ORNL/TM-13595

Advanced Industrial Materials (AIM) Program
Office of Industrial Technologies
Energy Efficiency and Renewable Energy
U.S. Department of Energy (DOE)

Advanced Industrial Materials (AIM) Program

Compilation of Project Summaries and Significant Accomplishments

FY 1997

Date Published: May 1998

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Prepared by the Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831
for the U.S. Department of Energy
under contract number DE-AC05-96OR22464

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INTRODUCTION

Introduction to the Advanced Industrial Materials (AIM) Program
Office of Industrial Technologies
Fiscal Year 1997

C. A. Sorrell, Program Manager

The Advanced Industrial Materials (AIM) Program is a part of the Office of Industrial Technologies (OIT), Energy Efficiency and Renewable Energy, U.S. Department of Energy (DOE). The mission of AIM is to "support development and commercialization of new or improved materials to improve energy efficiency, productivity, product quality, and reduced waste in the major process industries." Program investigators in the DOE National Laboratories are working closely with approximately 100 companies, including 11 partners under Cooperative Research and Development Agreements. Research and development is being performed in a wide variety of materials technologies, including metallic and intermetallic alloys, ceramic and metal matrix composites, polymers, inorganic membrane materials, and coatings.

OIT has embarked on a fundamentally new way of working with industries—the Industries of the Future (IOF) strategy—concentrating on the major process industries that consume about 90% of the energy and generate about 90% of the waste in the industrial sector. These are the aluminum, chemical, forest products, glass, metalcasting, and steel industries. OIT has encouraged and assisted these industries in developing visions of what they will be like 20 or 30 years into the future, defining the drivers, technology needs, and barriers to realization of their visions. These visions provide a framework for development of technology roadmaps and implementation plans, some of which have been completed. OIT then provides cost shared support for research and development of needs identified in the roadmaps and is working with other government agencies to leverage that funding. Since the IOF strategy was undertaken, other industries that serve the six IOFs in a crosscutting way have completed or are working on visions and roadmaps. These include the forging, heat treating, welding, and carbon products industries. These industries are also invited to form partnerships with the IOFs and respond to solicitations for research and development proposals.

The AIM Program supports IOF by conducting research and development on materials to solve problems identified in the roadmaps. This is done by National Laboratory/industry/university teams with the facilities and expertise needed to develop new and improved materials. Each project in the AIM Program has active industrial participation and support.

Assessments of materials needs and opportunities in the process industries are an on-going effort within the program. These assessments are being used for program planning and priority setting, followed by support of work to satisfy those needs. All the industries have identified materials as critical, particularly for high-temperature strength, corrosion resistance, and wear resistance. Also important from the energy efficiency viewpoint are membranes, catalytic membranes, and reactors for separations, both for processing and waste reduction. AIM focuses, therefore, on high-temperature materials, corrosion resistant materials, wear resistant materials, strong polymers, coatings, and membrane materials for industrial applications.

Recently AIM provided funding to designate the Metals Processing Laboratory User Center (MPLUS) at Oak Ridge to solve materials problems for the IOFs. To date, more than 60 projects have been completed or are in progress, with about 20 more potential projects being planned. Industry and university participants are unanimous in their praise and appreciation for MPLUS. An additional benefit to industry is that access to MPLUS also provides access to the other user centers at Oak Ridge. When funding reaches a satisfactory level, the plan is to refer potential users to other National Laboratories, as appropriate, and to provide funds to those Laboratories to work with the users.

This year, FY 1998, two of the projects initiated and supported by AIM are being funded by the Chemical Industry Team. In addition, funding for three AIM projects has been designated for materials for the glass industry. These examples show clearly that project selection in the AIM Program are relevant to the Industries of the Future, and every effort will be made to select and fund projects with a high probability that they will eventually be taken over by the IOF teams in order to ready the technologies for demonstration in industrial environments.

The success of AIM can be best illustrated by a statement made by the Panel on Intermetallic Alloy Development, Committee on Industrial Technology Assessment, National Materials Advisory Board, in their report, *Intermetallic Alloy Development: a Program Evaluation, 1997*. Although the statement was made about only one part of AIM at Oak Ridge, a good case can be made for applying it to the entire Program and to the other participating Laboratories. The quote is: "Work by the ORNL Metals Processing Lab and their technical support of industrial product development have been very important. The AIM Program strategy, the IOF focus, MPLUS, and changes in licensing strategy have established a framework for developing technologies that can be commercially successful."

ADVANCED CERAMICS AND COMPOSITES

PROJECT SUMMARY

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: High Temperature Particle Filtration Technology

PHASE: FY 1997 **COMPLETION DATE:** July 2000

PERFORMING ORGANIZATION(S): Oak Ridge National Laboratory and Dow Corning Corp.

PRINCIPAL INVESTIGATOR(S): Theodore M. Besmann/ORNL, (423-574-6852) and Charles A. Hall/Dow Corning, (502-732-2520)

PHASE OBJECTIVE: The main objective of the current year's work was to perform bench scale testing of the compatibility of high temperature filters in the Dow Corning applications.

ULTIMATE OBJECTIVE: The ultimate objective is to demonstrate utilization of high temperature filtration in two Dow Corning applications related to dimethyldichlorosilane production.

TECHNICAL APPROACH: The approach of the program is to acquire filter specimens from manufacturers and perform bench-scale compatibility testing. After prioritizing the filter samples based on the testing, full-scale filters will used in a small process unit at Dow Corning as a proof of concept evaluation.

PROGRESS: During this period we completed modification of a furnace system for bench-scale testing of the filter samples. A large number of filter samples were acquired and they were all tested for 24 h under simulated conditions for one Dow Corning application.

Patents: -	**Publications: -**	**Proceedings: -**
Books: -	**Presentations: -**	**Awards: -**

ACCOMPLISHMENTS: Determined that a significant number of commercial filter concepts are likely to perform well in one of the Dow Corning application environments.

Technology Transfer or Industrial Interaction: Information is provided to Dow Corning regarding the stability of the filters in their environments.

PROJECT SUMMARY (continued)

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: High Temperature Particle Filtration Technology

CRITICAL ISSUES: An issue is the appropriateness of the bench-scale environmental tests. To be determined is whether another set of test which more completely models the chemical environment will be needed.

FUTURE PLANS: Continued testing at the bench-scale for 1000 h in the initial application environment and to start testing in Dow Corning's second environment.

POTENTIAL PAYOFF: The payoff will be the application of high temperature filtration to major process systems used by Dow Corning and their suppliers.

ESTIMATED ENERGY SAVINGS: Potentially 12 trillion Btu/y by the year 2010.

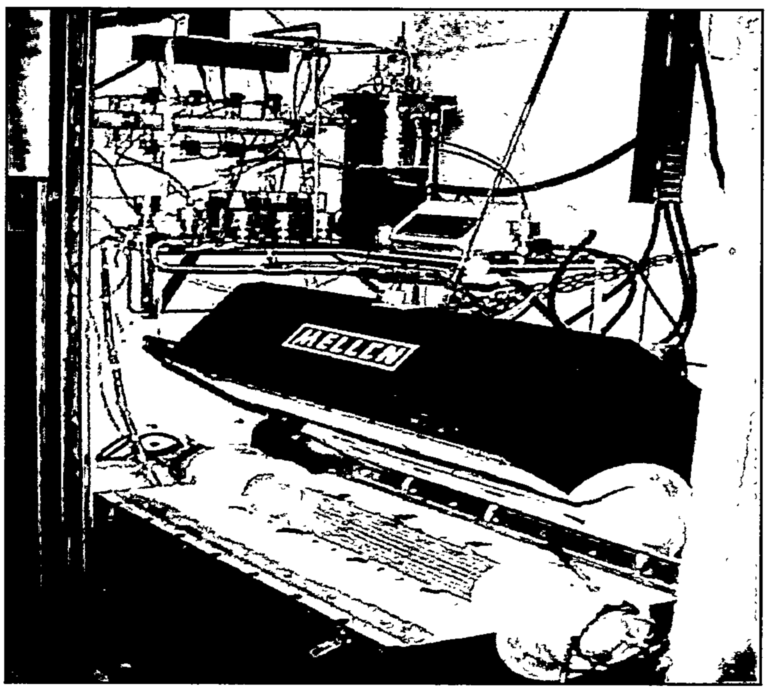
SIGNIFICANT ACCOMPLISHMENT

Advanced Industrial Materials (AIM) Program

High Temperature Particle Filtration Technology

ISSUE: Improve energy efficiency of a process for producing a silicone precursor feedstock, dimethyldichlorosilane.

RESULTS: Performed bench-scale testing of filter specimens for one of the process applications and demonstrated compatibility with most materials.



Bench-scale environmental compatibility test system for the Dow Corning dimethyldichlorosilane process.

ENERGY EFFICIENCY: Potentially 12 trillion Btu/y by the year 2010.

Research performed at the Oak Ridge National Laboratory, sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Industrial Materials Program, and Continuous Fiber Ceramic Composite Program; and Fossil Energy, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

PROJECT SUMMARY

Advanced Industrial Materials (AIM) Program

PROJECT SUMMARY: Intermetallic-Bonded Ceramic Composites

PHASE: FY 1997

PERFORMING ORGANIZATION: Oak Ridge National Laboratory

PRINCIPAL INVESTIGATOR: Paul F. Becher, (423-574-5157)

PHASE OBJECTIVE: The assessment of fabrication routes for intermetallic bonded carbides using nano-sized carbide powders and the characterization of resultant properties.

ULTIMATE OBJECTIVE: This program is designed to develop a range of wear resistant and structural composite materials that can be employed in applications requiring good corrosion resistance and the retention of high fracture toughness and strength at elevated temperatures (1000°C).

TECHNICAL APPROACH: The approach is based on the use of the ductile intermetallic Ni_3Al (or similar intermetallic alloys) as a binder phase for carbide or boride based composites for use in environments where corrosion and/or application temperatures are a problem. Industrially viable processing techniques for these new composite materials will also be developed.

PROGRESS:

Patents: -	**Publications: 3**	**Proceedings: 2**
Presentations: 1	**Invention Disclosures: -**	**Awards: -**

ACCOMPLISHMENTS: Recent efforts produced intermetallic-bonded carbides with submicron carbide grain size which is critical for wear and structural applications.

Industrial Interactions: Provided samples for evaluation to LTV Steel Company, Reynolds Metals, Cummins Engines, Kennametal, and Thixomat. A data book detailing the properties of various aluminide-bonded carbides and borides has been prepared and distributed.

PROJECT SUMMARY (Continued)

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Intermetallic-Bonded Ceramic Composites

CRITICAL ISSUES: None.

FUTURE PLANS: Continue assessing advanced composites of interest to industry.

POTENTIAL PAYOFF: Materials for use at elevated temperatures and in aggressive chemical environments offer increased productivity in metal forming/working and petrochemical industries.

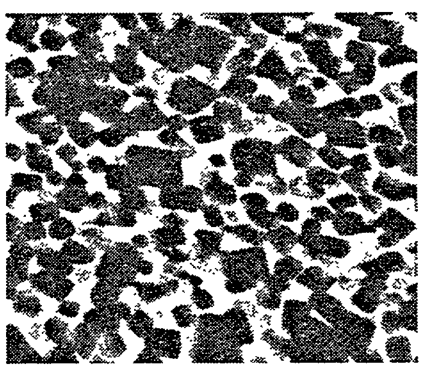
SIGNIFICANT ACCOMPLISHMENT

Advanced Industrial Materials (AIM) Program

Development Of Nano-Phase Intermetallic-Bonded Ceramic Composites

ISSUE: The tailoring of composites to develop high hardness and wear resistance requires the reduction of the grain size of the component phases. Nano-scale microstructural features are very advantageous not only to achieve high hardness and wear resistance but also to increase the fracture strengths. Further advances in the properties and applications of intermetallic-bonded carbides and borides could be substantially enhanced with the ability to fabricate composites with submicron grain sizes.

RESULTS: Recent processing developments utilizing TiC powders developed by Prof. R. Koc, Southern Illinois University, have produced fully dense Ni_3Al-bonded TiC composites with nano-sized grains. The average TiC grain size in this case is < 500 nm. Previous efforts with other powders only achieved TiC grain sizes of ~ 2 to 5 μm, an order of magnitude larger grain size. This represents a major advance in processing technology for these intermetallic-bonded systems. The reduction in microstructural scale achieved in the latest efforts clearly offers very exciting opportunities for overcoming material industrial needs related to high wear resistance and mechanical reliability.



Nano-size TiC (dark phase) grains in Ni_3Al binder phase.

Research performed at the Oak Ridge National Laboratory, sponsored by the U. S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Industrial Materials Program, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

PROJECT SUMMARY

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Membrane Systems for Energy Efficient Separation of Light Gases

PHASE: FY 1997

PERFORMING ORGANIZATION(S): Los Alamos National Laboratory

PRINCIPAL INVESTIGATOR(S): David J. Devlin, (505-667-9914)

PHASE OBJECTIVE: Development of a process based on vapor deposition techniques for the fabrication of membrane systems for light gas separation.

ULTIMATE OBJECTIVE: Development of a membrane system suitable for operation in a pilot scale system for the separation of C1 to C4 hydrocarbons from hydrogen.

TECHNICAL APPROACH: The technical approach involves the use of vapor deposition techniques to tailor pore size and shape in porous substrates. The pore properties will be engineered to effect capillary condensation of hydrocarbons near ambient temperatures and pressures less than 250 psig.

PROGRESS: A method for developing carbon pores for capillary condensation of hydrocarbons has been devised. Experiments demonstrate the feasibility of oblique angle vapor deposition as a means producing the desired pore structure. Separations of hydrocarbon gas mixtures with these membranes have been demonstrated.

ACCOMPLISHMENTS:

Licenses: none
Known Follow-on Products(s): none
Industry Workshop: none

Technology Transfer or Industrial Interaction: This effort will continue in '98 as a joint research effort with Amoco's Olefins R&D group. Amoco will develop characterization capabilities and design criteria for the membrane systems. With their guidance we will develop the materials and processing for the fabrication of these of membranes. Amoco's goal is a materials system capable of scaling for use in a pilot plant system.

PROJECT SUMMARY (Continued)

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Membrane Systems for Energy Efficient Separation of Light Gases

CRITICAL ISSUES: The precise control of pore structure in membrane materials.

FUTURE PLANS: Development of materials by vapor deposition techniques with engineered pore size and shape. Characterization and performance testing of developed materials by Amoco Olefins R&D.

POTENTIAL PAYOFF: There are important benefits that will result from the success of the proposed CRADA effort. The US. hydrocarbon industry has experienced a loss in competitiveness over the last decade that has resulted in the loss of nearly 500,000 jobs and has severely affected the US. balance of trade. Improvements in hydrocarbon-light gas separation processes represent the largest area for potential cost reductions for the entire hydrocarbon industry that includes natural gas processing, oil refining, and petrochemicals. For example, new grass-roots olefins units cost upward of $750 million; the separation section accounts for approximately 75% of this capital investment. Because of the high capital costs and market competitiveness, US. olefins producers have relied on de bottlenecking of existing units to meet capacity increases required to maintain market share. Shortly, these activities will not be feasible because of compressor train capacity limitations. Non-cryogenic breakthrough technologies for separating light gas byproducts from olefinic mixtures could lead to inexpensive capacity increases of 50%; this would significantly reduce manufacturing costs and promote industry growth. Also, the development of energy-efficient gas separation processes will have a positive impact on the environment. Lower energy consumption translates into less fuel burning to generate power resulting in a significant reduction in flue gas pollutants such as hazardous nitrogen and carbon oxides. Reducing pollutants is critical in the major industrial regions that have become non-attainment areas based on the National Ambient Air Quality Standards. Growth and job creation in these regions is dependent on reducing these emissions. Lower energy consumption would create the opportunity to expand and still meet the environmental guidelines while lessening the US. dependence on energy imports.

