Research Activity: Synthesis and Processing Science

Division: Materials Sciences and Engineering
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Portfolio Description:

Synthesis and Processing Science addresses the fundamental understanding necessary to extend from design and synthesis to the preparation of materials with desired structure, properties, or behavior. This includes the assembly of atoms or molecules to form materials, the manipulation and control of the structure at all levels from the atomic to the macroscopic scale, and the development of processes to produce materials for specific applications. The goal of basic research in this area ranges from the creation of new materials and the improvement of the properties of known materials, to the understanding of such phenomena as adhesion, diffusion, crystal growth, sintering, and phase transition, and ultimately to the development of novel diagnostic, modeling and processing approaches. This activity also includes development of *in situ* measurement techniques and capabilities for quantitative characterizations to understand the growth processes at atomic or nanometer length scales.

Unique Aspects:

- The Materials Preparation Center (MPC) at the Ames Laboratory is operated for the purposes of understanding and further developing innovative and superior processes and for providing small quantities of unique, research-grade materials that are not otherwise available to academic, governmental, and industrial research communities.
- Through the Center of Excellence for the Synthesis and Processing of Advanced Materials (CSP), coordinated, collaborative research partnerships related to synthesis and processing of advanced materials are promoted between DOE national laboratories, universities, and the private sector.
- Advanced growth techniques and *in situ* diagnostics have been developed for the synthesis of improved thin-film structures of advanced materials.
- Non-equilibrium processing methods are studied using advanced modeling techniques and experimental methods, including innovative real-time, *in-situ* techniques enabled by synchrotron radiation.

Relationship to Others:

This program is intimately related to the other research activities in the Division of Materials Sciences and Engineering as the synthesis and processing of materials is a critically important area of materials research and development.

Through materials supplied by the Ames MPC, linkage is provided to other federal offices, the academic community, the industrial community and international research institutions. During FY2003, the MPC supplied a variety of specialized material products to 117 different clients (157 orders) at 92 academic, national and industrial laboratories worldwide. The MPC continued to provide Sn-Ti alloy to Oxford Superconducting Technology for the fabrication of Sn-Ti/Nb superconductors. Working with the Jet Propulsion Laboratory (JPL), MPC continued its effort related to the preparation of $LaNi_{4.8}Sn_{0.2}$ cryocooler metal hydride sorbent alloy for the Planck space vehicle. One amorphous metal alloy project was continued FY2003, in which aluminum based metal powders were prepared for the DARPA's Structural Amorphous Materials program in conjunction with Pratt & Whitney. An additional project was initiated in late FY 2003 with the U.S. Army's Aberdeen Proving Grounds on a hafnium based kinetic energy penetrator program. MPC's unique high pressure gas atomization system played a major role in its involvement in these programs.

Additional linkages within the Department of Energy are provided through the Energy Materials Coordinating Committee and its subcommittees. Interagency coordination is provided by participation in the MatTec Communications Group on Metals, the MatTec Communications Group on Structural Ceramics, and the National Nanotechnology Initiative.

Significant Accomplishments:

This program has changed the way people understand and think about the preparation of materials. Experimental, theoretical and computational tools are developed and applied to advance the scientific understanding of complicated thermodynamic and kinetic phenomena underlying processes ranging from self-assembly to far-from-equilibrium reactions that take place in welding. In the epitaxial growth area, a new technique has been developed to deposit ultrathin metallic layers on oxide, which will help next-generation computers boot up instantly by making entire memories immediately available for use. The thin metal layer achieves epitaxial crystallinity after the deposition of only a few atomic layers. This process should be applicable to a wide range of metals on metal oxides. Significant progresses have been made in the growth of single crystalline thin films of ferroelectric and ferromagnetic oxides using the molecular-beam epitaxy technique. New candidate ferromagnetic semiconductors have recently been grown by doping transition-metal oxides with magnetic impurities, which are nontraditional but strongly magnetic and thermally robust diluted magnetic semiconductors. Recent breakthroughs in the synthesis of complex oxides have brought the field to an entirely new level, in which complex artificial oxide structures can be realized with an atomic-level precision comparable to that well known for semiconductor heterostructures. Not only can the necessary high-quality ferroelectric films be now grown, but ferroelectrics can be combined with other functional oxides, such as high-temperature superconductors and magnetic oxides, to create multifunctional materials and devices. The shrinking of the relevant lengths to the nanoscale produces new physical phenomena.

