Research Activity: **Synthesis and Processing Science**

Division: Materials Sciences and Engineering

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Portfolio Description:

Synthesis and Processing Science addresses the understanding necessary to extend from synthesis to the preparation of materials with desired structure, properties, or behavior. This includes development of *in situ* measurement techniques and capabilities to quantitatively determine variations in the energetics and kinetics of growth and formation processes on small length scales. It also includes matters related to the shaping and forming of structural metals by deformation processes, the packing and consolidation of ceramic particulates, the preparation of thin films and semiconductor junctions with controlled elemental dopant profiles, coating and surface modification processes including but not limited to those involving charged particle beams plasmas and lasers, and the joining or welding of metals, ceramics, and/or dissimilar materials by plasma, electron beam, laser and other techniques. The Materials Preparation Center (MPC) at the Ames Laboratory is operated to further advance and capitalize on this understanding to fabricate research grade materials and provide these materials to the research community.

Unique Aspects:

- The Materials Preparation Center 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 (CESYNPRO), coordinated, collaborative research partnerships related to synthesis and processing of advanced materials are promoted between DOE national laboratories, universities, and the private sector.
- 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 refines, further develops, and applies analytical techniques from other BES programs to the needs and opportunities of this research activity. Projects evolve from this CRA to other CRAs, e.g., Physical Behavior of Materials and Mechanical Behavior and Radiation Effects as scientific questions evolve concerning the dependence of the physical and mechanical behavior of materials on the parameters of synthesis and processing. The structure and composition of materials is also supremely dependent upon the parameters of synthesis and processing, this activity is intimately linked with the Structure and Composition CRA.

Through materials supplied by the Ames MPC, linkage is provided to the Office of Biological and Environmental Research, the academic community, the industrial community and international research institutions. In FY 2000 the MPC provided materials to 15 users at DOE laboratories, 4 at other federal laboratories, 38 at universities, 119 at industrial laboratories, and 35 at international universities and institutions. Total orders totaled $602K in FY2000. The MPC is currently working on a $150K order for the preparation of 24kg of $LaNi_{(5-x)}Sn_{(x)}$. This is to assist the Jet Propulsion laboratory in fabricating a Joule-Thompson cryocooler for the joint ESA-NASA PLANCK Mission scheduled for 2007.

Additional linkages within the Department of Energy are provided through the Energy Materials Coordinating Committee and its subcommittees on superconductivity, semiconductors and photovoltaics, structural ceramics and metals. Interagency coordination is provided by participation in the MatTec Communications Group on Metals, the MatTec Communications Group on Structural Ceramics, the MatTec Communications Group on Nondestructive Evaluation, and the Interagency Working Group on Nanotechnology.

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 welding. 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 are made possible using synchrotron radiation and provide invaluable data to support scientific modeling and simulation and leads to better electrode design. The later work received the 2001 Spararagen Award from the American Welding Society. A current application in electrode design is the new understanding of the consequences of "self shielding"--that is preventing adverse reactions with air through additions of aluminum or other powders to the electrodes on compound (phase) stability leading to understanding of potential changes in electrode composition on properties of welded components. Recognitions include the two recipients of the Warren F. Savage Award from the American Welding Society

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 duplicating template structures from the nanoscale to the macro scale. Uses of these templating techniques to prepare ceramic duplicates of complex biological tissues are currently under study.

Specific achievements include:

- Investigation of the physics of multilayer structures and the ability to fabricate artificial semiconductor structures has led unexpectedly to the development of a biological microcavity laser in which a single human cell acts an internal component of the laser. This work was recognized with a 1997 IR&D Award.
- A rapid, efficient self-assembly process for making nanophase composites that mimic the complex construction of seashells was developed and it resulted in a strong and tough (crack resistant on impact loading) material.
- Ceramic substrates were synthesized with tailored and regularly ordered nanoscale pore sizes of controlled shapes and sizes. It was then found that these substrates would remove deadly heavy metals such as mercury, lead and silver from water. Recognition included a 1998 R&D Award. This work was co-funded with the DOE Office of Environmental Management.
- A breakthrough in the fundamental understanding of the processing of ceramic aerogel films led to a new, non-toxic, low temperature and low-pressure process to produce such 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.
- Established a Materials Preparation Center to provide outside researchers from academia, industry and government laboratories with research quality and quantities of unique, carefully controlled research-grade materials and crystals that would not otherwise be available. The following technologies were enabled by this Center:
    - 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
- Nanocrystalline neodymium-iron-boron magnet alloys with matching crystallite and magnetic domain sizes that won an IR&D 100 Award in 1997.
- 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 coating, such as diamond-like carbon, that exhibit low sliding friction and superior wear resistance. This process achieves a uniform implantation rate over a very large surface, a very high rate of implantation, and has the ability to produce a uniform thickness and quality coating over complex three-

dimensional geometries, and is cost-effective. This achievement was recognized with an R&D 100 Award in 1997.
- 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.

Mission Relevance:

This research supports the Department of Energy's overarching goals for improved energy efficiency and protection of the environment. Specific relevant applications include 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 fuel-efficient 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); non-destructive analysis for early warning of impending failure and on-line flaw detection and quality assurance during production; response of magnetic materials to applied static and cyclical stress; and 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 thermodynamics and kinetics of reaction of nanoscale powders and nanoscale structures with each other and the elements of the processing environment are critical to the preparation of larger components. There are significant computational and experimental challenges in understanding what is occurring so that the benefits of nanoscale phenomena can be realized in larger scale components. Derivative questions involving nanoscale powders include reversible passivation, steric interactions during consolidation, and inhibition of grain growth and recrystallization during processing.

While silicon-based structural ceramics are now being used in demanding applications such as turbocharger rotors, fuel ignitors, and high-performance bearings, commercial applications fully exploiting the high-temperature load-bearing capacity and creep resistance of high-temperature structural ceramics are yet to be realized. The scientific challenges include a plethora of fundamental questions concerning the structure and behavior of intergranular films (that are frequently as thin as one atomic layer), the understanding of the microstructures (particularly as influenced by processing), and the mechanisms of toughening (resistance to brittle fracture).

One challenge for non-destructive evaluation is in developing understanding that will enable quantitative and predictive methods that will permit applications such as early warning of impending crack formation, fracture or other failure. Another challenge is on-line *in-situ* diagnostics coupled to a process controller so as to provide absolute manufacturing quality assurance. The scientific challenges lie both in improving the fundamental and quantitative understanding of materials degradation phenomena, and in improving analytical methods to probe changes at scales from atomic to microstructural. Novel methods being explored involve the use of physical acoustics and positron annihilation.

Funding Summary:

Dollars in Thousands

FY 2000	FY 2001	FY2002
$13,820	$12,801	$14,781

Performer	Funding Percentage
DOE Laboratories	86.0%
Universities	14.0%

Projected Evolution:

- Increased emphasis on understanding the opportunities and challenges presented by nanoscale materials and by the processing of larger components containing nanoscale materials.
- Science based understanding of advanced synthesis and processing methods such as self-assembly, molecular-directed nanostructure formation, and novel deposition methods will be investigated. This understanding will be applied of to attain new structures and compositions, 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, and semiconductor materials and material structures, including nanocrystalline materials, films, coatings, and crystals. Analytical techniques and modeling will developed and applied to determine and predict the relationship of synthesis and processing parameters to structure, purity, deformability, residual stresses, toughness, adhesion, and electronic and magnetic properties.
- Welding process models will be refined and extended to solidification of aluminum alloys. The relationship of process conditions to microstructure development in various steel welds will be studied by real-time, *in-situ* x-ray diffraction enabled by synchrotron radiation. Follow-on work in supercooling effects in welding. Determination of residual stress distribution in welds and spatial distribution of different phases in different locations of the weld heat-affected-zone.
- Processing capabilities of the Materials Preparation Center will be expanded to provide 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. Follow on efforts in microstructure-sensitive processing and providing real-time monitoring of processing 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.

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