ESTIMATED ENERGY SAVINGS: Initial economic analyses have shown that the commercialization of this novel separation concept could result in an energy reduction potential of 5 trillion BTUs per year for an olefins complex: this corresponds to a potential annual savings of nearly $8 million.

SIGNIFICANT ACCOMPLISHMENT

Advanced Industrial Materials (AIM) Program

Membrane Systems For Energy Efficient Separation Of Light Gases

ISSUE: Ethylene and propylene are two of the largest commodity chemicals in the US. and are major building blocks for the petrochemicals industry. These olefins are separated currently by cryogenic distillation which demands extremely low temperatures and high pressures. Over 75 billion pounds of ethylene and propylene are distilled annually in the US. at an estimated energy requirement of 400 trillion BTU's. Non-domestic olefin producers are rapidly constructing state-of-the-art plants. These energy-efficient plants are competing with an aging US. olefins industry in which 75% of the olefins producers are practicing technology that is over twenty years old. New separation opportunities are therefore needed to continually reduce energy consumption and remain competitive.

RESULTS: We have shown that oblique angle vapor deposition techniques can be used to produce thin films with pores in the desired range of 4-5 nm. Amoco has tested these materials and demonstrated the separation of butane gases from multicomponent mixtures. Amoco has built and commissioned a capillary condensation unit for further evaluation of these materials.

ENERGY EFFICIENCY: Initial economic analyses have shown that the commercialization of this novel separation concept could result in an energy reduction potential of 5 trillion BTUs per year for an olefins complex: this corresponds to a potential annual savings of nearly $8 million.

Research performed at the Los Alamos National Laboratory, sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Industrial Materials Program under contract to the Los Alamos National Laboratory.

PROJECT SUMMARY

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: New Method for Synthesis of Metal Carbides, Nitrides and Carbonitrides

PHASE: FY1997

PERFORMING ORGANIZATION: Southern Illinois University

PRINCIPAL INVESTIGATOR(S): Rasit Koc, (618-453-7005)

PHASE OBJECTIVE: To promote the transfer of the technology developed under AIM program. The technology deals with a new process for producing high purity, submicron, nonagglomerated titanium carbide (TiC) powders.

ULTIMATE OBJECTIVE: To develop a novel synthesis method using a carbothermic reduction reaction of carbon coated precursors for producing high purity, submicron, non-agglomerated powders of metal-carbide, metal-nitride and metal-boride systems. We also want to demonstrate the advantages of the process and provide information on the applicability of the process for synthesizing related advanced ceramic powders (e.g. SiC, WC, TiN, TiB_2).

TECHNICAL APPROACH: The process developed in this project utilizes a carbothermic reduction reaction of novel coated precursors that has potential as a low cost powder synthesis route. It minimizes kinetic barriers by improving the way carbon is introduced to the reactants. The process consists of two steps. The first step is the coating of interested metal containing powders with carbon by decomposing a hydrocarbon gas at temperatures of 400°- 600°C. The second step involves the formation of metal-carbide, metal-nitride and metal-boride powders by promoting the carbothermal reduction of the carbon-coated metal containing particles in an inert atmosphere at temperatures of 1200°-1600°C. This way of increasing contact area between reactants results in a more complete reaction and a purer product at comparatively lower temperatures. The complete separation of the metal containing (oxides) particles by coated carbon and the low temperature processing results in products with less particle agglomeration and uniform particle size.

PROJECT SUMMARY (Continued)

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: New Method for Synthesis of Metal Carbides, Nitrides and Carbonitrides

PROGRESS: The carbon coating method developed for production of submicron TiC powders during 1995 was further improved, and its application extended to the synthesis of tungsten (W), tungsten monocarbide (WC), and titanium diboride (TiB_2) powders.

Patent : U.S. 5,417,952	Publications: 4	Proceedings: 1
Books: 0	Presentations: 6	Awards: 2

ACCOMPLISHMENTS:
Awards: R. Koc - Recipient of 1998 Kaplan Research Award
Technology Transfer or Industrial Interaction
ART Inc. has licensed the SiC patent to produce SiC powders.
Greenleaf Inc. is in process of licensing TiC patent.
Kennametal Inc. and Fansteel Inc. are in process of licensing WC technology.

CRITICAL ISSUES: Applicability of the process to synthesize other metal carbides, nitrides and borides. Production of metal carbides, metal nitrides and metal borides with desired properties.

FUTURE PLANS: Continue TiC and WC powder technology transfer by providing information on the process. Clearly demonstrate the advantages of the process over the conventional process. It is our intention to build upon the success that has been obtained in the production of high quality, low cost advanced ceramic TiC, WC and TiB_2 powders by extending the application of the process in related carbides, nitrides and borides.

POTENTIAL PAYOFF: Acquisition of a new process for nonoxide advanced ceramic powders that will provide a supply of low-cost, high quality (high purity, fine particle size) powders. The low cost powders will have a major impact on the cost of finished products that may expand current markets.

ESTIMATED ENERGY SAVING: This technology will improve energy efficiency because it does not require high reaction temperatures for production of these powders.

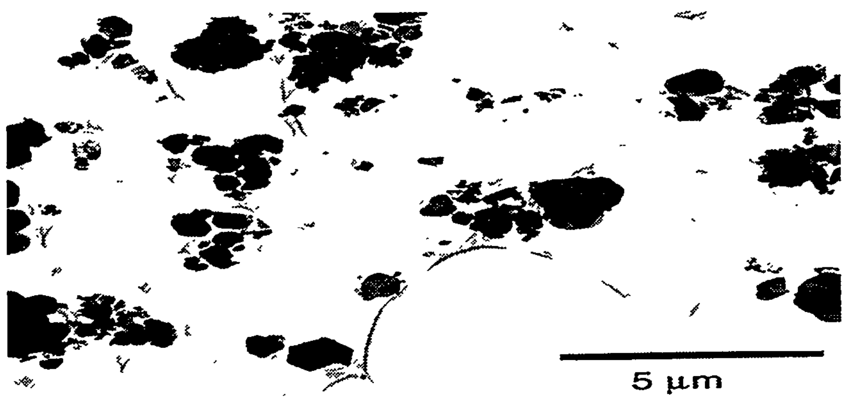
SIGNIFICANT ACCOMPLISHMENT

Advanced Industrial Materials (AIM) Program

New Method For Synthesis Of Metal Carbides, Nitrides And Carbonitrides

ISSUE: A novel synthesis process, based on a carbothermal reduction of metal oxides developed for producing TiC and WC powders was applied to produce high quality (submicron particle size, high purity) low cost titanium diboride (TiB_2) powders. After evaluating our TiC samples produced using this process, 3M Ceramic Technology Center approached Southern Illinois University to discuss the applicability of the process to synthesize submicron high purity TiB_2 powders and we are cooperatively investigating the potential of a new TiB_2 production process

RESULTS: We have succeeded in producing submicron TiB_2 with high purity. Using the produced submicron TiB_2 in specific application at 3M will provide products with superior properties. In aluminum metal industries, use of superior TiB_2 cathode will increase the competitive advantage of U.S. producer over major foreign producer in countries where electricity is much cheaper.



TEM micrograph of TiB_2 produced using the new process.

ENERGY EFFICIENCY: This technology will improve energy efficiency because it does not require high reaction temperatures for production of these powders. Using TiB_2 cathodes is proven for significant energy savings in the production of primary aluminum.

Research performed at Southern Illinois University, sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Industrial Materials Program, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

PROJECT SUMMARY

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Process Simulation For Advanced Ceramics Production. (1) Development of Advanced Energy-Efficient Coatings for Sun-Belt Low-E Applications (2) Process Simulation for Advanced Composites Production.

PHASE: FY 1997

PERFORMING ORGANIZATION: Sandia National Laboratories, Livermore, California

PRINCIPAL INVESTIGATOR: Mark D. Allendorf, (510-294-2895)

PHASE OBJECTIVE: To develop predictive computational models, process-control algorithms, and sensors for designing, optimizing, and operating coating processes for depositing ceramic coatings on materials such as continuous fiber preforms and float glass.

ULTIMATE OBJECTIVE: To improve the competitiveness of the US float-glass industry by developing new, cost-effective uses of glass. In addition, this project strives to reduce the costs of ceramic composites so that they can be applied more widely to corrosion, wear, and high-temperature problems in industrial settings.

TECHNICAL APPROACH: Industrial methods for the production of ceramic coatings are simulated using a high-temperature, variable-pressure flow reactor constructed as part of this project. The reactor provides an environment in which process parameters including temperature, pressure, chemical composition, and reactant mass flow rates are tightly controlled. In addition, access for mass spectrometric sampling and laser diagnostics is provided. Parallel development of computational models of physical and chemical mechanisms of deposition is also conducted.

PROGRESS:

- Developed a database of thermodynamic properties, and experimental and analytical techniques for probing high-temperature reactions of glass-coating precursors; and measured kinetics occurring in float-glass coating operations.
- Completed an analytical model to predict deposition of coatings on ceramic-fiber preforms and optimized model to quantitatively predict boron nitride coating rates in reactors used by DuPont Lanxide Composites (DLC; Newark, DE)

PROJECT SUMMARY (Continued)

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Process Simulation For Advanced Ceramics Production

Patents: none **Publications:** 8 **Presentations:** 8

ACCOMPLISHMENTS:

Technology Transfer: A three-year, $1.06 M CRADA with Libbey-Owens-Ford Co. (LOF) of Toledo, OH, began Oct. 1, 1996 (including $540 K of direct and in-kind funding from LOF). DLC provided access to its database of processing information to assist with the testing and validation of Sandia's fiber-coating model.

Licenses: none.

CRITICAL ISSUES: In the production of float glass, increasing the energy efficiency of fenestration through high performance on-line coatings is an important technological objective not only for glass producers, but also for window fabricators, architects, HVAC engineers, automobile manufacturers, and utilities. The costs of ceramic composite materials are currently too high to permit widespread use in industrial settings. One way to reduce these costs is to reduce the time required to develop, optimize, and scale up new processes for producing ceramic composites.

FUTURE PLANS: In the float-glass coatings portion of this project, we will continue to investigate the high-temperature reactions of indium-containing precursors and develop models to simulate deposition chemistry occurring in coating reactors. Diagnostic methods for on-line monitoring of coating reactors will also be explored. In the ceramic-composites area, efforts will be directed toward delivering a PC version of the model developed in FY97 for direct use by DLC process engineers.

POTENTIAL PAYOFF: Use of experimental data and computational models developed here are expected to shorten the time required to design, optimize, and scale up new coating processes and to facilitate extension of existing technology to new materials.

ESTIMATED ENERGY SAVINGS: New coatings on float glass developed in this project will save 1.4×10^{19} Btu/year when installed in place of clear glass windows. Energy benefits associated with widespread use of CFCCs are large, including up to 0.52 Quads/year in gas turbines, and 0.5 Quads/year in high-pressure heat exchangers.

SIGNIFICANT ACCOMPLISHMENT

Advanced Industrial Materials (AIM) Program

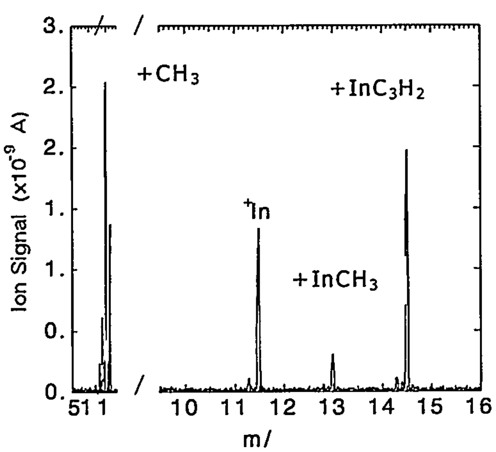
New Data Clarify Chemistry of Float-Glass Coating Processes

ISSUE: The complexity of industrial processes for depositing coatings on float glass, coupled with the costs associated with their development, make it difficult to make more than incremental improvements in coating effectiveness and process efficiency.

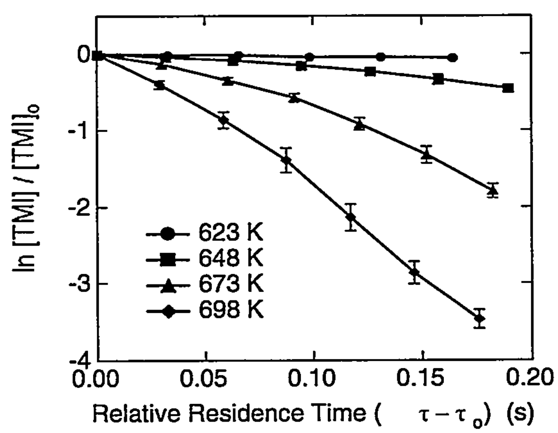
RESULTS: Experiments in a high-temperature flow reactor are being used to characterize chemical reactions occurring in chemical vapor deposition processes used to make indium tin oxide (ITO) films. A database of thermodynamic and kinetic information for indium-containing compounds is also being developed.

SIGNIFICANCE - FOR ENERGY CONSERVATION: In North America, 2.6 Million tons/year of float glass are used in residential and commercial construction. Use of so-called "low-E" (for "low emissivity") coatings on this glass can dramatically improve the energy efficiency of this material. New coatings developed in this project will save $1.4x10^{19}$ Btu/year when installed in place of clear glass windows.

SIGNIFICANCE - FOR MATERIALS TECHNOLOGY: Coatings on float glass hold the potential for making "smart" windows that can respond to changes in temperature or light level, flat-panel displays for computers and home electronics, and optical memory devices.



Mass spectrum of trimethlyindium, an ITO precursor, used to monitor its decomposition under coating conditions.



Decay of trimethylindium concentration as a function of residence time in a heated flow reactor. Analysis of these data yields the rates of chemical reactions occurring during the float-glass coating process.

PROJECT SUMMARY

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Synthesis and Processing of Composites by Reactive Metal Penetration

PHASE: FY 1997 **COMPLETION DATE:**

PERFORMING ORGANIZATION(S): Lead Organization: Sandia National Laboratories(SNL) Subcontractors: University of New Mexico (UNM), New Mexico Institute of Mining and Technology (NMIMT), and SRI International (SRI).

PRINCIPAL INVESTIGATOR(S): Ronald E. Loehman/SNL, (505-272-7601), Kevin G. Ewsuk/SNL, (505-272-7620); Subcontractor PIs: William G. Fahrenholtz/UNM, (505-272-7626); Ping Lu/NMIMT (505-835-5731);: Sylvia M. Johnson/SRI (415-859-4277).

PHASE OBJECTIVE: To identify compositions favorable for making composites and coatings by in situ reactive methods and to understand the mechanism(s) by which they are formed.

ULTIMATE OBJECTIVE: To learn to make high-quality composites and composite coatings economically, and to apply the results to problems of interest to industry.

TECHNICAL APPROACH: Composites are made by reacting molten metals with ceramics under controlled conditions. Coatings are made from organometallic polymers filled with ceramic and metal powders that are converted to adherent films. Mechanistic process models are developed from kinetic, microstructural, and phase diagram data combined with thermodynamic calculations. We use those results to make test specimens for determining physical properties.

