ADVANCED INTERMETALLICS/METALS AND COMPOSITES

PROJECT SUMMARY

ADVANCED INDUSTRIAL MATERIALS (AIM) PROGRAM

PROJECT TITLE: Advanced Ordered Intermetallic Alloy Development

PHASE: FY 1998, Materials Capability Development **COMPLETION DATE:**

PERFORMING ORGANIZATION: Oak Ridge National Laboratory

PRINCIPLE INVESTIGATORS: C. T. Liu (423-574-4459) and Lee M. Pike (423-574-4343)

PHASE OBJECTIVE: To develop nickel silicide alloys that exhibit good corrosion resistance, ease of fabricability, and good low- and high-temperature strength and ductility for chemical and other vision industrial use.

ULTIMATE OBJECTIVES: (1) To develop low-density, high-strength, corrosion-resistant intermetallic alloys for structural applications in chemical and other vision industry systems and (2) to help the U.S. material industry to compete in the world market.

TECHNICAL APPROACH: Both macroalloying and microalloying processes are employed to improve metallurgical and mechanical properties of nickel silicide intermetallic alloys. The selection of alloying additions will be based on physical metallurgy principles, structural maps, and empirical correlation. Emphasis will be placed on improving ductility and toughness as well as strength and corrosion resistance at ambient and elevated temperatures through composition adjustment, material processing, and structural control.

PROGRESS: A series of eight alloys based on Ni_3Si was produced, fabricated, and evaluated at room and elevated temperatures. Cr, Mo, and other alloying elements are effective in improving tensile strength at temperatures <600°C and corrosion resistance at boiling sulfuric acids. The alloys exhibit tensile strength much superior to advanced nickel aluminide alloys at intermediate temperatures. The silicide alloys showed environmental embrittlement at elevated temperatures in oxidizing environments.

Invention Disclosures: None. **Publications:** None. **Proceedings:** None.

Presentations: 2

ACCOMPLISHMENTS:

Licenses: Discussions with several U.S. industries for potential technology transfer.

Known Follow-On Product: Anticipated use in various industries.

PROJECT SUMMARY (continuted)

PROJECT TITLE: Advanced Ordered Intermetallic Alloy Development

OTHER SUCCESSFUL TECHNOLOGY TRANSFER ACTIVITIES AS EVIDENCE OF INDUSTRY INTEREST: At the DOE Workshop on Ordered Intermetallic Alloys held in Atlanta, Georgia, in June 1996, both Cummins Engine Company and United Defense expressed interest in nickel silicides for petrochemical and chemical industries. Haynes International is interested in Ni_3Si alloys with improved ductility and fabricability at elevated temperatures.

CRITICAL ISSUES: (1) To alleviate oxygen-induced environmental embrittlement at elevated temperatures and (2) to improve hot fabricability of nickel silicide alloys.

FUTURE PLANS: (1) To increase the tensile ductility and hot fabricability of Ni_3Si alloys by careful control of Si and other major alloys elements and (2) to initiate technology transfer of nickel silicide alloys with improved properties.

POTENTIAL PAYOFF: There are two major problems with intermetallic alloys: poor ductility at ambient temperatures and limited corrosion and creep resistance at elevated temperatures. Overcoming these problems would result in the development of a whole new class of ordered intermetallic alloys that are superior to those of existing superalloys. The use of ductile, lightweight intermetallic alloys would improve the thermal efficiency and system performance of energy-conversion systems, resulting in substantial energy savings.

SIGNIFICANT ACCOMPLISHMENT

ADVANCED INDUSTRIAL MATERIALS (AIM) PROGRAM

ORNL 99-03318A/tco

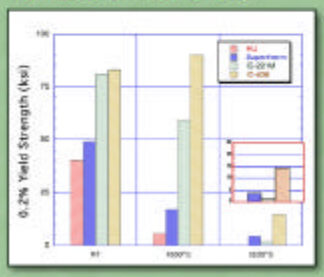
Advanced Intermetallic Alloys

The energy utilization and efficiency of industrial processes can be improved by the utilization of high temperature corrosion resistant advanced intermetallic alloys

Objective:

Provide alloy design, processing, fabrication, and materials property capabilities to develop and enable the use of advanced intermetallic alloys in high performance applications

Advanced Ni_3Al alloys



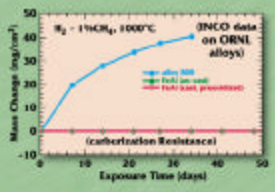
The chemical composition modification of IC438 increases the maximum use temperature of IC221M from 1100° to 1200°C

Alloy	Composition, wt%					
	Ni	Al	Cr	Mo	Zr	B
IC221M	Balance	8.0	7.7	1.45	1.5	0.080
IC438	Balance	8.1	4.3	7.0	0.13	0.080

Improved high temperature mechanical properties of Ni_3Al enables higher thermal efficiencies in industrial processes

FeAl alloys

Centrifugally cast FeAl tube



Superior carburization resistance of FeAl alloys will enable more efficient chemical processing

Ni_3Si alloys

Multi-pass welds made in cast bars confirm that some Ni_3Si alloys have excellent weldability

Ni_3Si alloys with Nb and Ti have exceptional corrosion resistance in boiling sulphuric acid

	Corrosion rate, mils/year	
Composition, at%	60% H_2SO_4	77% H_2SO_4
Ni-18.8Si	56.68	93
Ni-18.9Si-1.2Nb	0.68	1
Ni-18.9Si-3.2Ti	0.68	1

Development of castable and weldable Ni_3Si alloys will enable fabrication of complex components for use in chemical process systems

Partners

alloy engineering & casting company

DOW

DERALOY TECHNOLOGIES, INC.

