Industrial Materials for the Future (IMF) Program
Intermetallic Alloy Development Activities

Brief Description

Oxidation resistance and high temperature strength of intermetallic alloys, e.g., iron and nickel aluminides, make these materials strong candidates for energy-saving, emissions-reducing, and productivity-increasing use in Industries of the Future (IOF) including steel, chemicals, and petroleum, and in supporting industries including heat treating and forging. In fact, all of the IOFs have identified in their technology roadmaps the need for innovative materials with at least one of these two properties, and they have stated that substantial energy efficiency and productivity improvements could be realized with such materials. However, problems with embrittlement, leading to fabrication difficulties, low ductility, and creep in service environments presented significant technical barriers to the use of intermetallics in these industrial applications.

Beginning in 1981 with $75,000 of discretionary funding by Oak Ridge National Laboratory, and continuing with support from the Office of Basic Energy Sciences since 1982, the Office of Energy Efficiency and Renewable Energy (EERE) since 1983, and the Office of Fossil Energy since 1983, a comprehensive research and development program has overcome significant technical barriers to produce industrially useful intermetallic alloys. The overall program is an excellent example of cooperation between basic science and targeted applied research and development programs of the Department of Energy. In 1997, the National Academy of Sciences reviewed the overall program and documented this cooperative effort, including its outreach to universities, national laboratories and industry and the resulting licenses that led to today's commercial successes, as shown in Figure 1 which is taken from the NAS study.

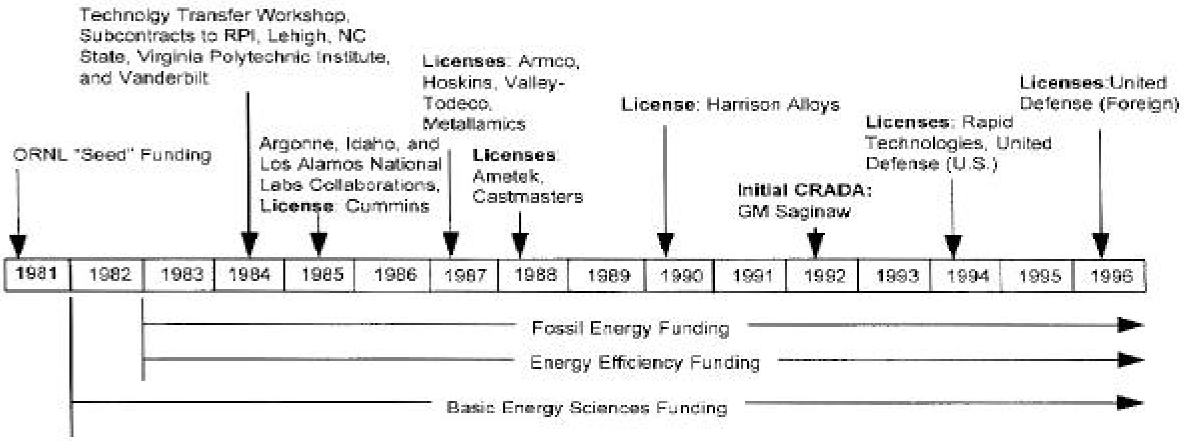

FIGURE 2-2 Time line of program management and interactions.

Figure 1

Studies were initially conducted, with the discretionary funding and later with Office of Science funding, to determine the mechanisms of embrittlement and reduced ductility. It was found that the presence of moisture and oxygen, respectively, cause these failures. With the EERE and Fossil Energy funding, ductility was improved through microalloying with boron and chromium, while embrittlement was avoided using standard alloying techniques such as solid-solution and dispersion strengthening. This provided the basis for work on alloy production methods for industrial applications, including the development of:

- an innovative, production-volume melting/alloying technique called Exo-melt® that maintains aluminum concentration while melting constituents with higher melting temperatures;
- low-cost casting processes; and
- materials and processes for making structural welds and weld repairs.

As a result of these developments, there are now several industrial-scale applications of intermetallic alloys. The two most notable applications currently being tested are heat-treating fixtures, and transfer rolls in steel manufacture. Potential energy and cost savings are impressive as these applications penetrate throughout the heat-treating and steel industries (see matrix of energy impacts below). Other applications of nickel aluminides that could see commercial use in the near future are also being developed, as described below. It is important to recognize, however, that intermetallic alloy development is a comprehensive sustained program to realize the industrial potential of a whole class of materials. In addition to nickel aluminide, the Oak Ridge National Laboratory researchers have developed a large body of knowledge about iron aluminide, titanium aluminide, and a number of silicides that also offer opportunities of commercialization in the near to medium term.