PROGRESS: Using data from recent TEM studies at NM Tech, we developed a detailed mechanistic model for composite formation that explains the observed microstructure and kinetics as a function of time and temperature. This new model has led to a process diagram showing the conditions where reactive metal penetration (RMP) is a practical process. We optimized RMP processing to make composites in the Al_2O_3-$MoSi_2$, Al_2O_3-$Mo(Si_{0.93}Al_{1.43})$, and Al_2O_3-$Mo(Si_{0.93}Al_{1.43})$-Mo_3Al_8 families, and then evaluated their properties, which showed them to have excellent strength and stiffness at room temperature: coated Al and steel coupons exhibited excellent corrosion resistance in 1000 hr. salt spray tests.

PROJECT SUMMARY (continued)
Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Synthesis and Processing of Composites by Reactive Metal Penetration

Patents: -	**Publications: 3**	**Proceedings: 2**	**Licenses: -**
Books: -	**Master's Theses:**	**Presentations: 11**	**Awards: -**

Technology Transfer or Industrial Interaction: Development of a CRADA among Sandia, A.P. Green, and an aluminum industry partner has been delayed by a company reorganization that required seeking another Al partner. Those discussions are underway. SRI has made presentations on their composite coatings results to Sherwin Williams, AISI, Loctite, Honeywell, ACC, Conversion Technology, and Azure Capital Corp.

CRITICAL ISSUES: Reliability and cost are critical issues for any new material. Learning how to control wetting and reactivity of ceramic preforms by molten metals is critical to developing reliable materials. Understanding reaction mechanisms and rate-limiting processes are necessary for development of a wider range of composites, such as those that contain $MoSi_2$ formed by in-situ reaction.

FUTURE PLANS: Our recently developed model for RMP formation will be applied to in situ formation of a broader range of reinforcing composite phases, both to test the model and to make other useful composites. Because much of what we are learning about molten metal wetting is applicable to refractory performance in aluminum reprocessing, we are pursuing cooperative arrangements with one or more aluminum and refractory companies.

POTENTIAL PAYOFF: Composites made by RMP could replace cast iron for many applications with significant savings due to their lower density. Improved refractories for aluminum processing would lead to large increases in process efficiency. We are learning that reactive metal penetration could be an economical process for manufacturing many of the advanced ceramic composites that are needed for light-weight structural and wear applications. Near-net-shape fabrication of parts has the additional advantage that costly and energy intensive grinding and machining operations are significantly reduced, and the waste generated from such finishing operations is minimized.

SIGNIFICANT ACCOMPLISHMENT

Advanced Industrial Materials (AIM) Program

Newly-Developed Reaction Mechanism Explains Rapid Kinetics for Formation of Ceramic-Metal Composites

ISSUE: Composite formation by Reactive Metal Penetration (RMP) exhibits rapid, linear kinetics between about 1000 and 1150°C. The rates are fast enough to be commercially attractive, but without a mechanistic understanding, process control will be by trial and error.

RESULTS: Kinetic studies of the reaction $8Al + 3Al_6Si_2O_{13} \rightarrow 13Al_2O_3 + 6Si$ showed that composite formation is fast and linear with time between 1000 and 1150°C, but above 1150°C the reaction stops after a brief period. This suggests different rate limiting mechanisms above and below 1150°C. The activation energy for forming Si by Reaction 1 is much greater than that for Si diffusion in Al. TEM micrographs (Figure 1) show increasing amounts of Si at the reaction interface in the temperature range where the kinetics are slow, suggesting that Si buildup inhibits composite formation. Diffusion calculations show that Si production is much faster than its transport above 1100°C, causing Si to build up near the reaction front, shutting down the reaction. Experimental results and modeling has enabled the construction of a process diagram showing where the RMP reaction between aluminum and mullite has favorable rates (Figure 2).

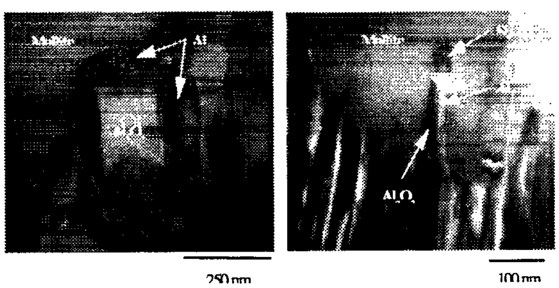


Figure 1. TEM micrographs of the aluminum-mullite reaction interface showing no Si at a) 900°C for 10 minutes, and presence of Si at (b) 1100°C for 10 minutes.

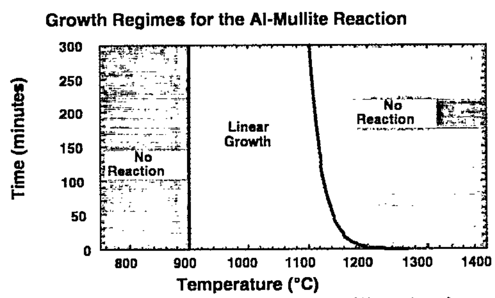


Figure 2. RMP processing map detailing the time-temperature response of the Al-mullite reaction.

ENERGY EFFICIENCY: Finishing and machining operations can represent up to 90% of the cost of making advanced ceramics and composites. Net shape forming offers the prospect of significantly reducing or even eliminating many finishing and grinding operations. Materials with improved strength and toughness at high temperatures will also allow higher processing temperatures resulting in higher energy efficiency.

Research sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Industrial Materials Program, under contract DE-AC04-94AL85000 with Sandia National Laboratories.

ADVANCED INTERMETALLICS/METALS AND COMPOSITES

PROJECT SUMMARY

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Advanced Ordered Intermetallic Alloy Development

PHASE: FY 1997

PERFORMING ORGANIZATION: Oak Ridge National Laboratory

PRINCIPLE INVESTIGATORS: C. T. Liu, (423-574-4459) and P. J. Maziasz (423-574-5082)

PHASE OBJECTIVE: To improve the tensile ductility and fracture toughness at ambient temperatures and the strength and creep and oxidation resistances at high temperatures of structural intermetallics based on TiAl, resulting in developing new light-weight structural materials for industrial use.

ULTIMATE OBJECTIVE: (1) To develop low-density, high-strength, ductile ordered intermetallic alloys for structural applications in advanced energy conversion systems and heat engines, and (2) to help U.S. material industries to compete in the world market.

TECHNICAL APPROACH: Both macroalloying and microalloying processes are employed to improve metallurgical and mechanical properties of ordered intermetallic alloys. The selection of alloying additions will be based on physical metallurgy principles, structural maps and some empirical correlations. Emphasis will be placed on improving ductility and toughness at ambient temperatures and strength and creep and oxidation resistances at elevated temperatures through material processing and structural control.

PROGRESS: Grain size has been identified as the key microstructural parameter controlling the room temperature tensile properties of TiAl alloys with lamellar structures. The Hall-Petch analysis shows a good linear relation between tensile elongation and grain size. The mechanical properties of the TiAl alloys with ultra-fine lamellar structure in cast and hot-extruded conditions are much superior to advanced TiAl alloys developed recently.

Invention Disclosures: 1 **Publications:** 3 **Proceedings:** 4
Books: 2 **Presentations:** 4

PROJECT SUMMARY (Continued)

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Advanced Ordered Intermetallic Alloy Development

ACCOMPLISHMENTS:

Licenses: Discussions with several U.S. industries for potential licenses
Known Follow-on Product(s): Anticipated use in various industries.

OTHER SUCCESSFUL TECHNOLOGY TRANSFER ACTIVITIES AS EVIDENCE OF INDUSTRY INTEREST: Philip Morris is very interested in the TiAl alloys development sponsored by AIM, and the company will provide new funding for ORNL in 1998 for further developing of TiAl alloys for industrial applications. Several companies have signed non-disclosure agreements with LMER for structural use of new high-temperature Ni_3Al alloys developed recently.

CRITICAL ISSUES: (1) To improve the mechanical properties and weldability of intermetallic alloys in cast conditions and (2) to develop Ni_3Si-base alloys with optimum properties for chemical industry applications.

FUTURE PLANS: (1) To increase the tensile ductility of cast intermetallic alloys and to improve fracture resistance of these alloys by control of microstructure and alloy compositions, (2) to control microstructure and alloy composition in order to develop Ni_3Si alloys with optimum mechanical and corrosion properties.

POTENTIAL PAYOFF: There are two major problems with many intermetallic alloys: poor room-temperature ductility and inadequate high-temperature creep resistance. If these problems are overcome, it will result in the development of a whole new class of ordered intermetallic alloys that have high-temperature properties that are superior to those of existing superalloys. The use of strong intermetallic alloys with light-weight would improve thermal efficiency and system performance of advanced engines and energy conversion systems, resulting in substantial energy savings.

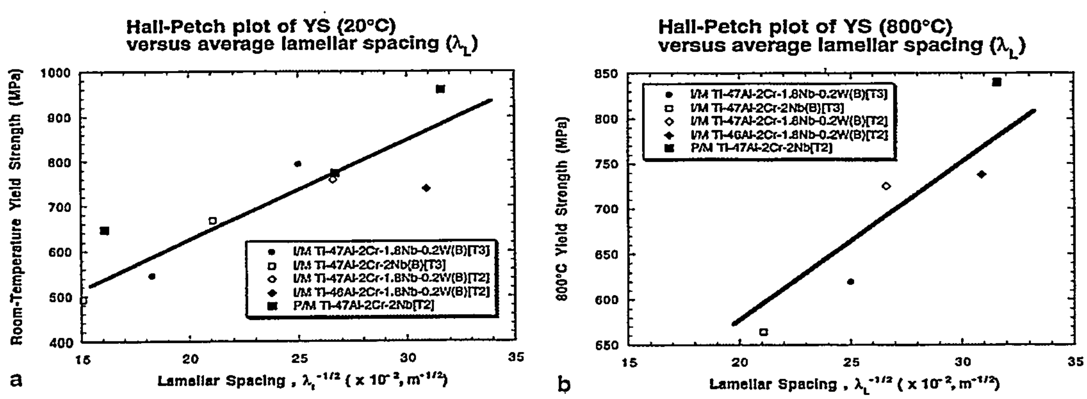
SIGNIFICANT ACCOMPLISHMENT

Advanced Industrial Materials (AIM) Program

Key Metallurgical Parameters Have Been Identified for Improving Mechanical Properties of TiAl Alloys

ISSUE: Two-phase TiAl alloys offer an attractive mix of low density (~4 g/cm^3), low thermal expansion, high melting point, and decent oxidation resistance at elevated temperatures. The major concerns for structural applications are limited ambient-temperature ductility and fracture resistance, and high-temperature strength and creep resistance. The current development effort has been focused on mechanical property improvement by control of microstructure and alloy composition in both cast and fabricated conditions.

RESULTS: Both interlamellar spacing and grain size are identified as the key parameters in controlling the mechanical properties of two-phase TiAl alloys. The tensile elongation ~5% at room temperature is obtained for TiAl alloys with ~ 40 μm grain size. The yield strength at levels of 1000 MPa at room temperature and 850 MPa at 800°C is achieved by reducing the interlamellar spacing to ~0.1 μm (see Figure). U.S. industries are very interested in TiAl and Ni_3Al alloys developed under the AIM Program, and several new non-disclosure agreements have been signed with U.S. industries.



The dependence of yield strength at (a) room temperature and (b) 800°C on interlamellar spacing, λ_l, for TiAl alloys.

SIGNIFICANCE - FOR ENERGY EFFICIENCY: The development of light-weight, high-strength TiAl and Ni_3Al alloys for high-temperature applications are expected to substantially improve the performance of many industrial systems as well as various energy conversion devices. All of these will result in a substantial energy savings.

PROJECT SUMMARY

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Development of Weldable, Corrosion-Resistant Iron-Aluminide (FeAl) Alloys

PHASE: FY 1997 **COMPLETION DATE:**

PERFORMING ORGANIZATION(S): Oak Ridge National Laboratory (ORNL)

PRINCIPAL INVESTIGATOR(S): P. J. Maziasz, (423-574-5082), and G. M. Goodwin (423-574-4809)

PHASE OBJECTIVE: Develop corrosion-resistance cast FeAl compositions with optimum mechanical properties that are also weldable. Determine the effects of metallurgical variables on mechanical properties and weldability. Develop both monolithic and weld-overlay cladding technology for FeAl.

ULTIMATE OBJECTIVE: Develop "super" corrosion-resistant FeAl alloys that can be used to replace conventional heat/corrosion-resistant steels, stainless steels and Fe-Cr-Ni alloys. Produce FeAl material or components for industrial testing. Define applications for FeAl as monolithic material or weld-overlay cladding and scale-up industry production.

TECHNICAL APPROACH: Micro-alloying additions of B, C, and Zr enhance ductility and cause more ductile fracture at room-temperature. ZrC or other fine precipitates produce high-temperature strength. Control of processing and heat-treatment conditions produces microstructures that give the best combination of room-temperature and high-temperature mechanical behavior. Good weldability and cold-cracking resistance relate to improved room-temperature ductility.

PROGRESS: Industrial corrosion testing demonstrates super-corrosion resistance of FeAl cast alloys for sulfidizing (816°C), carburization and oxidation (1000-1100°C). New cast FeAl alloys have up to 5% ductility and 14-20 J or more Charpy impact toughness in air at room-temperature, together with yield-strength of 400 Mpa at 700°C. Surprisingly, these FeAl alloys show strength comparable to cast heat-resistant austenitic alloys like HU and HK at temperatures of 1000-1100°C. Alloy development for improved high-temperature strength and creep-resistance continues. Processing that produces 10-15% ductility in air at room-temperature eliminates cold-cracking, and work to extend such behavior to cast alloys is in progress.

PROJECT SUMMARY (Continued)

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Development of Weldable, Corrosion-Resistant Iron-Aluminide (FeAl) Alloys

Patents: 2	**Publications:** 5	**Proceedings:** 9
Books: 1	**Presentations:** 13	**Awards:** 2

ACCOMPLISHMENTS: Results of tests performed at FMC, Inc. show cast grate-bars have excellent corrosion resistance in calcination of phosphate ores, and service for over 6 months at 1000°C is excellent.

Licenses: 1

Known follow-on Product(s): burner nozzles, radiant heating-tubes, tubes or pipes in chemical or petrochemical industry, steel rolls (molten zinc)

Technology Transfer or Industrial Interaction: FeAl materials are in-test for 8 different industrial applications. Duraloy has expressed interest in technology transfer for centrifugally-cast applications and INCO is interested in cladding applications, including weld-overlay.

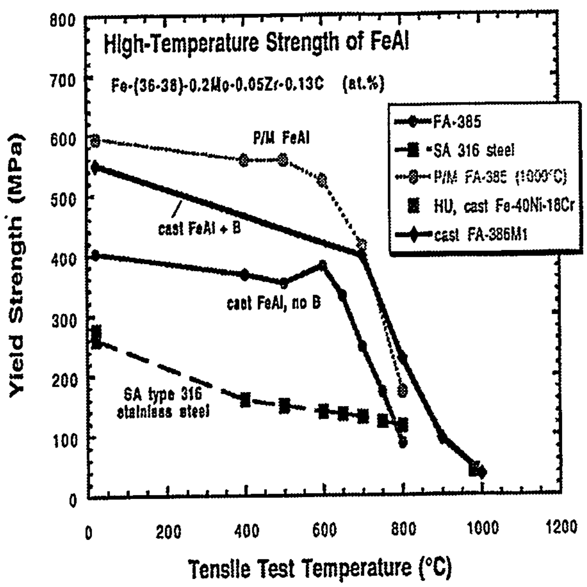
SIGNIFICANT ACCOMPLISHMENT

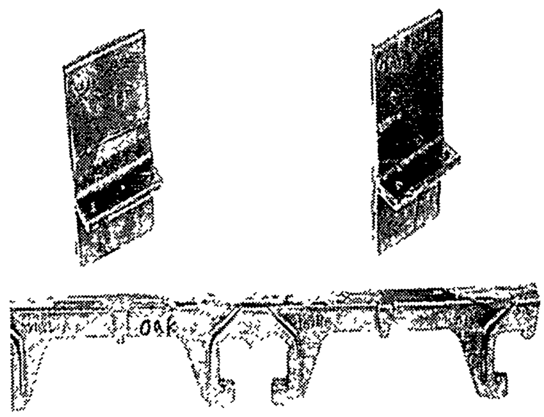
Advanced Industrial Materials (AIM) Program

Cast FeAl Alloys Show Strength and Sulfidation/Oxidation Resistance at 1000°C

ISSUE: FeAl iron-aluminides have outstanding oxidation, sulfidation, and even carburization resistance at 1000°C and above due to the formation of Al_2O_3 scale. However, the room temperature strength, ductility, and toughness need to be improved.