Ford

INCO ALLOYS INTERNATIONAL

ornl

SHENANGO

United Defense

Research and Development Sponsored by

Advanced Industrial Materials (AIM) Program,
Office of Industrial Technologies, Energy Efficiency and Renewable Energy;
and also based on research and development funded by Energy Research,
U.S. Department of Energy

PROJECT SUMMARY

ADVANCED INDUSTRIAL MATERIALS (AIM) PROGRAM

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

PHASE: FY 1998 **COMPLETION DATE:**

PERFORMING ORGANIZATION: Oak Ridge National Laboratory (ORNL)

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

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

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

PROGRESS: New cast FeAl alloys show up to 5% ductility and Charpy impact toughness of 14 to 20 J or more in air at room temperature, together with yield strength of 400 MPa at 700°C. Minor alloying can improve the creep-resistance at 700 to 800°C. Cast FeAl alloys have strength comparable to cast heat-resistant austenitic alloys like HU and HK at temperatures of 1000 to 1100°C. A postweld heat treatment was found that enables the cast FeAl alloys to be welded without any cold cracking.

Patents: 2	**Publications:** 7	**Proceedings:** 9
Books: 1	**Presentations:** 17	**Awards:** 2

PROJECT SUMMARY (continued)

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

ACCOMPLISHMENTS: FMC, Inc., cast grate bars for calcination of phosphate ores and then tested them in-service for over 6 months at 1000°C. Dow Chemical tested FeAl for coking resistance in ethylene furnace applications.

Licenses: 1

Known Follow-on Products: Burner nozzles, radiant heating tubes, tubes or pipes in chemical or petrochemical industry, and steel rolls (molten zinc).

Industry Workshop: None.

Technology Transfer or Industrial Interaction: FeAl materials are in-test for several different industrial (chemical, petrochemical or metals-processing) applications. Duraloy has expressed interest in technology transfer for centrifugally-cast tube applications, and other centrifugal casters have also expressed interest in FeAl for several different applications.

SIGNIFICANT ACCOMPLISHMENT

ADVANCED INDUSTRIAL MATERIALS (AIM) PROGRAM

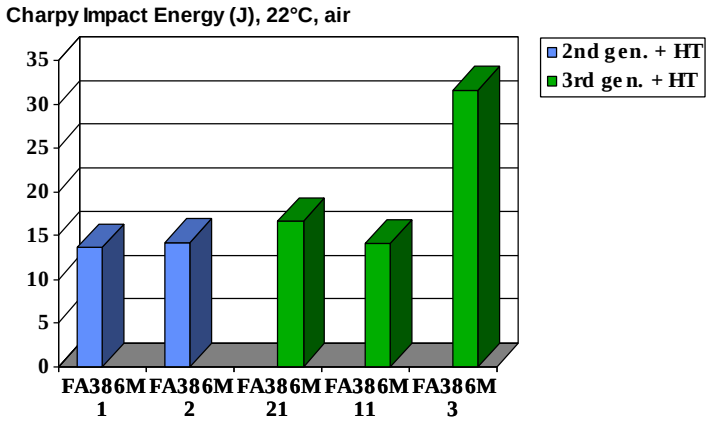
Cast FeAl Alloys Show Improved Creep Strength, Impact Toughness, and Weldability

ISSUE: FeAl iron-aluminides have outstanding oxidation, sulfidation, and carburization resistance at 1000°C and above due to protective Al_2O_3 scale formation. But, do FeAl alloys have the strength to be used at such temperatures? Do they have enough ductility and toughness at room temperature to allow them to survive in typical industrial applications?

RESULTS: New cast FeAl alloys (Fe– 36 to 40 at. % Al) designed for improved high-temperature strength and room-temperature ductility were developed that showed good tensile strength at 700 to 1000°C, and creep strength at 700 to 800°C. Tensile strength is comparable to type 310 austentic stainless steel or to cast HU and HK austenitic stainless alloys. Cast FeAl alloys were found to have up to 4 to 5% ductility in air at room temperature, and about 10 to 15 J Charpy impact toughness, or more. Such cast FeAl alloys were found to be weldable without any cold cracking after appropriate heat treatment, a significant accomplishment.

