
Electro-Motive Division of General Motors	62
E.I. du Pont de Nemours and Company.	42, 69
Energy Research Corporation	54
Engelhard/ICC	59
Enhanced Chemical Applications	98
Environmental Research Consortium	67
Enzymol International, Inc	77
eV Products Division of II-VI, Inc.	27
Exxon	89
Fauske and Associates, Inc.	45
Finnigan Corporation	36
Forrester Environmental Services, Inc.	64
General Atomics	86
General Motors Corporation Gear Center	10
General Motors Research and Development Center	47
Geotools Corporation	89
Grace Construction products (W.R. Grace & Co.)	14
Grumman Corporation	25
Health Tech Development	8
Hewlett-Packard Company	18
Honeywell, Inc.	24
Houston Applied Research Center	34
Howmet Corporation	11
Hughes Christensen	34
IBM	17, 87
Intermagnetics General Corporation	13
International Fuel Cells Corporation	52
InterScience, Inc	33
Kaiser Aluminum and Chemical Company	58
Kaiser Foundation	78
Landmark Graphics	87
Lear, Inc.	46

LeCroy Research Systems	26
Long Beach Department of Oil Properties (LBDOP)	92
Loral Control Systems	38
Lucent Technologies - Bell Laboratories	21
Magnequench International, Inc	19
Mallinckrodt Medical, Inc.	99
Marlow Industries Inc	23
Meridian Oil	91
Metal Matrix Composite Castings, Inc.	41
Mobile	87
Nartron Corporation	60
Nonvolatile Electronics, Inc.	24
Northrup Grumman Corporation	95
NWG Associates.	89
NZ Applied Technologies (NZAT)	6
PerSeptive Biosystems	83
Philips Petroleum Company	57
Power Conversion, Inc.	51
Pratt and Whitney	22
Praxair, Inc.	5
Quantum Group, Inc.	31
Rayovac	53
Research International	55
Reynolds Metals Company	39, 58
Rhone Poulenc-Rorer	76
Ross Products Division	9
Sagax Technology.	2
SCAAP Consortium	44
Schlumberger-GeoQuest	87
Scientific Software-Intercomp	87
Scripps Institution of Oceanography	89
SI Diamond Technology, Inc	1
Sigma Laboratories.	42
Smith and Nephew Richards Inc	12, 81
Society of Exploration Geophysicists	94
Solar Turbine	11
Software-Intercomp	87
Somatix Therapy Corporation	75
Sunsoft Corporation	16
South Bay Technologies	97

Southwire Corporation	7
Superconductive Components, Inc.	3
Symbol Technologies, Inc.	29
Teledyne Electronic Technologies	56
Teledyne-Laars	65
Texaco	87, 89
THUMS Long Beach Company	92
Tidelands Oil Production (TOPKO)	92
United Solar Technology, Inc.	42
University of California at San Diego	89
Unocal	87, 89
UOP Research Center	57, 72
USCAR	70
Waters Corporation	35
Western Atlas Software (WALS)	88, 92
Zimmer, Inc.	73
Zymogenetics, Inc.	82

APPENDIX C

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Adzic, Radoslav	52
Affinito, John	42
Ahluwalia, Rajesh	57
Alexander, Kathleen B.	22
Armstrong, Timothy	43
Atiya, Maged	26
Bader, Samuel	2
Barhen, Jacob	94
Bates, John B.	55, 56
Bertozzi, Carolyn	16
Bethel, Edward W. (Wes)	92
Boatner, Lynn A.	100
Buchanan, Michelle V.	67
Bullock, Morris	15
Butcher, Thomas	63
Butler, William H.	24
Cairns, Elton	53, 54
Campbell, Allison	8, 9
Chandler, Darrell P.	83
Cheng, Robert	65
Christen, David K.	7
Chu, William	86
Courtright, Edward	10, 11
Crabtree, George	3
Earnest, Thomas	79
Ellingson, William	73
Fairchild, Phillip D.	59
Francis, A.J.	64
George, E.P.	20
Glazer, Alexander	77
Gropp, Bill	87
Gruen, D.M.	4

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Gundel, Lara	31
Haller, Eugene E.	18
Hansen, James G.	41
Hayden, H. Wayne	39
Haynes, Tony E.	21
Hinchman, Ray	61
Holcomb, David E.	34
Horton, Joseph A.	19
Hrbek, J.	96
Isaacs, Hugh	12, 14
Jagust, William	75
Johnston, William	78
Jorgensen, James D.	49
Kendall, Richard	69
Knapp, F.F.	99
Krauss, Alan	1, 4
Kroeger, Donald M.	7
Lauf, Robert J.	40
Leung, Ka-Ngo	30
Ludtka, Gerald M.	46
Mahajan, Devinder	50
McBreen, James	51
McLarnon, Frank	54
McMillan, April D.	81
Metting, F. Blaine	82
Morrison, H. Frank	89
Myer, Larry R.	91
Negri, M. Cristina	61
Nicholas, John	71
O'Connor, Paul	27, 29
Orlando, Thomas	70
Patzek, Tadeusz W.	90
Padmore, Howard	17
Phelps, Max R.	98
Ramsey, J. Michael	35
Rubin, Michael	76
Sales, Brian C.	23
Schlyer, David	84
Sekar, Raj	62
Sigman, Michael	68

Smith, Mark	47
Smith, Neville	17
Smith, Richard	36
Somorjai, Gabor	66
Souw, E.L.	95
Srivastava, Suresh	85
Stubbs, Lisa J.	80
Sutherland, Betsy	74
Tackas, Peter	28
Taleyarkhan, Rusi P.	32
Thomas, C.E.	33
Thurston, Thomas	13
Vasco, Don	92
Visco, Steven J.	5
Wang, Yong	72
Warren, John	38
Wasielewski, Michael	48
Weber, David	44, 45
Williams, Gwyn	25
Xiang, Xiao-Dong	6
Xie, Sunney	37
Young, Jack P.	58
Zaluzec, Nestor J.	97