RESULTS: New cast FeAl alloys (Fe - 36-38 at.% Al) have strength at 900-1000°C comparable to that of typical type 310 austentic stainless steels or HU and HK cast austenitic stainless alloys. The new FeAl alloys were also found to have 4-5% ductility in air at room-temperature, and about 15 J Charpy impact toughness. Grate-bars and pallet tips for application as the conveyor belts that transport phosphate ores into and out of calcining furnaces are being tested in that severe oxidizing and sulfidizing environment, with pallet tips operating at over 1300°C and grate-bars at about 1000°C. Exposure for several months indicates some attack of the pallet tips and no evidence of attack for the grate-bars.





Pallet tips and grate bar

ENERGY EFFICIENCY: FeAl iron-aluminides can be melted by the Exo-Melt™ process, which saves 50% of the energy used by conventional melting. Longer lifetime and lower metal wastage of "super" corrosion-resistant FeAl in aggressive high-temperature environments will save the energy in materials processing operations.

PROJECT SUMMARY

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Development of Materials for Black Liquor Recovery Boilers

PHASE: FY 1997 **COMPLETION DATE:**

PERFORMING ORGANIZATION(S): Oak Ridge National Laboratory, Pulp and Paper Research Institute of Canada, and Institute of Paper Science and Technology.

PRINCIPAL INVESTIGATOR(S): James R. Keiser, (423-574-4453)

PHASE OBJECTIVE: Measure residual stresses in unexposed and exposed 304L/SA210 composite tubes, predict the stresses in recovery boiler floor tubes during normal operation and during thermal transients, determine if the microstructure of an exposed tube has features characteristic of thermal cycling, identify environments that can cause stress corrosion cracking of 304L stainless steel, determine the effect of thermal cycling on cracking, continue the characterization of cracked tubes, and measure temperatures and strains in floor tubes.

ULTIMATE OBJECTIVE: Identify an alternate material or materials or an operational approach that can be used to prevent cracking of 304L/SA210 composite floor tubes.

TECHNICAL APPROACH: Metallographic examination techniques coupled with advanced analytical techniques are used to characterize unexposed and exposed, cracked tubes. Neutron and X-ray diffraction are used to measure the residual stresses in composite tubes, and finite element modeling is used to predict stresses under operating conditions. Environments that can cause stress corrosion cracking, and tests which can be used to determine the effects of thermal cycling are being evaluated by performing laboratory tests (includes tests which can be used to determine the frequency and magnitude of thermal cycles).

PROJECT SUMMARY (continued)

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Development of Materials for Black Liquor Recovery Boilers

PROGRESS: Stresses in operating composite tubes can be predicted, and studies are identifying environments that can cause stress corrosion cracking. Studies are also addressing the effects of thermal cycling on cracking of tubes.

Patents: --	**Publications: 5***	**Proceedings: --**
Books: --	**Presentations:12****	**Awards: 1*****

* Two papers published in conference proceedings, one laboratory report published, 2 papers published in the open literature and several manuscripts have been submitted for publication

** Not including presentations made at triannual program review meetings

*** Received the Best Paper Award for the TAPPI 1996 Engineering Conference

ACCOMPLISHMENTS:

Licenses: --

Known Follow-on Product(s): --

Industry Workshop: Program review meetings are held three times per year.

Technology Transfer or Industrial Interaction: Seventeen paper companies, four recovery boiler manufacturers, and two tube fabricators are active program participants.

CRITICAL ISSUES: Determination of dominant cracking mechanism(s), identification of materials that are resistant to dominant mechanism(s) or operating modifications that change conditions that cause cracking.

FUTURE PLANS: Continue to characterize cracked tubes provided by paper companies, measure and model the stresses developed in composite tubes with some emphasis on tubes that are on the edge of the floor or that form smelt spout openings and air ports, and work with paper companies to collect smelt samples, wash water samples, and floor tube temperature data.

POTENTIAL PAYOFF: The financial cost to paper companies resulting from recovery boiler material problems is enormous due to maintenance costs and the value of lost production. Identification of alternate materials, process changes and/or modifications of operating conditions should lessen or eliminate this financial burden.

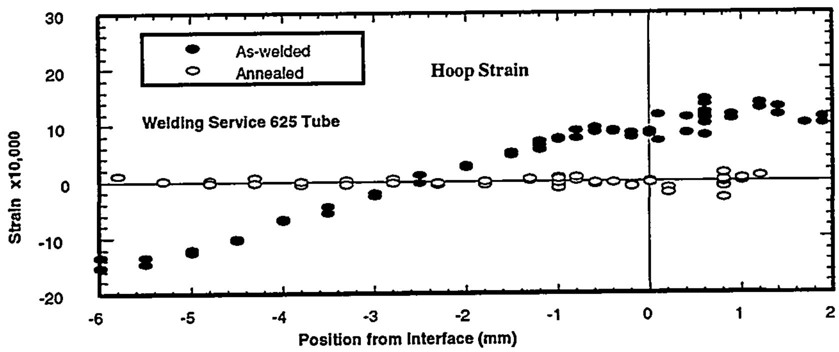
SIGNIFICANT ACCOMPLISHMENT

Advanced Industrial Materials (AIM) Program

Measurement and Modeling of Residual Stresses in Alloy 625 Weld Overlay on Carbon Steel Tubing

ISSUE: Cracking was found in tube panels (for use in kraft recovery boilers) made from carbon steel tubing with an Alloy 625 weld overlay. In order to determine if high stresses were associated with the cracking and to identify a means to avoid this cracking, residual stress measurements and finite element modeling were performed on samples of as fabricated and heat treated weld overlay tube.

RESULTS: Neutron and X-ray diffraction methods were used to measure the residual stresses in as fabricated carbon steel tubing with an Alloy 625 weld overlay. High tensile residual stresses were determined to be present in these tubes. Finite element modeling was also used to predict the stresses, and the results agreed well with the measured values. In an effort to relieve these stresses, a sample of tubing was given a 900°C heat treatment. Subsequent stress measurements (see Figure below) and finite element modeling studies established that the stress level in the tubes was greatly reduced. This stress reduction was attributed to the fact that this temperature resulted in stress relieving of the Alloy 625 and annealing of the carbon steel. Since the Alloy 625 and carbon steel have very similar coefficients of thermal expansion, no significant stresses were developed during cooling unlike what happens with a stainless steel and carbon steel.



Research performed at the Oak Ridge National Laboratory, sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Industrial Materials (AIM) Program, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

PROJECT SUMMARY

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Ni_3Al **and FeAl Technology Transfer**

PHASE: FY 1997 **PHASE COMPLETION DATE:**

PERFORMING ORGANIZATION(S): Oak Ridge National Laboratory (ORNL)

PRINCIPAL INVESTIGATOR(S): V. K. Sikka, (423-574-5112); G. Aramayo, (423-574-6503); M. L. Santella, (423-574-4805); and R. W. Swindeman, (423-574-5108)

PHASE OBJECTIVE: To promote the technology transfer of Ni_3Al-based nickel aluminide and FeAl-based iron aluminide alloys in the broadest spectrum of industry possible.

ULTIMATE OBJECTIVE: The ultimate objective is to take advantage of excellent oxidation and carburization resistances and higher strength of nickel and iron aluminides for a broad range of manufacturing-related industry applications. The applications identified to date include: furnace furniture (trays, fixtures, transfer rolls, belts, and conveyors), hot-pressing or forging dies, cast heating elements, and burners for gas fired furnaces.

TECHNICAL APPROACH: Since castings are the most likely near-term applications for both nickel and iron aluminides, the technical approach has been to address issues related to castings. These include: (1) optimization of a high-strength castable composition; (2) castability (mold type, fluidity, hot-shortness, porosity, and solidification modeling); (3) weld repairability of castings, welding for component fabrication and weldment properties; and (4) workability of cast or powder metallurgy product to sheet, bar, and wire.

PROGRESS: Production, welding, and application-related issues were addressed. New compositions were identified to be free of the eutectic formation at 1174°C and high-temperature (1150 to 1200°C) mechanical property data were developed for the new compositions (IC-396LZr and IC-438). A composition (B13) was identified for its excellent corrosion resistance in various aqueous solutions and for its high room-temperature hardness. A weld wire composition was identified and produced in pilot quantities for producing crack-free root passes in component welds.

PROJECT SUMMARY (Continued)

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Ni_3Al and FeAl Technology Transfer

Patents:	2	**Publications:**	5	**Proceedings:**	4
Books:	1*	**Presentations:**	6	**Awards:**	1

*Co-edited the volume entitled *Proceedings of the International Symposium on Nickel and Iron Aluminides: Processing, Properties, and Applications*, ASM-TMS Materials Week 96, Cincinnati, Ohio, 1997.

ACCOMPLISHMENTS:

Licenses: Three new licenses were signed. These were for sand and centrifugal castings with Alloy Engineering & Casting Company, centrifugal castings with Sandusky International, and weld wire with Polymet Corporation.

Other Successful Technology Transfer Activities as Evidence of Industry Interest: 1.) Testing was continued on a number of fixtures and trays by Delphi Saginaw in their production batch and pusher carburizing furnaces; 2.) Rolls at Bethlehem Steel Corporation (two of the rolls have completed three and one-half years of successful testing); 3.) A full furnace replacement order for trays from The Timken Company for their carburizing furnace and a full furnace order of tube hangers from Chevron.

CRITICAL ISSUES: Although significant progress was made during FY 1997, welding of nickel aluminides still continues to be a critical issue. Hot fabrication of aluminides is the second critical issue.

FUTURE PLANS: Continue Ni_3Al- and FeAl-base alloy technology transfer through solving technical issues, prototype manufacturing, and in-plant testing.

POTENTIAL PAYOFF: Nickel and iron aluminide components for a range of applications with major benefits of U.S. industry are gaining a competitive position in the world. Industries benefiting from nickel aluminide technology include steel, chemical, heat treating, forging, and the manufacturing sector.

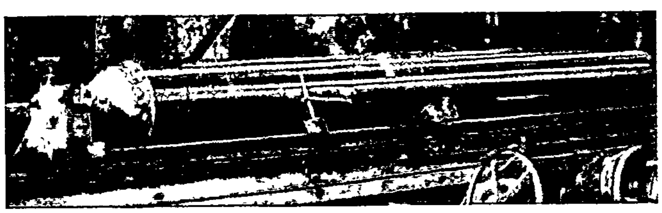
SIGNIFICANT ACCOMPLISHMENT

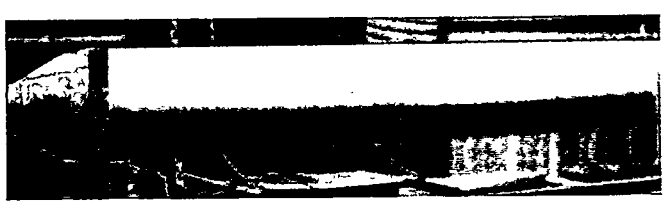
Advanced Industrial Materials (AIM) Program

High-Velocity Oxy Fuel (HVOF) Spraying of Nickel Aluminide on Cast Stainless Steel Rolls Paves the Way for New Applications

ISSUE: Nickel aluminides offer excellent resistance to high-temperature oxidation and carburizing, and, in most cases, it is being used as cast components. However, there are situations where it is desirable to only modify the surface of the components with nickel aluminide. The high-velocity oxy fuel (HVOF) process enables the succcessful application of fully dense coatings of nickel aluminide onto cast stainless steel pipe.

RESULTS: Nickel aluminide alloy IC-50 powder, produced by Ametek Specialty Metal Products Division (Eighty-Four, Pennsylvania), was HVOF sprayed by National Thermospray, Inc. (Houston, Texas) onto aluminum, carbon steel, and stainless steel test blocks. Following coupon testing, a full-size super 22H roll was coated to a thickness of 0.125 in. with IC-50 followed by its grinding to 0.110 in. Photographs of the rolls being sprayed and after grinding are shown below. The HVOF-coated roll has been in service in a stainless steel annealing furnace over the past six months.





ENERGY EFFICIENCY: Both extended roll life and reduced furnace down time contribute significantly to energy savings in the stainless steel annealing process.

Research performed at the Oak Ridge National Laboratory, sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Industrial Materials Program, and the Steel Industry Team under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

PROJECT SUMMARY

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Parallel Implementation of Casting Modeling Program

PHASE: FY 1997

PERFORMING ORGANIZATION: Oak Ridge National Laboratory

PRINCIPAL INVESTIGATORS: Thomas Zacharia, Srdan Simunovic, Srinath Viswanathan, and Philip F. Locascio

PHASE OBJECTIVE: Develop software design for parallel implementation of casting modeling program

ULTIMATE OBJECTIVE: Develop massively parallel implementation of casting modeling program

PROGRESS: A parallel version of heat flow conduction module in ProCAST has been ported on ORNL's parallel computers SGI Onyx and Intel Paragon. The performance of the implementation is currently being assessed. The function call trees and program profiling were performed to identify the main program functions for each of the modules and determine a proper sequence and subsets of functions that need to be parallelized.

ACCOMPLISHMENTS: Collaboration with industrial partner is under development to establish multi-organizational approach to parallelization of casting modeling program.

SIGNIFICANT ACCOMPLISHMENT

Advanced Industrial Materials (AIM) Program

Parallel Implementation of Casting Modeling Program

ISSUE: The commercial casting code ProCAST, from UES, Inc., has been chosen to be implemented on massively parallel computers at ORNL. ProCAST is a finite element code that can be used for the analysis of complex casting shapes by modeling coupled effects of heat flow, fluid flow, solidification and stress. The program complexity requires careful analysis and assessment of the code in order to create efficient parallel implementation.

RESULTS: A parallel version of heat flow conduction module in ProCAST has been ported on ORNL's parallel computers SGI Onyx and Intel Paragon. The function call trees and program profiling were performed to identify the main program functions for each of the modules and determine a proper sequence and subsets of functions that need to be parallelized.

Research performed at the Oak Ridge National Laboratory, sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Industrial Materials Program, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

PROJECT SUMMARY

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Synthesis and Design of Silicide Intermetallic Materials

PHASE: FY-1997 **COMPLETION DATE:**

PERFORMING ORGANIZATION(S): Los Alamos National Laboratory (LANL)

PRINCIPAL INVESTIGATOR(S): J. J. Petrovic, (505-667-0125) and R. G. Castro, (505-667-5191)

PHASE OBJECTIVE: Conduct CRADA with Johns Manville Corporation on silicide materials for fiberglass processing components. Collaborate with the Institute of Gas Technology (IGT) to test silicide materials in gas radiant tube combustion environments. Develop $MoSi_2$-based composites, the plasma spraying of $MoSi_2$-based materials, and the joining of $MoSi_2$ materials to metals.