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 radically lower metal wastage of "super" corrosion-resistant FeAl in aggressive high-temperature enviroments will save the energy used to produce and process conventional Fe-Cr-Ni steels, stainless steels, and heat/corrosion-resistant alloys for such applications. It also will save the wasted time and energy (and hence cost) associated with downtime and lost production.

Microstructure control and minor alloying additions improve Charpy-impact energy of cast FeAl Alloys.



Fe-(36-38)Al-0.2Mo-0.05Zr-0.13C (at.%)

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-AC05-96OR22464 with Lockheed-Martin Energy Research Corporation.

PROJECT SUMMARY

ADVANCED INDUSTRIAL MATERIALS (AIM) PROGRAM

PROJECT TITLE: High-Density Infrared Processing of Materials

PHASE: FY 1998 **COMPLETION DATE:**

PERFORMING ORGANIZATION: Oak Ridge National Laboratory (ORNL)

PRINCIPAL INVESTIGATORS: C. A. Blue (423-574-4351) and V. K. Sikka (423-574-4351)

PHASE OBJECTIVE: To develop an advanced coating technique to implement advanced materials.

ULTIMATE OBJECTIVE: To develop and implement advanced materials and processing techniques utilizing infrared.

TECHNICAL APPROACH: Infrared heating has many advantages over commonly used heating techniques. Infrared heating provides (1) an inherently clean noncontact heating method; (2) high heat fluxes resulting in heating rates up to 5000°C/s, materials dependent, due to the fact that only the sample is heated to the desired temperature; (3) provides heating only to the part (i.e., low thermal mass); (4) fast cooling rates due to the "cold wall" nature of the process in which only the sample is heated; (5) high heat fluxes, which can be delivered unidirectionally over large areas; and (6) a precise process that essentially is shape independent, utilizing proper furnace designs.

Thus, infrared provides a versatile and flexible answer to heat-transfer problems throughout the industrial spectrum. Industrial heat treating, joining, and coating problems have been identified. The appropriate experimentation has been accomplished in order to explore the feasibility of a potential infrared system. Additional experimentation has been explored when appropriate through funding developed in the initial phase. The technology has been demonstrated to be an invaluable tool for advanced joining, heat treating, polymer joining, and coating.

PROGRESS: ORNL has developed a high-density infrared (HDI) transient-liquid coating (TLC) process to produce wear- and corrosion-resistant coatings on a multitude of surfaces. The HDI TLC process combines infrared with power densities of up to 3.5 kW/cm^2 with a room-temperature spray process to form wear- and/or corrosion-resistant coatings in seconds to minutes. This process has been demonstrated with Cr_2C_3- and WC-reinforced coatings with nickel-based binders. Volume fractions as high as 70% have been accomplished with coating thicknesses of 10 μm to 2 mm.

Patents/Disclosures: 9 **Publications:** None. **Proceedings:** None.

Presentations: 5

PROJECT SUMMARY (continued)

PROJECT TITLE: High-Density Infrared Processing of Materials

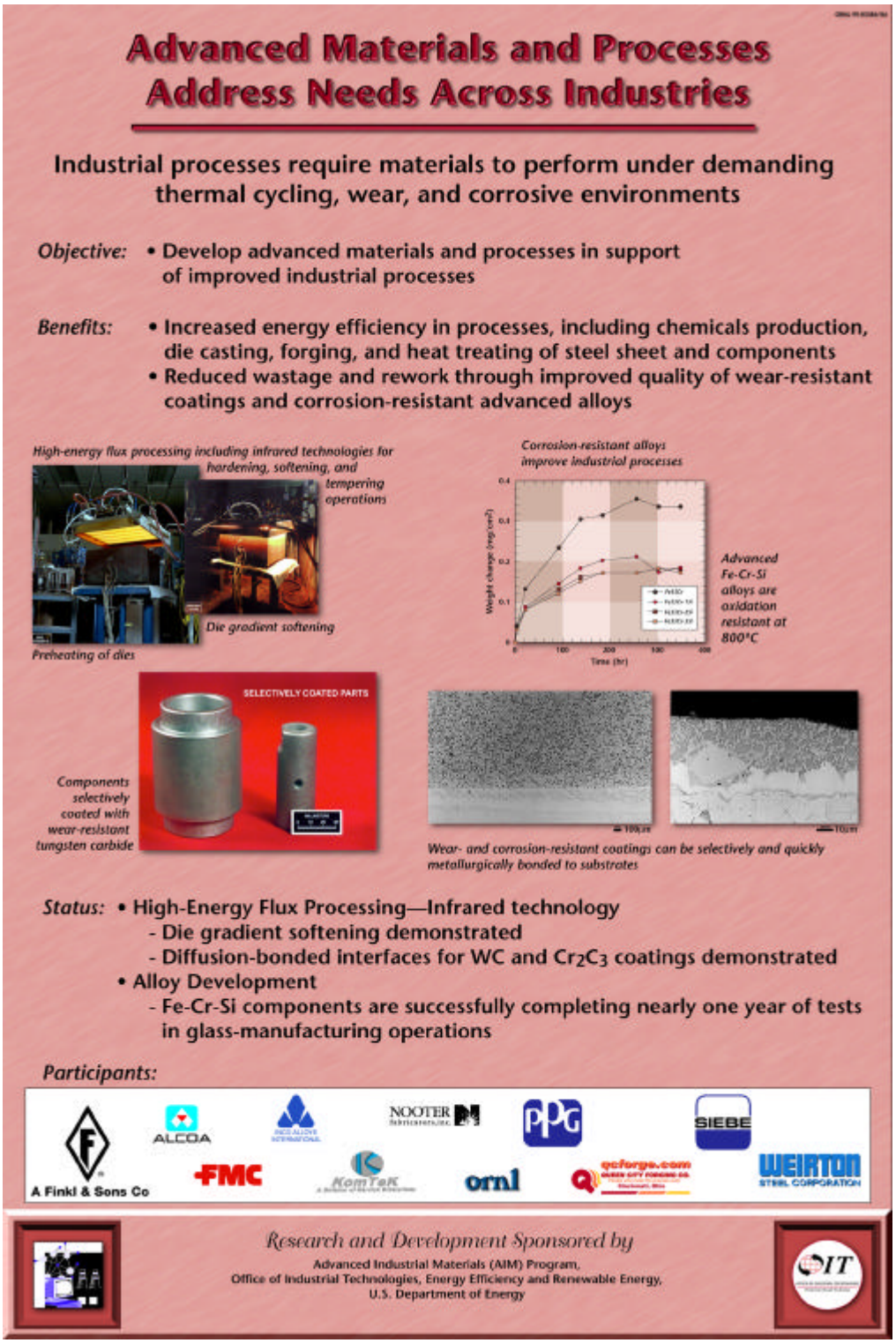
CRITICAL ISSUES: The survivability of the infrared system in an industrial environment.