Factors Influencing R&D Funding Allocations

A key component of these achievements was the EERE activities (e.g., Energy Conversion and Utilization Technologies (ECUT) Program, which became the Advanced Industrial Materials (AIM) Program, the predecessor to the Office of Industrial Technologies (OIT) Industrial Materials for the Future (IMF) Program). This program allocated funding with the objective of capitalizing on the understanding of fracture and degradation mechanisms, as well as the effects of composition and stoichiometry, gained from the Office of Science research, in order to develop a new class of ductile, castable, weldable aluminides usable by industry. The AIM support, which focused on service in IOF industrial environments, was complementary to that of Fossil Energy, which focused on service in sulfidizing environments.

The AIM funding of intermetallics was driven by its mission, now that of IMF, to research, design, develop, engineer, and test new and improved materials, as well as more profitable uses of existing materials, for the IOFs. The emphasis of the IMF program is on filling gaps between the basic science efforts conducted by BES and other agencies and the near-term research and development (R&D) projects of the IOF Vision Teams.

IMF plays a crucial role in nurturing promising materials technologies to the point that they can be demonstrated in industrial applications. IMF seeks to develop classes of materials with suites of properties beyond the capabilities of existing commercial materials, to provide benefits that are significantly greater than the incremental improvements possible using existing materials approaches. Accordingly, approximately one-third of IMF funding is dedicated to core research, including intermetallic alloy development, that underpins these multiple industry materials research challenges.

The core research advances are achieved through mechanisms including Cooperative Research and Development Agreements (CRADAs) and applied via license agreements, as indicated in Figure 1. The intermetallic research has led to the current six materials producer and user industry licensees: Alcon Industries, Cleveland, OH; Alloy Engineering and Casting Company, Champagne, IL; Ametek, Paoli, PA; Sandusky International, Sandusky, OH; Stoody, Bowling Green, KY; United Defense, Anniston, AL; and transfer of alloy application projects to the OIT Vision Teams. ASTM specification for nickel aluminide castings have recently been approved, the first for any intermetallic alloy, which will now allow users to order by specification for commercial applications.

Products Finding Application in Areas Other Than The Original Program

As noted in the 1997 National Academy report, the program began as basic materials research to address gaps in our knowledge of a fundamental class of materials that showed promise to break through current technical barriers in materials degradation under high temperature, oxidizing, and carburizing industrial service environments. The intermetallic alloy R&D undertaken by IMF and its predecessors was aimed at developing the compositions, characterization data, and processing and fabrication methods required so that these alloys could be used in demanding, high temperature environments by multiple IOFs. Two such applications that have already occurred are described in the following paragraphs. (See matrix of energy impacts below for quantitative benefit estimates.)

Nickel aluminide trays and assemblies, cast by Alcon Industuries, Cleveland, OH and Alloy Engineering and Casting Company, Champaign, IL under license to Oak Ridge, are being used in carburizing heat-treating furnaces by GM Delphi Saginaw Steering Systems in Saginaw, MI. The furnace assemblies include base trays, upper fixtures, lower fixtures, and support posts. Each assembly carries about 340 kg (750 lbs) through the furnace. The project started in 1992 as a three-year Cooperative Research and Development Agreement between Delphi and ORNL with the purpose of evaluating and testing the nickel aluminide materials and developing casting and weld repair procedures. Over 65 fixtures were later successfully fabricated and installed in a continuous carburizing furnace, where they have been successfully operating over the last 5 years (estimated DOE funding $1 million; industrial in-kind cost-share $5 million). This year, Delphi made the decision to commercialize the technology in their heat treating facilities. The nickel aluminide furnace assemblies have greater carburization and oxidation resistance, as well as higher elevated temperature strength and creep strength than the steel alloys currently in use. The trays last more than three times as long, thus reducing both scheduled and unscheduled furnace down time considerably. In addition, the lower mass of the assemblies reduces energy requirements for heat-treating by an additional 11 percent per furnace. Due to these improvements, Delphi was able to have the same capacity installing just two new carburizing furnaces rather than the three that would have been required with the current steel fixtures, saving 60 MBtu per year, an estimated one million dollars/furnace of capital investment, and additional furnace operating energy and equipment costs.