ULTIMATE OBJECTIVE: To develop $MoSi_2$-based high temperature structural silicide materials with optimum combinations of properties for applications of importance to U.S. processing industries, and particularly the glass processing industry.

TECHNICAL APPROACH: Develop silicide-based high temperature structural materials, with current focus on $MoSi_2$-Si_3N_4, $MoSi_2$-SiC, and $MoSi_2$-oxide composites. Develop processing methods for silicide materials, with emphasis on plasma spraying and plasma spray forming. Develop $MoSi_2$-based prototype fiberglass processing components and other components for glass processing. Develop gas combustion-related applications for silicide materials

PROGRESS: CRADA No. LA95C10271-A001 "Advanced High Temperature Materials for Glass Applications" with Johns Manville Corporation is continuing. New interactions with the Institute of Gas Technology and Combustion Tec Inc. have been initiated.

Patents: -	**Publications:** 12	**Proceedings:** 3
Books: -	**Presentations:** 13	**Awards:** 2

PROJECT SUMMARY (Continued)

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Synthesis and Design of Silicide Intermetallic Materials

ACCOMPLISHMENTS: The corrosion behavior of $MoSi_2$ materials in molten fiberglass has been initially evaluated, and is similar to AZS refractory. Maximum corrosion rates occur at the glass-air interface. Efforts have been initiated with IGT to test $MoSi_2$ materials in a gas radiant tube environment. This will be a 500 hour test at 1800 °F under gas combustion conditions.

Licenses: -
Known Follow-On Product(s): $MoSi_2$ gas injection tube for glass melting (Kanthal)
Industry Workshop: None
Technology Transfer or Industrial Interaction: Johns Manville, IGT, Combustion Tec, Exotherm

CRITICAL ISSUES: Improvement in $MoSi_2$-based material corrosion behavior at the glass-air melt line. Behavior of $MoSi_2$ materials in the gas combustion environment.

FUTURE PLANS: With Johns Manville, we will continue the development of $MoSi_2$ applications for fiberglass processing. We plan to test $MoSi_2$ materials in the gas combustion enivironment using test facilities at IGT. A $MoSi_2$ sight tube for a glass furnace monitoring system is being fabricated for test by Combustion Tec. We are developing the near-net-shape directed light fabrication approach for silicide materials.

POTENTIAL PAYOFF: The potential payoff for industrial applications of high temperature $MoSi_2$-based structural silicides is very high. Major industrial applications exist in the areas of glass and metal processing equipment, industrial gas burners and lances, furnace heating elements and radiant gas heaters, and high temperature industrial components. An environmental payoff also exists since $MoSi_2$-based radiant burners and heaters that can burn mixtures of pure oxygen and natural gas will reduce NO_x and CO_2 emissions.

ESTIMATED ENERGY SAVINGS: Potential energy savings of 0.2 Quads (1 Quad = 10^{15} btu) will occur from the use of $MoSi_2$-based materials in industrial applications.

SIGNIFICANT ACCOMPLISHMENT

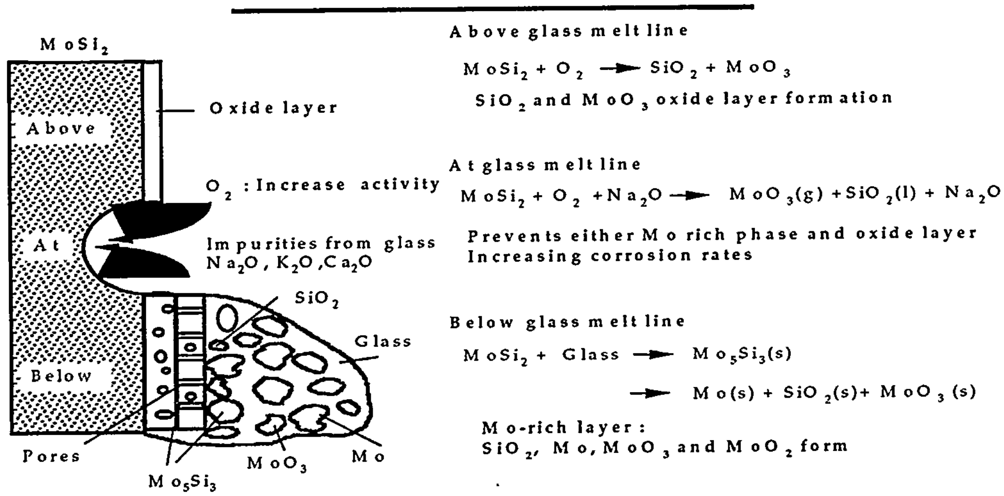
Advanced Industrial Materials (AIM) Program

Corrosion Behavior of $MoSi_2$ in Molten Glass

ISSUE: $MoSi_2$ is a material with potential for the use in contact with molten glass. Understanding the rates and mechanisms of corrosion is key to the successful adoption of $MoSi_2$-based materials in industrial glass operations.

RESULTS: The corrosion behavior of $MoSi_2$ in molten fiberglass compositions has been examined. This has involved both static and dynamic corrosion experiments at temperatures in the range of 1050 °C-1550 °C. Corrosion rates of $MoSi_2$ are comparable to those of AZS refractory. Dynamic corrosion is similar to static corrosion. Maximum corrosion rates for $MoSi_2$ occur at the glass-air line. Above the glass line, the formation of a protective SiO_2 layer occurs, while below the glass line, a protective Mo-rich layer is formed. At the glass line, no protective layers are formed in pure $MoSi_2$. Efforts are now concentrating on optimizing the corrosion behavior at the glass line, through composite, alloying, and anodic protection approaches.

Corrosion Mechanisms in $MoSi_2$



ENERGY EFFICIENCY: Potential energy savings of 0.2 Quads will occur from the use of $MoSi_2$-based materials in industrial applications, such as glass processing.

Research performed at the Los Alamos National Laboratory, sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Industrial Materials Program, and the Glass Industry Team under contract to the Los Alamos National Laboratory.

PROJECT SUMMARY

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Uniform-Droplet Process

PHASE: FY 1997

PERFORMING ORGANIZATION(S): Oak Ridge National Laboratory (ORNL), Massachusetts Institute of Technology (MIT), and Northeastern University

PRINCIPAL INVESTIGATOR(S): C. A. Blue/ORNL, (423-574-4351); V. K. Sikka/ORNL, (423-574-4351); J. H. Chun/MIT, (617-253-1759); and T. Ando/Tufts, (617-628-5000 ext. 5163)

PHASE OBJECTIVE: To translate the uniform-droplet spray forming process to high-temperature materials (including aluminides) and promote technology transfer of the process to the broadest spectrum of industry as possible.

ULTIMATE OBJECTIVE: To develop the process for a broad range of manufacturing-related industrial applications. The applications identified to date include: water filtration systems (bronze), shot production, spray forming, ball bearing production, aluminum sheet production, ball-grid array (BGA) type integrated-circuit (IC) packaging, and powder production.

TECHNICAL APPROACH: Since many applications exist for uniform droplets of high-temperature materials, the technical approach has been to address issues relating to the development of a high-temperature uniform droplet system. Efforts focus on: 1.) development of low- and medium-temperature systems, 2.) determination of the low- and medium-temperature optimum spraying conditions for materials with melting points between 230 and 1250°C, and 3.) development of a high-temperature uniform-droplet powder and spray system.

PROJECT SUMMARY (Continued)

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Uniform-Droplet Process

PROGRESS: The uniform-droplet project requires that three droplet heating systems, one for low-temperature metals (T_m< 400°C), one for medium-temperature metals (T_m 400 to 1250°C), and one for high-temperature metals (T_m 1250 to 1650°C) be assembled. The fabrication of the low-temperature system provided a basis for the design of the medium-temperature unit. Construction of the low-, medium-, and high-temperature uniform-droplet systems at ORNL has been completed. The main focus now is in the area of materials and orifice development for spraying high-temperature materials.

The high-temperature apparatus has been assembled, and preliminary testing is in progress. This system has enabled the fabrication of uniform droplets of materials with melting points as high as 1250°C. These materials include: aluminum, bronze, and copper. Also, this system was designed for higher volume melts (15 to 20 lb, materials dependent). Therefore, this system will aid in the development of the uniform-droplet spray system for industrial practices. Two companies visited ORNL gathering the technical knowledge for translation of the process to industry.

Patents:	**0**	**Publications:**	3	**Proceedings:**	**0**
Presentations:	**5**				

CRITICAL ISSUES: Orifice clogging and materials compatibility.

FUTURE PLANS: Knowledge will be gained through solving technical issues with the medium- and high-temperature uniform droplet and applying the uniform-droplet spray technology to near-net-shape fabrication of parts. Efforts related to multinozzle systems applicable for spray forming of sheet will be initiated. Interactions with the industrial sector and transfer of technology of the low-, medium-, and high-temperature systems will continue.

POTENTIAL PAYOFF: The uniform-droplet spray process allows for the fabrication of mono-sized powders near 100% yield, which eliminates the sieving of powder as performed with the present powder-forming technologies. Also, the powders are spherical and possess a more uniform microstructure, which results in a superior powder. The development of multinozzle spray systems can have positive impact on metal sheet production processes.

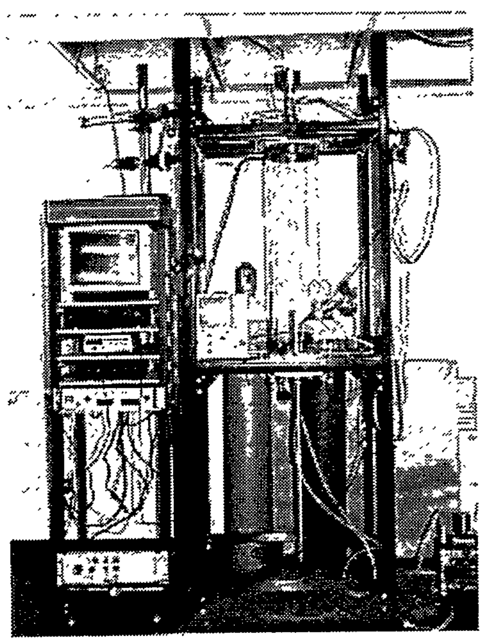
SIGNIFICANT ACCOMPLISHMENT

Advanced Industrial Materials (AIM) Program

Fabrication of the Medium-Temperature (1250°C) and High-Temperature Uniform-Droplet Spray Facilities is Completed, and Full Development of the Process is Under Way

ISSUE: The initial uniform-droplet spray (UDS) process has been developed at the Massachusetts Institute of Technology for spraying low-melting-point materials. The current project seeks to develop the technology where it has the greatest potential for industrial applications; that is in the medium- to high-melting-temperature alloys.

RESULTS: Medium-, and high-temperature UDS systems have been fabricated at the Oak Ridge National Laboratory. The UDS process allows for fabrication of powders of a single size with no sieving and spraying of materials with identical masses and thermal histories. The high-temperature unit has been utilized for spraying materials with melting points as high as 1250°C. This unit (see figure below) has been utilized successfully for spraying aluminum, bronze, and copper materials.



ENERGY EFFICIENCY: The UDS process allows for the fabrication of mono-sized powders near 100% yield, which eliminates the sieving of powder as performed with the present powder-forming technologies. Also, the powders are spherical and possess a more uniform microstructure, which results in a superior powder. The development of multinozzle spray systems can have positive impact on metal sheet production processes.

NEW MATERIALS AND PROCESSES

PROJECT SUMMARY

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Advanced Industrial Materials (AIM) Fellowship Program

PHASE: FY 1997

PERFORMING ORGANIZATIONS: Oak Ridge Institute for Science and Education, Oak Ridge, TN; Virginia Polytechnic Institute and State University, Blacksburg, VA; University of Cincinnati, Cincinnati, OH; Oak Ridge National Laboratory, Oak Ridge, TN.

PRINCIPAL INVESTIGATORS: D. D. McCleary, Oak Ridge Institute for Science and Education; B. D. Dickerson, S. B. Desu, M. K. Lian, and B. J. Love, Virginia Polytechnic Institute and State University; A. Jordan and O. N. C. Uwakweh, University of Cincinnati; J. R. Keiser, E. A. Kenik, P. J. Maziasz, and D. F. Wilson, Oak Ridge National Laboratory.

PHASE OBJECTIVE: The Graduate Fellowship Program focused toward helping students who are currently under represented in the nation's pool of scientists and engineers, enter and complete advanced degree programs.

ULTIMATE OBJECTIVE: The objectives of the program are to: 1) establish and maintain cooperative linkages between DOE and professors at universities with graduate programs leading toward degrees or with degree options in Materials Science, Materials Engineering, Metallurgical Engineering, and Ceramic Engineering, the disciplines most closely related to the AIM Program at Oak Ridge National Laboratory (ORNL); 2)strengthen the capabilities and increase the level of participation of currently under represented groups in master's degree programs, and 3) offer graduate students an opportunity for practical research experience related to their thesis topic through the three-month research assignment or practicum at ORNL. The program is administered by the Oak Ridge Institute for Science and Education (ORISE). The following abstracts summarize the activities of three of the participants.

TECHNICAL APPROACH: Offer fellowships for master's degree program in materials science-related academic fields.

PROGRESS: Three fellowship recipients are involved in the project. (See following Significant Accomplishments.)

SIGNIFICANT ACCOMPLISHMENT

Advanced Industrial Materials (AIM) Program

Section 1.
Optically Monitoring Conductive Bi_2O_3 Content in Ferroelectric Capacitors

ISSUE: A systematic analysis method based on effective-media approximations (EMA) enabled variable angle spectroscopic ellipsometry (VASE) to accurately characterize second phase Bi_2O_3 contamination in ferroelectric $SrBi_2Ta_2O_9$ (SBT) films. Excess Bi_2O_3 is often included in precursor solutions for metal-organic deposition (MOD) of SBT to promote grain growth at lower annealing temperatures. However, any second phase Bi_2O_3 which remains in the final crystallized film may significantly increase the leakage current.

RESULTS: As expected from the presence of a more conductive second phase, the conductivity of contaminated films was generally several orders of magnitude higher than that of standard SBT. Ellipsometry was able to estimate second phase Bi_2O_3 contents that were consistent with measured electrical properties.

Optically detectable Bi_2O_3 in SBT films generally reduced grain size and increased leakage current. Ellipsometry was particularly useful in this system because XRD did not show any consistent trends in Bi_2O_3 peak heights, even between films with drastically different electrical properties. A more detailed description of these results has been submitted for publication.

Research performed at Virginia Polytechnic Institute, sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Industrial Materials (AIM) Program; and Division of Materials Sciences, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

SIGNIFICANT ACCOMPLISHMENT

Advanced Industrial Materials (AIM) Program

Section 2.
FeSi Phase Formation in Mechanically Alloyed Fe-Zn-Si Intermetallics

ISSUE: The galvanization of Si-bearing steels have been known to give rise to abnormal coating structures, which has been linked to a number of problems including, poor corrosion resistance, coating non-uniformity and early deterioration. In order to enhance to performance of the coating upon Si-induced steels, a better understanding on how the influence of Si affects the coating properties is needed.

RESULTS: Our results have shown that the formation of an FeSi phase, which is found in several of the single and mixed phase Fe-Zn binary alloys, is most likely responsible for the reactive process associated with the Zn coating of Si-bearing steels. Several articles, indicating these findings, have been published in various technical journals or are in press.

Research performed at University of Cincinnati, sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Industrial Materials (AIM) Program, and administered by the Oak Ridge Institute for Science and Education (ORISE).