FUTURE PLANS: Continue applying the HDI TLC process to a variety of industrial parts in need of wear and corrosion resistance.

POTENTIAL PAYOFF: The potential payoff in the HDI TLC lies in the large energy and money savings observed through less downtime on equipment and through extended life on components.

SIGNIFICANT ACCOMPLISHMENT

Advanced Industrial Materials (AIM) Program



PROJECT SUMMARY

ADVANCED INDUSTRIAL MATERIALS (AIM) PROGRAM

PROJECT TITLE: Intermetallic Alloy Development and Technology Transfer

PHASE: FY 1998 **COMPLETION DATE:**

PERFORMING ORGANIZATION: Oak Ridge National Laboratory (ORNL)

PRINCIPAL INVESTIGATORS: V. K. Sikka (423-574-5112) and M. L. Santella (423-574-4805)

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 the excellent oxidation and carburization resistances and the higher strength of nickel aluminides for a broad range of manufacturing-related industry applications. The applications identified to date include materials of construction for chemical processes, furnaces, hot-pressing or forging dies, cast heating elements, and burners for gas-fired furnaces.

TECHNICAL APPROACH: The FeAl-based iron aluminides have beneficial properties including (1) oxidation resistance, (2) sulfidation resistance, (3) carburization resistance, (4) lower cost, (5) lower density, and (6) molten salt resistance. 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.

PROGRESS: Worked with all five licensees [Alcon Industries, Alloy Engineering & Casting (AEC) Company, Sandusky International, Stoody Company, and United Defense LP] in addressing production, welding, and application-related issues. Thermal expansion and selected tensile, creep, and fatigue data were developed on the cast eutectic-free alloy IC-438. The specific data generated on IC-438 assisted in the identification and design for the new applications. Several coupons of IC-438 alloy were shipped to potential users. The melting and centrifugal casting trials on FeAl alloy were carried out at AEC. Several coupons of FeAl were supplied to the chemical industry for developing corrosion data. Several potential users were visited. An HVOF-coated component (air deflector) was installed into service.

Patents: 2 **Publications:** 3 **Proceedings:** 5

Books: None. **Presentations:** 14 **Awards:** None.

PROJECT SUMMARY (continued)

PROJECT TITLE: Intermetallic Alloy Development and Technology Transfer

ACCOMPLISHMENTS:

Licenses: Three new licenses were signed. These were for sand castings with Alcon Industries, weld wire with Stoody Company, and weld wire with Polymet Corporation.

Other Successful Technology Transfer Activities as Evidence of Industry Interest:

1. Six batch trays and 68 pusher furnace assemblies continued operation in carburizing furnaces at Delphi Saginaw. One pusher tray was removed and characterized for its microstructure. The data were reported in CRADA report No. ORNL92-0076.
2. The number of rolls at Bethlehem Steel-Burns Harbor Plant in Indiana were increased from 2 to 20 during the last year. Two of the rolls have successfully completed 4.5 years of service.
3. The first set of trays in a carburizing furnace at The Timken Company resulted in 2.7 times longer life than HT trays. Additional trays are in operation.
4. Fifty trays of IC-221M are operating in carburizing furnaces at Ford Motor Company. In this application, trays are quenched in a salt bath as opposed to an oil bath for Delphi and Timken applications.
5. An air deflector, HVOF coated with FeAl alloys, was put into application and testing in a burner using high-sulfur and vanadium fuel.