New techniques, developed to measure local electromagnetic properties, now permit a fundamental understanding of the mechanism by which solid-solid interfaces and crystalline defects control the behavior of nanostructured as well as macroscopic materials. For the first-time, suppression in dielectric-constant has been observed directly at grain boundaries, contradicting traditional assumptions generally made about grain-boundary behavior, utilizing scanning impedance microscopy and nano-impedance spectroscopy. In the welding area, a coupled thermodynamic and kinetic model was developed to describe stability of the principal phases in stainless steels. This knowledge has led to the modification of the standard diagram used to choose welding electrode compositions for stainless steels. Additional modeling work utilizing massively parallel computers has permitted the linkage of macro- and microscopic scale phenomena during the melting and solidification of a weld. This permits simulation and visualization of weld microstructure as a function of processing conditions, e.g. during the melting, addition of new compounds, and resolidification that occurs during welding. Experimentally, tracking of real-time phase transformations that occur during weld solidification was made possible using synchrotron radiation and provided invaluable data to support scientific modeling and simulation leading to better electrode design. Recognitions include the recipients of the Spararagen Award and the Warren F. Savage Award from the American Welding Society.

Specific achievements include:

- Investigations of self-assembled heteroepitaxial semiconductor quantum dots using real-time stress sensing and light scattering showed how elastic repulsion determines evolution of dot arrays. Repulsion promotes spatial ordering, accelerates ripening kinetics, and enhances quantum dot phase transition.
- In the self-assembly area, developing scientific understanding of surfactant interactions with ceramic compounds and other materials, including biological tissues, has permitted the growth of ordered porous ceramic structures with hierarchical architecture spanning from the nano- to the macro-scale.
- A rapid, efficient self-assembly process for making nanophase composites that mimic the complex construction of seashells was developed resulting in a strong and tough (crack resistant on impact loading) material.
- Ceramic substrates were synthesized with tailored and regularly ordered nanoscale pores of controlled shapes and sizes. These substrates were found to remove deadly heavy metals such as mercury, lead and silver from contaminated water.
- A breakthrough in the fundamental understanding of the processing of ceramic aerogels led to a new, non-toxic, low temperature, and low-pressure process to produce films in an environmentally benign manner. This discovery overcame the sixty-year barrier to the large-scale commercial utilization of these films, won the prestigious Iler Award of the American Chemical Society and was cited as an important discovery by the Wall Street Journal.
- The Materials Preparation Center has completed over 3700 requests for specialized materials preparation and characterization services since its establishment. In addition to the previously mentioned accomplishments, MPC has enabled the following technologies:
    - ♦ Lead free solder
    - ♦ Magnetocaloric gadolinium-silicon-germanium alloys
    - ♦ Recyclable lightweight automotive composite materials

  - ♦ Terfenol-D which is a mangetostrictive alloy containing terbium, dysprosium, and iron that was developed at the Center and led to the spin-off of a new private sector company which now markets this material
- Quasicrystal coatings produced by plasma-arc spray that have superior wear resistance and thermal insulation behavior coupled with reduced surface friction for potential thermal barrier wear resistant coating applications in aircraft-engine components.
- A uniform three-dimensional coating process known as "Plasma Ion Immersion Processing" was improved so as to fabricate hard coatings, such as diamond-like carbon, that exhibit low sliding friction and superior wear resistance. This process is cost-effective and achieves a high rate of implantation over a very large surface area with uniform thickness and coating quality over complex three-dimensional geometries.
- A nanophase molecular template method was developed to synthesize films that exploit the dielectric properties of air to achieve ultra-low dielectric constants for the next generation of microelectronic devices and computers.
- Developed a unified, fundamental understanding of the multifaceted behavior of H in Mg-doped, p-type GaN that, in several respects, goes beyond what has previously been achieved for H in compound semiconductors. These studies represent a new level of quantitative understanding of hydrogen behavior within compound semiconductors.
- Investigation of the dynamics of self-assembled supramolecular organic oligomers, polymers, and metal chelate systems has revealed methods to control both intra- and intermolecular interactions leading to systems with tunable optical properties.