SIGNIFICANT ACCOMPLISHMENT

Advanced Industrial Materials (AIM) Program

Section 3.
Mechanical Behavior of Epoxy Bonded Joint For Pipe Repair

ISSUE: There are instances where efficiency and safety may be compromised as a result of damaged pipes. The development of methods that can repair the damage for a temporary period without shutting down the operations are critical. The objective of this project is to evaluate the mechanical properties of the epoxy bonded joints in aqueous corrosion environments.

RESULTS: The epoxy adhesive studied included EPON® resin 828, dicyandiamide, and 2-methylimidazole and low-alloy carbon steel was chosen as the substrate. The epoxy bonded joints were exposed in either distilled water or 3.4% NaCl solution at 50°C for one or two weeks and three-point bend test was used to evaluate the mechanical properties of the bonded joints. The shear stress at the interface between the epoxy and the steel was calculated, and the results are shown in Figure 1.

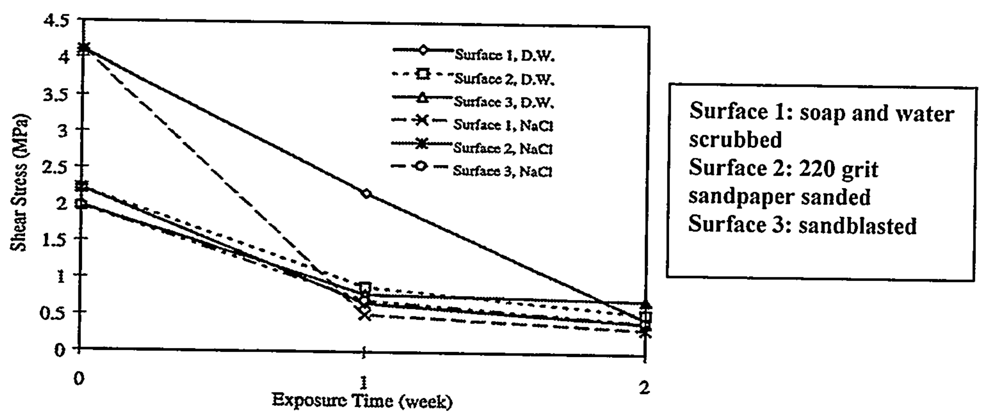


Figure 1: Shear Stress for Epoxy Bonded Steel

The average shear stress of the epoxy bonded joints decayed with time for all three surface treatments in both aqueous environments. The environment may have had penetrated and weakened the interface. Therefore, the bond strength decreased with increasing exposure time. The samples made with sandblasted steel had the highest shear stress after two weeks of exposure in 3.4% NaCl solution.

Research performed at Virginia Polytechnic Institute, sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Industrial Materials Program, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

PROJECT SUMMARY

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Development of Improved Refractories

PHASE: FY 1997

PERFORMING ORGANIZATION(S): Oak Ridge National Laboratory (ORNL) and the University of Missouri at Rolla (UMR).

PRINCIPAL INVESTIGATOR(S): Andrew A. Wereszczak/ORNL (423-574-7601), Kenneth C. Liu/ORNL (423-574-5116), and Robert E. Moore/UMR (573-341-4401)

PHASE OBJECTIVE: Construct mechanical test frames to facilitate high temperature mechanical testing of refractories and survey the glass manufacturing community to determine which refractory materials for superstructure applications they desire engineering data for.

ULTIMATE OBJECTIVE: Determine the thermomechanical and thermophysical properties of commercial refractories that are candidate materials for glass-melting furnace superstructures.

TECHNICAL APPROACH: High temperature mechanical data will be generated on commercially available refractories which are candidate materials for glass furnace superstructures. The creep rate of these materials will be measured as a function of stress and temperature; chosen stresses and temperatures will bracket those which exist during service in the superstructure. The data will be made available to glass manufacturers so that they may (1) make equitable comparisons which would facilitate their choice of refractory, and (2) provide design data for furnace-design-engineers to optimize superstructure design.

PROGRESS: Two test frames were constructed during FY97 and the load cells and extensometers for both frames were conditioned and calibrated. The frames are capable of accurately measuring compressive creep strain in refractory specimens up to 1800°C. A glass industry advisory committee (GIAC), comprised of refractory vendors and glass manufacturing representatives, was formed so the present project would receive insight, advice, and input from them. Representatives from thirty-four domestic glass manufacturers were surveyed to determine which refractory materials their furnace designers desired engineering data. Eight refractory materials were identified as the most popular (fused-cast alumina, andalusite, bonded AZS, fused-cast AZS, fused-grain mullite, conventional silica, fused silica, and bonded zircon. Conventional silica and fused-cast alumina were prioritized and characterization and mechanical testing was begun.

PROJECT SUMMARY (Continued)

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Development of Improved Refractories

Patents: -	**Publications:** -	**Proceedings:** -
Books: -	**Presentations:** 4	**Awards:** -

ACCOMPLISHMENTS: There were two primary accomplishments during FY97. First, the Glass Industry Advisory Committee (GIAC) was formed to provide guidance. The GIAC is comprised of nine representatives from refractory vendor companies, glass manufacturers, and academia. All members are recognized leaders in their respective fields of refractory work. Second, a survey was completed in which container-, fiber-, flat-, and special-glass manufacturing representatives identified which refractory materials the project is to mechanically test. The results from this survey were the first of their kind (i.e., glass manufacturing competitors willingly identified refractory materials of interest) used in a manner to identify refractory materials and mechanical test matrices for such an objective test program.

CRITICAL ISSUES: Identifying glass manufacturers who are willing to allow the present project to place refractory specimens in their glass furnaces in order to expose the materials to service environments.

FUTURE PLANS: With the refractory materials identified (eight in all), conventional silica and fusion-cast alumina refractories will be the first to be tested. Specimens will also be subjected to service environments and their corrosion resistance will be examined, as well as the effect of exposure on mechanical performance.

POTENTIAL PAYOFF: More efficient and economical designs of glass-melting furnace superstructures will be a consequence of this project. Furnace superstructure designers will know which refractory materials perform better than others (from objectively generated data). Additionally, accurate engineering data will be available to describe the high temperature mechanical performance of candidate refractory materials and furnace superstructures made from them.

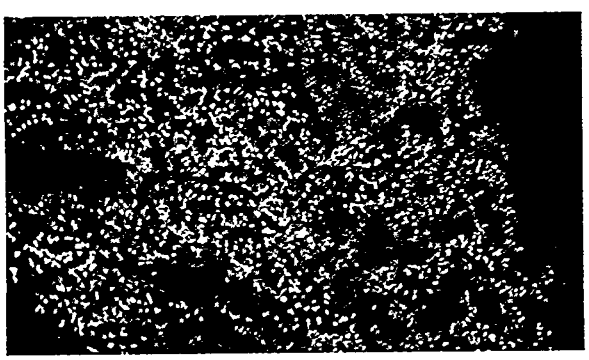
ESTIMATED ENERGY SAVINGS: Energy efficient glass melting furnaces will enhance overall process efficiencies. Improved refractory life time and stability will improve energy efficiency, productivity, and minimize waste glass.

SIGNIFICANT ACCOMPLISHMENT

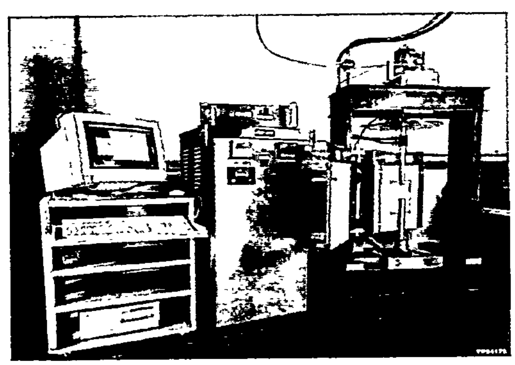
Advanced Industrial Materials (AIM) Program
Development of Improved Refractories

ISSUE: There are literally several dozen candidate commercially-available refractories for use in glass furnace superstructures. Refractory vendors supply technical data on their own brands, but it is difficult for the glass manufacturers to equitably judge them because the data is almost always generated or reported differently among the vendors. The present project wanted to survey domestic glass (container, fiber, flat, and specialty) manufacturers to determined and identify which classes of refractories they needed mechanical engineering data on. The present project will then objectively generate such data so these glass manufacturing companies would be able to (1) compare performances and choose among competing brands, (2) provide engineering data which furnace design engineers could use for the optimizing the design of furnace superstructures.

RESULTS: Representatives from the glass companies were asked to prioritize refractories from thirteen categories [fused-cast alumina, andalusite, bonded AZS, fused-cast AZS, chrome (5-15%), chrome (30%), chrome (50-80%), magnesia, fused-grain mullite, conventional silica, fused silica, spinel, and bonded zircon]. The results showed the companies were enthusiastic about having eight (fused-cast alumina, andalusite, bonded AZS, fused-cast AZS, fused-grain mullite, conventional silica, fused silica, and bonded zircon) of these thirteen categories mechanically tested. These results are being used to formulate the test matrices for the present project. These survey results are important because industry is providing the driving force behind which refractories are to be tested.



Catholuminescence image of the microstructure of a silica refractory tested in the program. Silica grains are shown as blue, the dark regions are pores, and the light regions are calcium silicate.



Compressive creep facilities were constructed to accurately measure time dependent deformation of refractories at elevated temperatures.

Research performed at the Oak Ridge National Laboratory, and the University of Missouri, Rolla, sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, and the Glass Industry Team, Office of Industrial Technologies, Advanced Industrial Materials Program, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

PROJECT SUMMARY

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Gelcasting Polycrystalline Alumina

PHASE: FY 1997 **COMPLETION DATE:** FY 1998

PERFORMING ORGANIZATION(S): Oak Ridge National Laboratory (ORNL)

PRINCIPAL INVESTIGATOR(S): Mark A. Janney, 423-574-4281

PHASE OBJECTIVE: Develop gelcasting of optical grade alumina for lamp applications

ULTIMATE OBJECTIVE: The purpose of the CRADA with Osram-Sylvania Inc. is to determine the feasibility of making polycrystalline alumina items using gelcasting having sufficient optical quality that they are useful in lighting applications.

TECHNICAL APPROACH: Gelcasting is an advanced powder forming process. It is most commonly used to form ceramic or metal powders into complex, near-net shapes. Turbine rotors, gears, nozzles, and crucibles have been successfully gelcast in silicon nitride, alumina, nickel-based superalloy, and several steels.

Osram-Sylvania would like to explore using gelcasting to form PCA tubes for Lumalux® lamps, and eventually for metal halide lamps (known as quartz-halogen lamps). Osram-Sylvania, Inc. currently manufactures PCA tubes by isostatic pressing. This process works well for the shapes that they presently use. However, there are several types of tubes that are either difficult or impossible to make by isostatic pressing. It is the desire to make these new shapes and sizes of tubes that has prompted Osram-Sylvania's interest in gelcasting.

PROGRESS: We demonstrated in FY 1996 that we could gelcast optical quality straight, thin-walled tubes. In FY 1997, we extended this work to complex-shaped tubes. Several methods were developed for making complex-shaped parts using fugitive (meltable) cores for casting. Over 100 complex-shaped parts were fabricated using these fugitive cores. The parts fired well and had excellent microstructures. The optical properties of these parts were equivalent to those obtained for commercial straight tubes.

Patents: 1 in preparation **Publications:** none **Proceedings:** none
Books: none **Presentations:** none **Awards:** none

PROJECT SUMMARY (continued)

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Gelcasting Polycrystalline Alumina

ACCOMPLISHMENTS:

Licenses: none

Technology Transfer or Industrial Interaction: Osram-Sylvania is actively pursuing applications of gelcast tubes to their product line.

CRITICAL ISSUES: Developing a manufacturing line from successful laboratory tests will be critical to the successful transfer of this technology.

FUTURE PLANS: Osram-Sylvania will evaluate if this technology can be used in mass production.

POTENTIAL PAYOFF: Gelcasting could eliminate several geometric constraints in the manufacture of complex-shaped arc tubes. Potential increases in lighting efficiency of 3% are predicted. Since lighting consumes about 25% of all the electricity used in the United States, this would represent a major energy conservation effort.

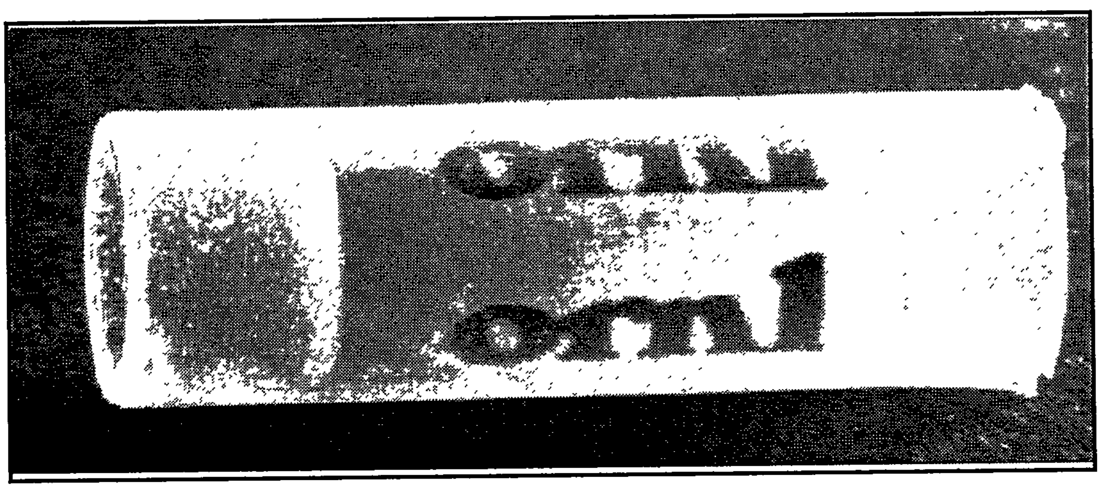
SIGNIFICANT ACCOMPLISHMENT

Advanced Industrial Materials (AIM) Program

Gelcasting Polycrystalline Alumina for High Pressure Sodium Arc Lamps

ISSUE: High-pressure sodium vapor arc lamps are used for industrial, highway, and street lighting. The key to the performance of these lamps is the polycrystalline alumina (PCA) tube, which contains the plasma that is formed in the electric arc. Currently, there are severe shape limitations on the alumina tubes. Gelcasting tubes should eliminate most of the geometric constraints on fabricating lamp tubes. Successful completion of the work done under this cooperative research and development agreement (CRADA) with Osram-Sylvania Inc. could result in a new family of energy-saving lamps. Lighting efficiency increases up to 3% are predicted.

RESULTS: In FY 1997, we further demonstrated that high transmittance could be maintained in gelcast complex-shaped tubes of a proprietary design (see Figure). We also applied several fugitive core strategies to gelcasting complex-shaped, thin-walled objects using meltable core materials such as waxes and fusible alloys. These approaches allowed production of thin-walled objects in large numbers (over 100 were cast) with excellent quality and repeatability.



A thin-walled gelcast PCA tube (0.3 inch diam, 0.020 inch wall) with excellent transmission.

Research performed at the Oak Ridge National Laboratory, sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Industrial Materials Program, under contract DE-ACOR-96OR22464 with Lockheed Martin Energy Research Corporation.

PROJECT SUMMARY

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Materials R&D — Student Internships

PHASE: FY 1997 **COMPLETION DATE:** September 30, 1997

PERFORMING ORGANIZATION: Ames Laboratory

PRINCIPAL INVESTIGATORS: R. Bruce Thompson, (515-294-9649), and Scott Chumbley, (515-294-7903)

PHASE OBJECTIVE: To continue programmatic research while educating minority students.

ULTIMATE OBJECTIVE: The conduct of programmatic research of the Advanced Industrial Concepts Materials Program while training minority graduate students in the process.