CRITICAL ISSUES: Additional test data on IC-438, the weldability of IC-438, and identification of hot working parameters for a nickel-aluminide composition such as IC-218LZr are still critical issues for Ni_3Al-based alloys. The commercial melting and casting, mechanical properties of commercial heats, and weldability of FeAl are the critical issues for FeAl-based alloys.

FUTURE PLANS: Continue Ni_3Al- and FeAl-based 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 will yield major benefits of energy savings, and U.S. industry will gain the competitive position in world markets. Industries benefiting from nickel aluminide technology are automotive, steel, chemical, heat treating, forging, and manufacturing.

SIGNIFICANT ACCOMPLISHMENT

ADVANCED INDUSTRIAL MATERIALS (AIM) PROGRAM

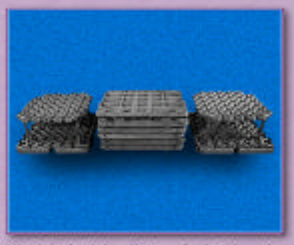
ORNL 99-03319A/tcc

Intermetallic Alloy Development for the Steel Industry

Ni_3Al alloys have exceptional high temperature strength and corrosion resistance

Objective: Enable increased energy efficiency and process efficiencies in the steel industry through the application of intermetallic alloys

Benefits:
- energy savings of up to 32 T Btu by 2010
- improved productivity and product quality
- improved environmental benefits



Ni_3Al Carburizing fixtures for batch and continuous heat treating furnaces –3 to 5 times longer life

View of heat treating furnace at 160" plate mill in Bethlehem Steel Burns Harbor Plant

Nickel aluminide rolls show no sign of blistering

Completed radiant burner assembly prior to dye penetrant inspection

Tubes were successfully welded

Status:
- 21 rolls under test at Bethlehem steel
- testing in various steel plants are planned
- radiant burner tubes are being developed and being tested at Timken and Ford

Participants:

R&D Organization — ornl

Licensees/Producers — alloy engineering & casting company; Sandusky International; Thermadyne Stoody; United Defense; Polymet; Alcon

Industry Applications — Ford; FMC; TIMKEN; Bethlehem Steel; Delphi; Caterpillar

Research and Development Sponsored by

Steel Vision, and Advanced Industrial Materials (AIM) Program, Office of Industrial Technologies, Energy Efficiency and Renewable Energy; and also based on research and development funded by Energy Research, U.S. Department of Energy

PROJECT SUMMARY

ADVANCED INDUSTRIAL MATERIALS (AIM) PROGRAM

PROJECT TITLE: Materials for the Pulp and Paper Industry

PHASE: FY 1998 **COMPLETION DATE:**

PERFORMING ORGANIZATION: Oak Ridge National Laboratory

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

PHASE OBJECTIVES: (1) Identify environments that can cause stress-corrosion cracking of 304L stainless steel, (2) determine the effect of thermal cycling on cracking, (3) continue the characterization of cracked tubes, (4) measure temperatures and strains in floor tubes, (5) measure residual stresses in unexposed and exposed 304L/SA210 composite tubes, (6) predict the stresses in recovery-boiler floor tubes during normal operation and during thermal transients, and (7) determine whether the microstructure of an exposed tube has features characteristic of thermal cycling.

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

TECHNICAL APPROACH: Use standard metallographic examination techniques coupled with advanced analytical techniques to characterize unexposed and exposed cracked tubes. Use neutron and X-ray diffraction to measure the residual stresses in composite tubes, and use finite-element modeling to predict stresses under operating conditions. Conduct laboratory studies to identify environments that can cause stress-corrosion cracking and to determine the effects of thermal cycling. Determine the frequency and magnitude of thermal cycles.

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

Patents: None. **Publications:** 7* **Proceedings:** None.

Books: None. **Presentations:** 15** **Awards:** 1***

---

*Five papers have been published in conference proceedings, and two papers have been published in the open literature; several manuscripts have been submitted for publication.

**Does not include presentations made at triannual program review meetings.

*** Received the Best Paper Award at the 9th International Symposium on Corrosion in the Pulp and Paper Industry.

PROJECT SUMMARY (continued)

PROJECT TITLE: Materials for the Pulp and Paper Industry

ACCOMPLISHMENTS:

Licenses: None. **Known Follow-On Product(s):** None.

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.