Mission Relevance:

This research supports the Department of Energy’s overarching goals for improved energy efficiency, protection of the environment, and the advancement of scientific knowledge. Specific relevant applications include discovery of new materials for efficient energy production and use; hard and wear resistant surfaces to reduce friction and wear; high-rate, superplastic forming of light-weight, metallic alloys for fuel efficient vehicles and other structures needed in land and air transportation applications; high-temperature structural ceramics and ceramic matrix composites for high-speed cutting tools, bearings, engines and turbines (to enable fuel efficiency and low-pollutant emissions); ordered intermetallic alloys for harsh applications (requiring heat, load, wear and corrosion resistance), including engines and turbines (also to enable fuel efficiency and low pollutant emissions); response of magnetic materials to applied static and cyclical stress; plasma, laser, and charged particle beam surface modification to increase corrosion and wear resistance; and welding and joining, including dissimilar and non-metallic materials.

Scientific Challenges:

Understanding the physics and chemistry of the synthesis and processing, as well as the thermodynamics and kinetics of reaction, of nanoscale materials and structures and the elements of the processing environment are critical to the preparation of larger components. There are significant experimental, theoretical and computational challenges in understanding what is occurring so that the benefits of nanoscale phenomena can be realized in larger scale components. Major scientific challenges also remain in the fabrication and the fundamental understanding in the non-trivial assemblies of inorganic, organic, composite, and biomimetic materials. There is a need for creative and innovative methods to investigate complex systems, such as composite materials with multifunctionality.

Future efforts are required to synthesize new materials for the advancement of science and technologies, to gain the fundamental understandings for better control of materials manipulation and properties, and to solve materials problems, such as adhesion and stability under thermal and environmental stress. Although there is steady progress in the synthesis and processing of materials, there still exists a serious deficit in the ability to produce (new) materials with desired properties and microstructures by rational design and synthesis. Experimental methods and theoretical models need to be developed to achieve mesoscopic structures via various methods, such as self-organized and directed growth. Scientific challenges also lie in new composite materials with various matrices, and in ecologically-benign materials.

Funding Summary:

Dollars in Thousands

FY 2003	FY 2004	FY 2005 Request
11,839	12,000	12,975

Performer	Funding Percentage
DOE Laboratories	60%
Universities	40%

These are percentages of the operating research expenditures in this area; they do not contain laboratory capital equipment, infrastructure, or other non-operating components.

Projected Evolution:

- Increased emphasis on understanding the opportunities and challenges presented by nanoscale materials and by the processing of larger components containing nanoscale materials and structures.
- Further progresses will be made in the growth and understanding of epitaxial heterostructures of ferromagnetic, ferroelectric and/or superconducting compounds using the molecular-beam epitaxy technique.
- Science-based understanding of advanced synthesis and processing methods such as self-assembly, molecular- and supramolecular-directed nanostructure formation, and novel deposition methods will be investigated. This understanding will be applied to attain new structures, compositions, and materials behavior, to fabricate materials with new functionalities, and to reduce the energy and environmental impact of processing.
- Processing research will be extended to include new ceramic, intermetallic, semiconducting, organic, composite, and biomimetic materials and material structures, including nanocrystalline materials, films, coatings, and crystals. Analytical techniques and modeling will be developed and applied to determine and predict the relationship of synthesis and processing parameters towards structure, purity, deformability, residual stresses, toughness, adhesion, and electronic, optical and magnetic properties.
- Processing capabilities of the Materials Preparation Center will be expanded to include intelligent process control and modeling and to provide new innovative synthesis and processing techniques. Plans include the use of a plasma-heated skull-melting furnace with capabilities to cast or atomize materials, and microstructure-sensitive processing to provide real-time monitoring and correlation with process models.
- An understanding of interfacial interactions at the molecular level will be developed, and their influence on molecular conformation and ordering will be shown for evolving nanostructures. An understanding of how short and long-range molecular forces mitigate the formation of ordered amorphous structures will be extended to develop an understanding of how biological systems control the formation of ordered assemblages of crystalline nanoparticles.