TECHNICAL APPROACH: The approach for involving minorities involves recruiting students for participation in the program, matching them with projects of programmatic interest to the Advanced Industrial Materials Program, conduct of the research, continuous mentoring and graduation.

The specific technical approach will depend on the projects selected. The primary work in the current year is directed towards development of plasma torch techniques for production of nanocrystalline materials in large quantities and for depositing Cr layers without hazardous baths.

PROGRESS: Work in the development of plasma techniques was successfully concluded with the graduation of the student involved in this study. The conditions under which a plasma torch could be used to produce nanocrystalline intermetallic compounds were defined and confirmed. The feasibility of using a plasma torch to produce a thin Cr layer was also demonstrated, although experiments showed that tighter control over the deposition chamber atmosphere was required to produce coatings having the required wear characteristics. Papers were prepared on both areas of research.

PUBLICATIONS: Two

PROJECT SUMMARY (Continued)

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Materials R&D — Student Internships

ACCOMPLISHMENTS:

Licenses: None

Known Follow-on Product(s): None

Industry Workshop: None

Technology Transfer or Industrial Interaction: Collaboration was established with John Deere and Eaton Corp. on the Cr plating portion of the study. A key component was industrially relevant samples provided by the companies.

CRITICAL ISSUES: Identifying appropriate students and assisting them in what can be a significant transition from their undergraduate institution to a major research university.

FUTURE PLANS: Continued recruitment and education of minority students.

POTENTIAL PAYOFF: Increasing the fraction of the population available to perform technical jobs for the nation.

SIGNIFICANT ACCOMPLISHMENT

Advanced Industrial Materials (AIM) Program

Materials R&D — Depositing a Hard Cr Layer on a Metallic Substrate Student Internships

ISSUE: Depositing a hard Cr layer on a metallic substrate. If mechanical properties can be improved through improved atmospheric control, it may be possible to produce wear resistant Cr films without the use of environmentally harmful chemical baths.

RESULTS: Using plasma techniques and an atmospherically controlled chamber (Figure 1) thin Cr layers were deposited on Cu and steel substrates. Two different feed materials were used in the plasma system, Cr powder and chromium hexacarbonyl, $Cr(CO)_6$. Of these two materials, the Cr produced films using the $Cr(CO)_6$ precursor were generally more uniform and exhibited better adherence to the substrate than those produced using Cr powder, (Figure 2). The mechanical properties of both Cr films were below the properties of electroplated Cr films, due principally to the presence of oxygen and various Cr oxides in the films. Additional work is planned.

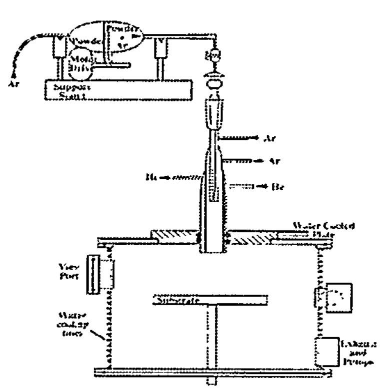


Figure 1. Schematic of Deposition Chamber.

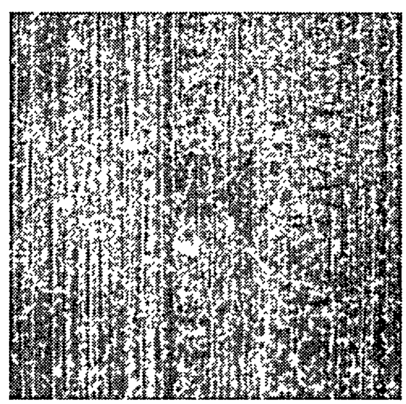


Figure 2. $Cr(CO)_6$ Powder films deposited on mild steel substrate.

ENERGY EFFICIENCY: Plasma processing could result in significant energy savings by eliminating the energy costs associated with shipping, storing, and cleaning the presently used chemical solutions.

Research performed at the Ames Laboratory, sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Industrial Materials Program, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

PROJECT SUMMARY

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Metals Processing Laboratory User Facility (MPLUS)

PHASE: FY 1997 **COMPLETION DATE:**

PERFORMING ORGANIZATION(S): Oak Ridge National Laboratory (ORNL)

PRINCIPAL INVESTIGATOR(S): G. Mackiewicz-Ludtka (423-576-4652)

PHASE OBJECTIVE: Partner with DOE Vision Industries and Universities by providing access to the unique Technical Expertise and Facilities at MPLUS.

ULTIMATE OBJECTIVE: To assist U.S. industries improve energy efficiency and enhance their competitiveness of in the global market by partnering with National Laboratories.

TECHNICAL APPROACH: Started as a small-scale, pilot User Facility Program in February of 1996, MPLUS's initial goal was to determine whether a need existed for such a program, and if so, to demonstrate its value. As of September 30, 1997, a total of 76 MPLUS Proposals were received from 60 companies and universities representing 26 states. This represents approximately a 50% increase in proposals, and greater than a 50% increase in the number of states that have requested MPLUS facilities and expertise since the inception of the MPLUS Program on February 7, 1996. The continued demand for and the success of the MPLUS facilities are evidenced by a.) the sheer number of users (60 Companies); b.) the twelve (12) organizations who requested repeated assistance and submitted multiple (2 to 4) proposals for different MPLUS projects; and c.) the five (5) companies who chose to do Proprietary (P) MPLUS projects and hence, pay full-cost-recovery to access MPLUS. In fact, two (2) of these five (5) P Users did multiple P MPLUS projects. In addition a total of 546 user days were logged during FY1997, which is approximately a 100% increase since FY96. This tremendous response to the MPLUS program continues to demonstrate, through the continually increasing numbers of industrial requests, that U.S. industry approves of, and supports the mission of MPLUS program, and that these companies view the MPLUS Program as beneficial in achieving their corporate missions.

PROJECT SUMMARY (Continued)

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Metals Processing Laboratory User Facility (MPLUS)

PROGRESS: Due to industrial continued requests/needs and support, MPLUS has succeeded in demonstrating (within its twenty (20) months of existence), that the unique Technical expertise and Facilities accessible through the National Laboratories are a valuable resource to U.S. Industries.

Patents: ERID0344	**Publications:**	**Proceedings: -**
Books: -	**Presentations: ~20**	**Awards: 1**

ACCOMPLISHMENTS:

Licenses: - **Known Follow-on Product(s):** Some companies have funded several proprietary (P) MPLUS projects at the conclusion of their NP project, while others have continued *other* subsequent industrial/ORNL interactions.

Technology Transfer or Industrial Interaction: Occurs with every project.

CRITICAL ISSUES: Can National Laboratories be a valuable asset to U.S. industries by providing unique and valuable input to materials processing issues so that U.S. industries can reclaim their competitive edge in the global market?

FUTURE PLANS: Expand MPLUS to incorporate facilities and expertise at other sites.

POTENTIAL PAYOFF: Restoration of U. S. industry's competitive edge in the global market.

ESTIMATED ENERGY SAVINGS: Varies per individual project.

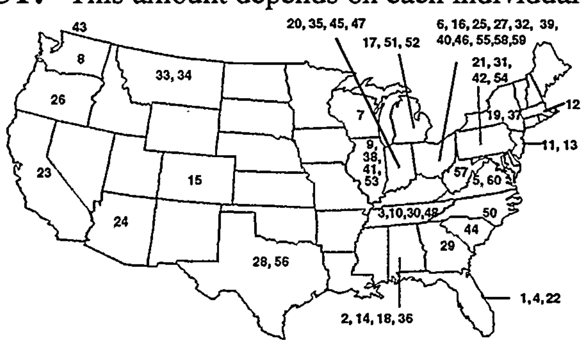
SIGNIFICANT ACCOMPLISHMENT

Advanced Industrial Materials (AIM) Program
The Metals Processing Laboratory User Center (MPLUS)

ISSUE: Continuation of pilot test of the Metals Processing Laboratory User (MPLUS) Facility.

RESULTS: As of September 30, a total of **76 MPLUS Proposals** were received from **60 companies and universities representing 26 states** (see figure below). The continued demand for and the success of the MPLUS facilities are evidenced by a.) the sheer number of users (60 Companies); b.) the twelve (12) organizations who requested repeated assistance and submitted multiple (2 to 4) proposals for different MPLUS projects; and c.) the five (5) companies who chose to do Proprietary (P) MPLUS projects and hence, pay full-cost-recovery to access MPLUS. In fact, two (2) of these five (5) P Users did multiple P MPLUS projects. A total of **546 user days** were logged during FY1997.

ENERGY EFFICIENCY: This amount depends on each individual MPLUS project.



1. Westinghouse (2)
2. Reynolds(2)
3. ForMat Industries
4. Memtec
5. E.R. Johnson
6. Sandusky Int'l
7. Waukesha Electric
8. Weyerhauser(2)
9. A.Finkl(3)
10. Jeffrey Chain Corp
11. Materials Tech.
12. ABB C-E Serv. (3)
13. Union Camp Corp
14. United Defense(2)
15. CO School of Mines
16. Doehler-Jarvis
17. General Motors
18. Univ. of AL(2)
19. Cornell Univ.
20. Cummins Engine(3)
21. Bethlehem Steel(2)
22 Anchor Glass
23. FMC Corp.
24. Arizona State U
25. Ohio State Univ
26. Goldendale Al
27. Lincoln Electric
28. CarboMedics
29. IPST
30. Univ. of TN
31. PPG industries (4)
32. Owens Corning(2)
33. Columbia Falls Al
34. AFFCO
35. Allison Engine
36. MacMillan-Bloedel
37. Gray-Syracuse
38. Wagner Castings
39. Rhenium Alloys
40. Uniform Metal Tech
41. Amoco
42. J&L Specialty Steel
43. Process Simulations
44. Milliken Res. Ctr.
45. Welding Services
46. General Electric
47. MicroPyretics
48. Pathway Bellows
49. Stoody Co.
50. Torrington(2)
51. Ford Motor Co.
52. Eaton Corp.
53. Hoskins Mfg.(2)
54. AHT Inc
55. Timken Co.
56. B.F.Goodrich
57. INCO Alloys Int'l
58. LTV Steel
59. Park OH Transp.
60. VA Poly.& St. U.

Research performed at the Oak Ridge National Laboratory, sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Industrial Materials Program, under contract DE-AC05-99OR22464 with Lockheed Martin Energy Research Corporation.

PROJECT SUMMARY

Advanced Industrial Materials (Aim) Program

PROJECT TITLE: Microwave Joining of SiC

PHASE: FY 1997

PERFORMING ORGANIZATION: FM Technologies, Inc.

PRINCIPAL INVESTIGATOR: Richard Silberglitt, (703-425-5111)

PHASE OBJECTIVE: To investigate use of polymer precursors to form SiC interlayers in situ during microwave joining of monolithic and composite SiC materials.

ULTIMATE OBJECTIVE: To identify and develop the most effective microwave joining methods for scale-up to large tube assemblies of silicon carbide that are required for industrial applications.

TECHNICAL APPROACH: In order to form a SiC interlayer in situ, polymer precursor decomposition in an inert environment is required. A microwave applicator capable of vacuum baking and back-filling with inert gas, and large enough to allow insertion of tubes up to 10 cm (~ 4") in diameter was designed, fabricated and tested. This applicator is being used to join monolithic SiC and continuous fiber-reinforced (CFCC) SiC/SiC composites using different polymer precursors.

PROGRESS: SiC/SiC CFCC and sintered SiC specimens were joined with SiC formed in situ from pyrolysis of either polysiloxane (silicone resin) or allyhydridopolycarbosilane (AHPCS). Maximum values of average shear strengths of microwave processed CFCC joints were comparable to joints produced with conventional heating. The effect of processing conditions on mechanical strength of sintered SiC specimens was investigated, and processing conditions selected for fabrication of 8" long, 1" OD sintered SiC joined specimens under the HiPHES project. These specimens are undergoing high temperature tensile testing at ORNL.

Patents:[-]	**Publications:**[-]	**Proceedings:**1
Books:[-]	**Presentations:**2	**Awards:**[-]

PROJECT SUMMARY (continued)

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Microwave Joining of SiC

ACCOMPLISHMENTS:

Licenses: [-] **Known Follow-on Product(s):**[-] **Industry Workshop:**[-]
Technology Transfer or Industrial Interaction: Customer-supplied materials include DuPont Lanxide Composites (DLC) CFCCs and polymer precursor. Joining methods developed under this contract will be used under the CFCC program (DLC/Foster Wheeler.) Discussions underway with INEX for application to reaction bonded SiC radiant burner tube assemblies. Presentation of joining results on CFCC SiC/SiC composites was planned for the CFCC Review Meeting and BES/CFCC Workshop in Lake Tahoe, CA on October 6-8, 1997.

CRITICAL ISSUES: Optimization of polymer precursors for strong and adherent SiC interlayers, as well as the development of applicators for cost-effective fabrication of industrial size specimens.

FUTURE PLANS: Joining of test specimens supplied by industrial collaborators that properly simulate the components to be fabricated, followed by tests conducted by the industrial collaborators that simulate performance environments, and then scale-up to fabricate prototype components, and prototype testing.

POTENTIAL PAYOFF: The markets for SiC radiant burner tubes and heat exchangers have been estimated to be greater than 100 million dollars annually. These components cannot currently be cost-effectively fabricated in lengths and shapes required. Microwave joining could be an enabling technology by allowing fabrication through the joining of several small, simply shaped pieces. Development of a transportable microwave applicator would provide the capability for on-site fabrication and repair of ceramic tube assemblies, which would further enhance the cost effectiveness of the approach.

ESTIMATED ENERGY SAVINGS: Use of SiC tube heat exchanger in externally fired combined cycle coal power plants is projected to produce a 20% increase in thermal efficiency, together with a 20% reduction in CO_2 emissions and a 90% reduction in SO_x emissions.

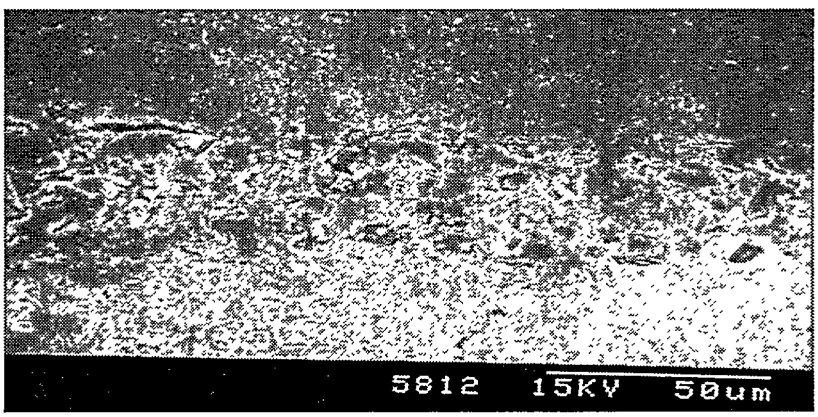
SIGNIFICANT ACCOMPLISHMENT

Advanced Industrial Materials (AIM) Program

Microwave Joining of Sintered SiC with Pure SiC Interlayer Formed In Situ from Polymer Precursor

ISSUE: Sintered SiC materials can increase service temperatures of turbines, heat exchangers and other energy system components, but are not commercially available in the sizes and shapes needed for many of these applications. Joining methods are needed to cost-effectively fabricate industrial components such as large tube assemblies.

RESULTS: Microwave heating was used to join pairs of sintered SiC disks via decomposition of a polymeric precursor to form a SiC interlayer in situ (see figure below). The interlayer contains pure SiC which under microscopic evaluation looks identical to the material that was joined. This same method was then used to fabricate an eight-inch-long, one-inch diameter tube section that is undergoing high temperature testing.