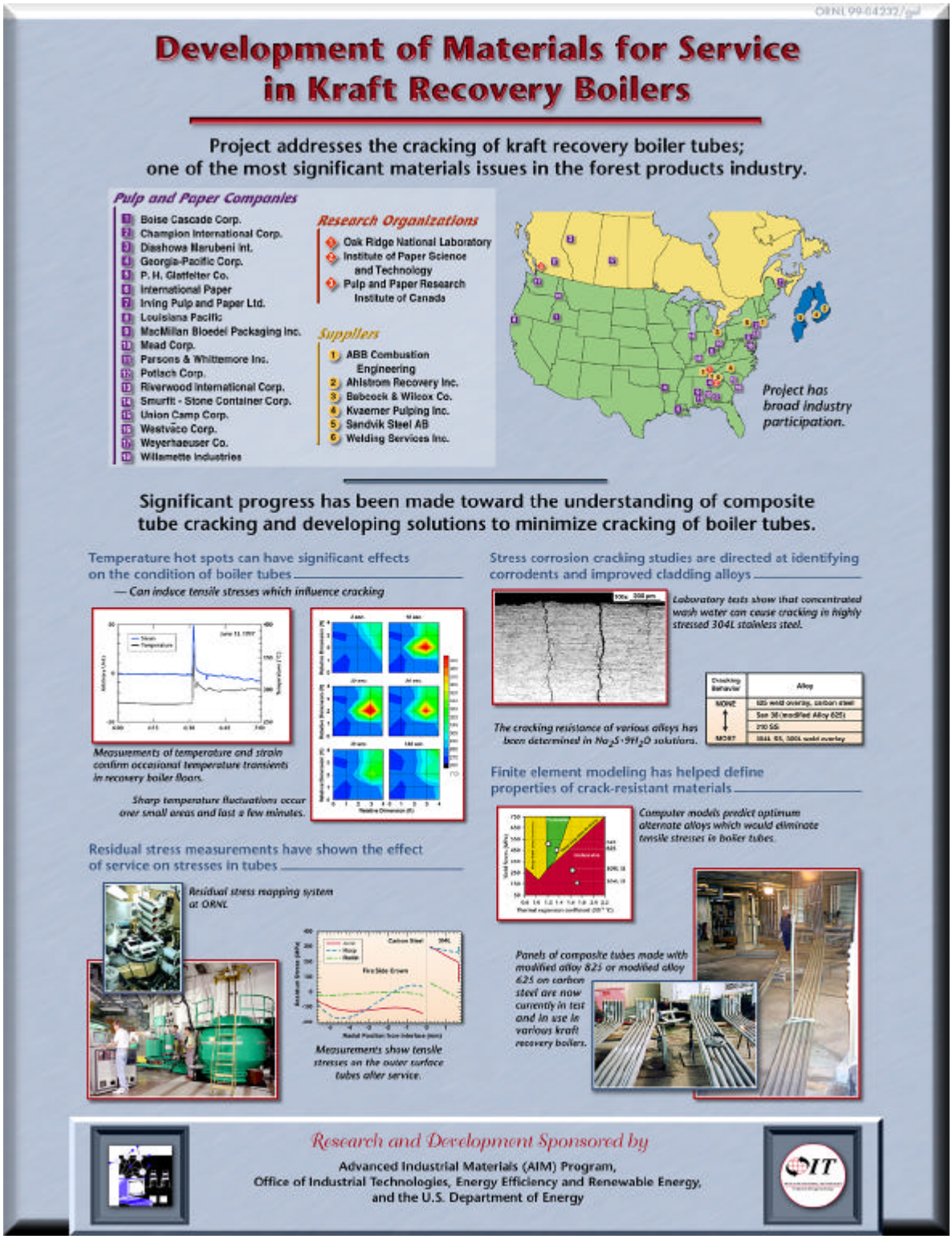
FUTURE PLANS: Continue work to identify corrodent responsible for stress-corrosion cracking; characterize cracked tubes provided by paper companies; measure and model the stresses developed in composite tubes with some emphasis on tubes made from alternate alloys being considered; and work with paper 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.

ESTIMATED ENERGY SAVINGS:

SIGNIFICANT ACCOMPLISHMENT

ADVANCED INDUSTRIAL MATERIALS (AIM) PROGRAM



PROJECT SUMMARY

ADVANCED INDUSTRIAL MATERIALS (AIM) PROGRAM

PROJECT TITLE: Synthesis and Design of Silicide Intermetallic Materials

PHASE: FY 1998 **COMPLETION DATE:**

PERFORMING ORGANIZATION: Los Alamos National Laboratory (LANL)

PRINCIPAL INVESTIGATORS: 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. Interact with Combustion Tec Inc. and Accutru International Corporation to initially explore the use of silicides in glass-processing sensors. Interact with the Institute of Gas Technology (IGT) to test silicide materials in gas radiant tube combustion environments. Develop the plasma spraying and plasma spray–forming of $MoSi_2$-based materials and composites.

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, particularly to the glass-processing industry.

TECHNICAL APPROACH: Develop plasma-spray and plasma-spray–forming techniques for the fabrication of high fracture toughness, high thermal shock resistant $MoSi_2/Si_3N_4$-SiC fiber hybrid composites and $MoSi_2$-oxide laminate composites with high oxidation/corrosion resistance. Develop $MoSi_2$-based protection sheaths for molten-glass and glass-furnace high temperature sensors. Develop gas combustion–related applications for silicide materials.