ENERGY EFFICIENCY: Improved energy efficiency will occur through the reduction of feedstock consumption and decoke fuel and steam requirements with an advanced ethylene production process using a high pressure SiC heat exchanges 63.9 trillion BTUs per year.

Research performed at FM Technologies, Inc.-George Mason University, and at Los Alamos National Laboratory, sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Industrial Materials Program.

PROJECT SUMMARY

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Selective Inorganic Thin Films

PHASE: FY 1997

PERFORMING ORGANIZATIONS: Sandia National Laboratories, University of New Mexico, and University of Western Australia

PRINCIPAL INVESTIGATOR: Tina M. Nenoff, Dept. 1845, MS 0710, Sandia National Laboratories, Phone: (505-844-0340), Fax: (505-845-9500)

PHASE OBJECTIVE: The objectives are to 1.) study inorganic membranes capable of separating light gases and mixtures of arene isomers via molecular sieving, and 2.) investigate methods for crystallizing zeolite films from sol-gel, clay, and metal precursor films on porous substrates.

ULTIMATE OBJECTIVE: The focus will be on developing new class of inorganic membranes for light gas and arene isomers separation and possibly catalysis, using this technology to improve upon separation efficiencies and thermal stability currently available with polymer or oxide membranes, emphasizing application to petroleum and natural gas refining, and transferring the technology to industry.

TECHNICAL APPROACH: The approach is to 1.) use computational modeling to select appropriate microporous phases for desired separation; nucleate and crystallize zeolite and microporous zinc phosphate phases from sol-gel, clay, and metallic films, using porous filters and gas membranes as supports for these films; and 3.) investigate novel microporous zinc phosphates as molecular sieves.

PROGRESS: Methods for synthesizing and caulking Zeolite A and MFI zeolite membranes were studied extensively. A CRADA was signed with Amoco Chemical Co. to study the feasibility of using shape selective molecular sieve membranes to enrich *p*-xylene from mixtures of the isomers. Cesium zinc oxide phosphate ($CsZn_2OPO_4$) molecular sieve membranes on porous zinc oxide wafers were made.

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Selective Inorganic Thin Films

These membranes are permeable to water and helium, but not to gases with larger kinetic diameters. Thin films were also made of zinc oxide phosphates with the larger-pore $M_3Zn_4(PO_4)_3$ structure type. We discovered a methylammonium zinc oxide phosphate phase that remains microporous after the water and template are removed by calcination; this was synthesized as a thin film as well.

ACCOMPLISHMENTS:

Publications: 3 **Presentations:** 2 **Disclosures:** 0

CRITICAL ISSUES: Issues include: 1.) improving permeabilities of zinc phosphate films by reducing film thickness while maintaining complete substrate coverage, and 2.) improving techniques for eliminating intercrystalline gaps and porosity in MFI zeolite films via the caulking process.

FUTURE PLANS: Plans include: 1.) continue studying alkali zinc phosphate membranes for hydrogen recovery, organic zinc phosphates for natural gas purification, 2.) continue to improve methods for caulking MFI zeolite membranes, and 3.) model permeation of mixtures of xylene isomers through known zeolite phases, 4.) synthesize membranes containing phases with optimum selectivity, and 5) measure relative permeabilities of xylenes through these membranes.

POTENTIAL PAYOFF: The petroleum and natural gas refining industries would significantly benefit from high permeability molecular sieve films capable of separating light, fixed gases, particularly if the membranes can be used at high temperatures. The alkali metal zinc phosphates that have been synthesized as membranes are stable up to 700°C, which is compatible with hydrogen recovery. With sufficiently high permeability and low unit area cost, energy savings of several quad/yr could be achieved when all feasible applications of inorganic membranes are considered. We have negotiated and begun a CRADA with Amoco Chemical Co. to study the feasibility of using shape selective molecular sieve membranes to enrich *p*-xylene from mixtures of the isomers. If these membranes eliminate the first crystallization step in *p*-xylene production worldwide, approximately 0.070 quad/yr will be saved, principally in the form of decreased natural gas consumption.

SIGNIFICANT ACCOMPLISHMENT

Advanced Industrial Materials (AIM) Program

Membranes for Xylene Separations

On June 16, 1997, Sandia and Amoco Corporation signed a 3 year CRADA entitled "Advanced Materials for Reducing Energy Consumption and Mnaufacturing Costs in the Chemicals and Petroleum Refining Industries." The goal of this research is to demonstrate the technical, economic, and commercial potential of using composite inorganic membranes (or films) for separation of predominantly aromatic hydrocarbons.

CRADA Tasks:

Phase I

1. Modeling permeation of mixtures of predominantly aromatic hydrocarbons
2. Synthesis of leak-free supported inorganic composite silica/zeolite films
3. Characterization of composite films

Phase II

1. Refinement of permeation modeling to guide optimization work
2. Synthesis of zeolite films on porous supports
3. Characterization of zeolite films
4. Integration of catalysis and separation

Phase III

1. Determine preferred substrate for commercial module
2. Scale up zeolite membrane synthesis
3. Characterization of zeolite films
4. Evaluation of long-term stability and performance of membranes
5. Design commercially scaleable pilot plant module

Research sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Industrial Materials Program, under contract DE-AC04-95AL85000 and at the Amoco Corporation. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the U.S. Department of Energy.

POLYMERS

PROJECT SUMMARY

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Industrial Applications of Conducting Polymers: Polymer Electrolyte Electrochemical Reactors of Lowered Energy Consumption

PHASE: FY-1997

PERFORMING ORGANIZATION: Los Alamos National Laboratory

PRINCIPAL INVESTIGATOR: Shimshon Gottesfeld, (505-667-0853)

PHASE OBJECTIVES: Reach stable performance for, at least, one week with chlor-alkali electrochemical reactor (ECR) employing an oxygen cathode, exhibiting cell voltage lower by, at least, 30% vs. present day industrial chlor-alkali ECR at a current density near 300 A/ft^2 .

ULTIMATE OBJECTIVES: Develop and test electrochemical reactor (ECR) for the chlor alkali industry, based on a polymer membrane/electrode assembly (MEA) and an oxygen or air electrode, demonstrating energy consumption lower by 40% vs. the present day industrial chlor-alkali ECR at same product throughput and at current efficiency >90%.

TECHNICAL APPROACH: Novel ECR configuration, based on effective oxygen cathodes developed at LANL, are assembled and tested at LANL. Optimization is based on detailed analysis of losses in the cell and on modification of cell elements following identification of sources of loss. Collaboration with Dow Chemical ensures meaningful comparison with present day technology by testing at LANL with hardware provided in part by Dow, by receiving technical information on various aspects of chlor-alkali cells and by selecting specific experimental routes based on techno-economic evaluation performed by Dow Chemical.

PROGRESS: (1) Using cell hardware employed at LANL in fuel cell work, operation of a chlor alkali cell was demonstrated at target voltage level. (2) A standard, 58 cm^2 chlor-alkali cell, of the type employed routinely by Dow Chemical for lab testing was adapted to test oxygen cathode based ECRs at LANL. An ECR lab testing system was rebuilt, accommodating continuous operation of 3 test cells in parallel and engineering controls including water make-up to brine and caustic, a chlorine scrubber system to safely convert chlorine gas to "bleach" for disposal, and brine purification system.

PROJECT SUMMARY (Continued)

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Industrial Applications of Conducting Polymers: Polymer Electrolyte Electrochemical Reactors of Lowered Energy Consumption

ACCOMPLISHMENTS: Accomplishments include: 1.) Utilizing fuel cell hardware, we demonstrated operation of a chlor alkali cell at target voltage level (0.4 A/cm^2 at 2.0V) with a LANL oxygen cathode and a commercial chlor-alkali membrane of high current efficiency (>95%), and 2.) Lowering cell voltage for a chlor-alkali ECR from the ordinary level of 3.2 V to 2.0 V at constant, commercial throughput level, corresponds to savings of 38% in electric energy invested per unit weight of product (and 38% lowering in CO_2 emissions).

CRITICAL ISSUES: The most critical issue in this project is demonstration of stable cell performance in operation in the energy saving mode based on an oxygen cathode. Performance stability depends, in turn, on optimization of cathode structure and of cell operation conditions.

FUTURE PLANS: Following the demonstration of stable performance in operation with an oxygen cathode, the ECR will be further optimized to achieve maximized energy savings (minimized voltage), and high current efficiency. This will entail overall cell optimization, in addition to, primarily, cathode structure optimization. Pilot plant scale testing by Dow Chemical could follow if stable performance is demonstrated on lab scale.

POTENTIAL PAYOFF: Payoffs and energy savings include 1.) potential to save up to 50% of the electric energy consumed by an industry which uses 2% of the total electric power generated in the US; 2.) Introduction of new chlor-alkali reactor technology giving US industry a competitive edge, and 3.) Cutting CO_2 emissions by up to 50% in and industry which is major consumer of electric power.

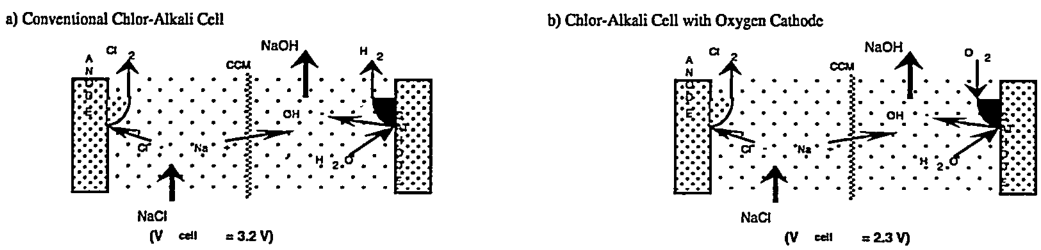
SIGNIFICANT ACCOMPLISHMENT

Advanced Industrial Materials (AIM) Program

Energy Efficient Electrochemical Reactors (ECRs)

ISSUE: The chlor-alkali industry consumes 2% of the total electric power generated in the US. The products at the cathode are NaOH and hydrogen gas, and gaseous chlorine is generated at the anode. Cell voltage directly reflects the electric energy consumption per ton of chlorine (or caustic) product. Lowering of the cell voltage is an important energy savings target for this particular industrial process, as well as several other industrial processes involving electrochemical oxidations.

RESULTS: The figure shows on the left the conventional configuration of an ECR used in the chlor-alkali industry, generating chlorine and caustic soda by electrolysis of sodium chloride brine. The cell voltage required at typical production rate of 300 Amperes/ft^2 is 3.2-3.3 V. If the hydrogen evolving cathode were replaced by an oxygen consuming cathode (case b), the voltage of the cell could be reduced, in principle, by about 0.9V. We have developed in recent years at LANL advanced oxygen/air electrodes, and adaptation of elements of our approach for chlor-alkali ECRs seemed possible. Results indicate a 38% energy savings per unit product weight. Subsequently, an industrial cell configuration was adapted with assistance from Dow Chemical.



Scheme for reducing energy consumption in a chlor-alkali ECR, using an effective oxygen cathode and a cation conducting polymeric membrane (CCM) as basis for an effective membrane/electrode assembly.

ENERGY EFFICIENCY: (1) Potential to save up to 50% of the electric energy consumed by an industry which uses 2% of the total electric power generated in the US. (and corresponding lowering in CO_2 emissions).

Research performed at Los Alamos National Laboratory (LANL), sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Industrial Materials Program.

PROJECT SUMMARY

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Polymerization and Processing of Polymers In Magnetic Fields

PHASE: FY 1997 **COMPLETION DATE:**

PERFORMING ORGANIZATION(S): Los Alamos National Laboratory, MST-7

PRINCIPAL INVESTIGATOR(S): Mark E. Smith, (505-665-6858)

PHASE OBJECTIVE: Produce large scale parts to demonstrate feasibility of economic development

ULTIMATE OBJECTIVE: Produce and develop the processing methodologies of magnetic orientation of liquid crystalline thermosets for lightweight materials applications.

TECHNICAL APPROACH: The process has been demonstrated to produce a 50% increase in tensile properties through use of a standard grade MRI magnet. Further improvements on tensile properties and reduction of process time are being explored.

PROGRESS: Plaques of 8" x 8" x 0.125" were successfully produced in the past year. The current focus is to increase the material thickness, and subsequently the overall volume of shape.

Patents: Application # 08/608,343	**Publications: 2**	**Proceedings: 2**
Books: -	**Presentations: 4**	**Awards: 1**

ACCOMPLISHMENTS: production of prototype scale industrial part

Licenses:
Known Follow-on Product(s):
Industry Workshop:
Technology Transfer or Industrial Interaction: Work is performed under a CRADA with the Dow Chemical Company.

PROJECT SUMMARY (continued)

Advanced Industrial Materials (AIM) Program

PROJECT TITLE: Polymerization and Processing of Polymers in Magnetic Fields

CRITICAL ISSUES: None.

FUTURE PLANS: Determine economic feasibility of the processing technique.

POTENTIAL PAYOFF: Lightweight materials for structural applications

ESTIMATED ENERGY SAVINGS: Significant energy savings exist through the magnetic processing technique presented for the production of high strength, lightweight structural components. Additional energy savings would result from the use of self-reinforcing liquid crystalline resins with faster cure times, as the faster time requires less energy per part in a continuous production scheme. Additionally, liquid crystalline materials do not require reinforcing fillers, mechanical compounding or excessive handling in a manufacturing process.

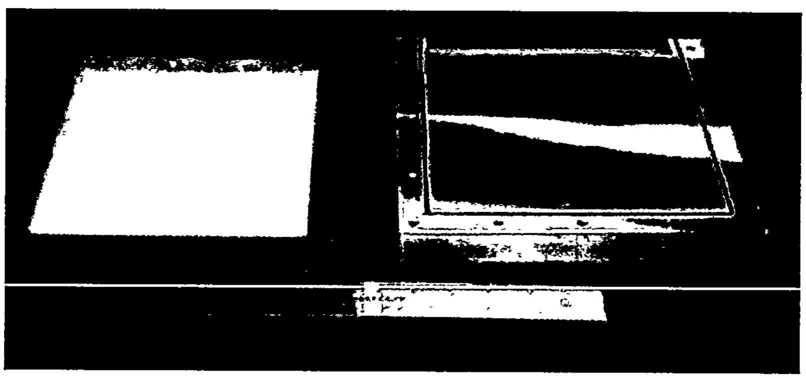
SIGNIFICANT ACCOMPLISHMENT

Advanced Industrial Materials (AIM) Program

Polymerization and Processing of Polymers In Magnetic Fields

ISSUE: The ability and economic feasibility of using magnetic fields in the processing of a novel structure epoxy to increase the tensile strength and produce lightweight structural materials.

RESULTS: FY1997 results were excellent with respect to scale-up of the process from small (2.5"x 1.75"x 0.125") plaques to a large plaque part (8"x 8"x 0.125"). This was achieved through the use of a conventional magnet typically used for Magnetic Resonance Imaging (MRI) in the medical field.



Plaque and mold face employed in magnetic field processing. The ruler shown is a standard 12" length. In this sample, the orientation of the material is horizontal (left-right as pictured).

ENERGY EFFICIENCY: Significant energy savings exist through the magnetic processing technique presented for the production of high strength, lightweight structural components. Additional energy savings would result from the use of self-reinforcing liquid crystalline resins with faster cure times, as the faster time requires less energy per part in a continuous production scheme. Additionally, liquid crystalline materials do not require reinforcing fillers, mechanical compounding or excessive handling in a manufacturing process.

Research performed at the Los Alamos National Laboratory, sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Industrial Materials Program.

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