PROGRESS: Our CRADA with Johns Manville Corporation is continuing, with emphasis on corrosion of silicide materials in molten fiberglass. Silicide materials have been tested in a gas radiant tube combustion environment by IGT. A new effort involving Accutru International Corp. and Combustion Tec Inc. has begun that is targeted at developing high-temperature protective sheaths for glass-processing sensors.

Patents: None. **Publications:** 11 **Proceedings:** 7

Books: None. **Presentations:** 10 **Awards:** 1

ACCOMPLISHMENTS: The mechanisms of corrosion of $MoSi_2$ in molten fiberglass have been established. A protective silica layer forms above the glass line, while a protective complex molybdenum-rich layer forms below the glass line. No protective layers form at the glass line. Silicide materials were tested at IGT for 339 h at temperatures in the range of 990 to 1030ºC in a natural gas combustion radiant tube furnace system.

PROJECT SUMMARY (continued)

PROJECT TITLE: Synthesis and Design of Silicide Intermetallic Materials

Licenses: None.

Known Follow-On Products: $MoSi_2$ gas injection tube for glass melting (Kanthal).

Industry Workshop: Engineering Foundation High Temperature Structural Silicides Conference, May 25–29, 1998, Hyannis, Massachusetts.

Technology Transfer or Industrial Interaction: Johns Manville, IGT, Combustion Tec, Accutru International, and Exotherm.

CRITICAL ISSUES: Development of plasma-spray and plasma-spray–forming techniques for the fabrication of high fracture toughness, high thermal shock resistant $MoSi_2$-based composite tube geometries.

FUTURE PLANS: We have initiated a new activity to develop strong, tough, and thermal shock–resistant $MoSi_2$ composite tubes by plasma spray-forming techniques, for use as high-temperature protective sheaths for advanced sensors for the glass industry. We will be interacting with Accutru International Corp., Combustion Tec Inc., Exotherm Inc., IGT, and Plasma Process Inc. to develop this area.

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

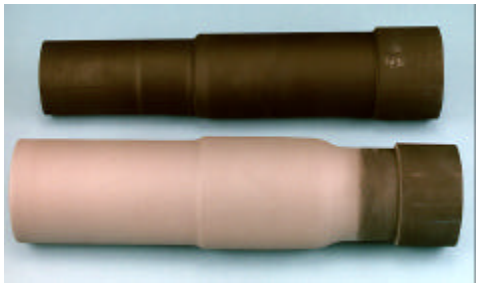
ADVANCED INDUSTRIAL MATERIALS (AIM) PROGRAM

$MoSi_2$ Periscope Sight Tube for Glass Furnace Video Monitoring System

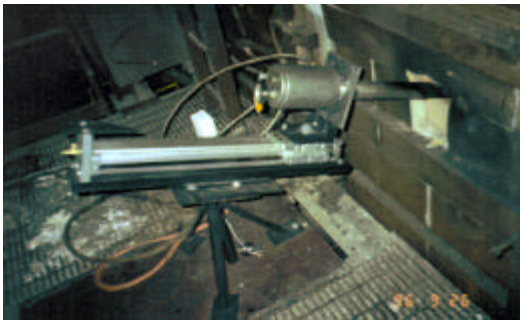
ISSUE: Advanced sensors for glass processing and glass furnaces require protective sheath materials to protect them from the highly oxidative and corrosive high-temperature glass environment so that their sensor performance will not be degraded.

RESULTS: Plasma spray–forming techniques were applied to successfully fabricate a prototype $MoSi_2$ periscope sight tube for testing in a glass furnace closed-circuit video monitoring system developed by Combustion Tec Inc. The single-phase, polycrystalline $MoSi_2$ tube was approximately 6 in. in length and 1.5 in. in outer diameter. This tube serves as a protective sheath for a water-cooled camera lens assembly that is rapidly inserted directly into the wall of a glass-melting furnace, to monitor in real time the surface characteristics of the glass melt and the furnace heating flames.

$MoSi_2$-based Periscope Sight Tubes:



- Combustion Tec Inc.,
- Enhanced lens cooling system
- Heat resistant monitoring system



Closed-circuit monitoring systems for glass melters.

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

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 to Los Alamos National Laboratory.

PROJECT SUMMARY

ADVANCED INDUSTRIAL MATERIALS (AIM) PROGRAM

PROJECT TITLE: Uniform-Droplet Spray Forming

PHASE: FY 1998 **COMPLETION DATE:**

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

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

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

ULTIMATE OBJECTIVE: To take advantage of the extremely uniform droplet and size distribution produced by 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 metal 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 system. These issues include (1) development of low- and medium-temperature systems to identify a potential high-temperature assembly, (2) determine the low- and medium-temperature optimum spraying conditions for materials with melting points between 230 and 1250ºC, (3) verify size distribution and repeatability of droplets, (4) identify and resolve any problems encountered with the low- and medium-temperature systems, and (5) translate acquired knowledge to the construction of a high-temperature UDS system.

PROGRESS: ***High-Temperature System*.** The high-temperature apparatus has been assembled, and preliminary testing is in progress. This system has allowed for the fabrication of uniform droplets of materials with melting points in excess of 1250ºC. These materials include aluminum, bronze, and copper. This system has aided in the development of the UDS system for industrial practices. Three companies have spent substantial time at ORNL gathering the technical knowledge for translation of the process to industry.

PROJECT SUMMARY (continued)

PROJECT TITLE: Uniform-Droplet Spray Forming

Two important areas of advanced materials processing are being addressed, namely advanced particulate production and controlled spray deposition. Products in the particulate area are spherical balls and flakes of metals and alloys produced by the solidification of a UDS. Strong interest already has been identified in electronics packaging in which UDS-processed solder balls are expected to replace those produced by conventional, cut-wire spheroidization. The advantage of the UDS process is expected to drive the technology toward higher melting temperature BGA balls with smaller diameters required in higher-density packaging. Other applications of UDS-processed particulate materials are expected to follow with the development of methods for multiple-orifice spraying, controlled secondary quenching, melt cleaning/filtration, high-temperature melt delivery, and stable jet breakup in finer droplet-size ranges, all of which are important aims in the joint study. Products in the spray deposition area vary from coatings to near-net-shape or net-shape spray deposits and are characterized by ultimate levels of microstructural control permitted by the decoupled spray parameters. Among the new opportunities envisioned are porosity-free deposition, precise microstructural control, production of novel microstructures and materials, and the potential for net-shape and near-net-shape processing. The joint study has generated significant fundamental knowledge on the droplet generation, droplet manipulation, in-flight solidification, droplet deposition, with the particular objective of applying the UDS process to the continuous production of flat products (e.g., sheet, strip, and coatings). Current efforts are focused on aluminum sheet production.

Patents: 1 **Publications:** 1 **Proceedings:** 3 **Presentations:** 3

CRITICAL ISSUES: Orifice clogging and materials compatibility.

FUTURE PLANS: Continue acquiring knowledge through solving technical issues with the medium- and high-temperature uniform droplet. Continue to apply the UDS technology to near-net-shape fabrication of parts. Also, continue developing interest in the industrial sector and transfer technology of the low-, medium-, and high-temperature systems.

POTENTIAL PAYOFF: 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 elimination of sieving translates into a reduction of the overall production cost through energy savings. Also, work is in progress to raster the jet electromagnetically in order to accomplish near-net-shape fabrication of parts. This will result in parts with a controlled microstructure and allow for the elimination of tooling. It can also allow for the formation of parts from materials that are inherently difficult to machine and/or cast. Energy savings will occur through the utilization of new materials.

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: A uniform-droplet spray (UDS) process has been developed at Massachusetts Institute of Technology for spraying low-melting-point materials. The most potential for the UDS process is in the medium- to high-melting-temperature alloys.

RESULTS: The low-, medium-, and high-temperature UDS systems have been fabricated at the Oak Ridge National Laboratory. Tremendous work has been accomplished in the spray deposition area. Products in this area vary from coatings to near-net-shape or net-shape spray deposits and are characterized by ultimate levels of microstructural control permitted by the decoupled spray parameters. Among the new opportunities envisioned are porosity-free deposition, precise microstructural control, production of novel microstructures and materials, and the potential for net-shape and near-net-shape processing. The joint study has generated significant fundamental knowledge on the droplet generation, droplet manipulation, in-flight solidification, and droplet deposition, with the particular objective of applying the UDS process to the continuous production of flat products (e.g., sheet, strip, and coatings). Current efforts are focused on aluminum sheet production.

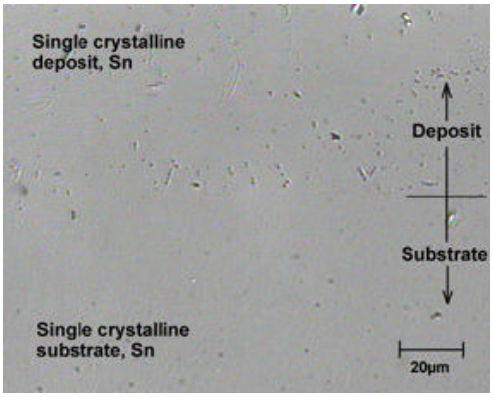


Fig. 1. Incremental epitaxial growth of rapidly solidified single-crystalline deposits: Pure Sn single-crystal deposit on a single-crystal Sn substrate.

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, eliminating rework. The elimination of sieving also translates into a reduction of the overall production cost through energy savings. Also, work is in progress to raster the jet electromagnetically in order to accomplish near-net-shape fabrication of parts. This will result in parts with a controlled microstructure and allow for the elimination of tooling. Significant energy savings can occur with the utilization of new materials.

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-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.