This example illustrates that it takes significant time and effort even beyond a CRADA period for industry to build confidence in introducing new materials in commercial processes. GM Delphi heat treats over 5,000 tons of steel components/day and is so pleased with the results of several years of testing that they are in the process of changing over all of the fixtures at their Saginaw facility to nickel aluminide and plan to ultimately replace all of their steel heat-treating fixtures worldwide with nickel aluminide fixtures. Replication of this success can impact 30% of the overall heat treating industry and result in improved efficiencies of approximately 20%, leading to energy savings of nearly 30 trillion Btu/yr and energy cost reductions alone of nearly 100 million dollars/yr by 2020.

Nickel aluminide furnace rolls to transfer steel in reheat furnaces were cast by Sandusky International under license to ORNL and installed for testing and operation at the Bethlehem Steel Burns Harbor plant in Chesterton, IN. Testing and operations of the rolls is continuing (estimated DOE funding $2 million; industrial in-kind cost-share $2 million). In this application nickel aluminide offers improved high temperature oxidation resistance, improved high temperature strength of up to three times over current alloys, and the elimination of the incidence of surface blemishes on the rolls, leading to improvement in the quality of the steel slabs. The nickel aluminide rolls result in increased yield (from elimination of surface blemishes on steel plate), reduced rework and remelting of steel slabs, and significantly reduced scheduled and unscheduled downtime for roll maintenance and replacement (scheduled down time of approximately 80 days/yr with current rolls, and only approximately 12 days down time with nickel aluminide rolls) with concomitant energy savings and emissions reductions. Also, following the successful initial testing and evaluation of the nickel aluminide steel mill rolls, other applications for the material are being tested at Bethlehem Steel, USX, and Weirton Steel, with OIT Steel Team funding. The energy-savings impact of nickel aluminide materials for use in the manufacturing of steel is estimated to be approximately 8 trillion Btu/yr, with energy related cost benefits of approximately 25 million dollars/yr, by 2020.

Several additional applications are currently in the pipeline of this transition stage from IMF to the IOF Vision Teams. For example, under the aegis of AIM (the predecessor to IMF), iron aluminide coupons were tested at Dow Chemical's Freeport, Texas ethylene operation. Ethylene is the world's most important petrochemical building block based upon the number of tons produced (over 20 million tons/yr in the U. S.) and dollar value. Various intermetallic alloy materials exposed in laboratory tests showed essentially no carburization, and more importantly, did not exhibit catalytic coking. Because of these results, a team of eleven companies–chemical producers, materials suppliers, tube fabricators, and ORNL–submitted a proposal to the OIT Chemical Team solicitation to develop composite tubes for ethylene cracking, with stainless steel for strength and an aluminide-based alloy for carburization and coking resistance. The proposal was funded (estimated DOE funding $1.5 million; industrial in-kind cost-share $10 million). The first evaluation of at least two tubes is planned at the Exxon Mobil plant in Baytown, Texas. The energy savings to be realized by improved mass flow inside tubes, improved heat transfer through the tubes, elimination of shutdowns for de-coking and tube replacement will be enormous. A 5% impact on the chemical industry would result in energy savings of approximately 250 trillion Btu/yr, and an energy cost benefit of approximately 1.25 billion dollars/yr, by 2020. The savings would occur over various energy sources including petroleum products, natural gas, electricity, and coal. Primarily as a result of the alloy development program R&D performed at the Oak Ridge National Laboratory, four other projects for materials development for the chemical industry are also now being funded by the OIT Chemical Team.

These are just a few examples of the nurturing of technologies to a stage of maturity at which they can be taken over by IOF teams for the three-year types of projects, with cost-share, that will lead to full-scale commercialization. Additional applications resulting from the intermetallic alloy development projects that are currently being tested by industry include forging dies and radiant burner tubes. Companies that are testing aluminide materials products include The Timken Company (Canton, OH), FMC Corporation (Pocotello, ID), United Defense (Anniston, AL), Wyman Gordon (Houston, TX), E. I. DuPont de Nemours & Company (Wilmington, DE), and The Dow Chemical Company (Freeport, TX). For example, the forging dies used by United Defense outlasted conventional dies by a phenomenal amount. In the initial tests, a buster die lasted for 160,000 hits, compared with about 5,000 for a steel die. Radiant tubes and guide rolls for continuous steel casting are also now being successfully tested.

Cost of R&D Support

As noted above, Oak Ridge National Laboratory began funding research on intermetallics with discretionary funds, and soon thereafter other programs within DOE began supporting the intermetallic effort. The OIT IMF Program and its predecessors have provided almost half of the funding for intermetallic alloy development, as shown in the table below.

Table 1[1]

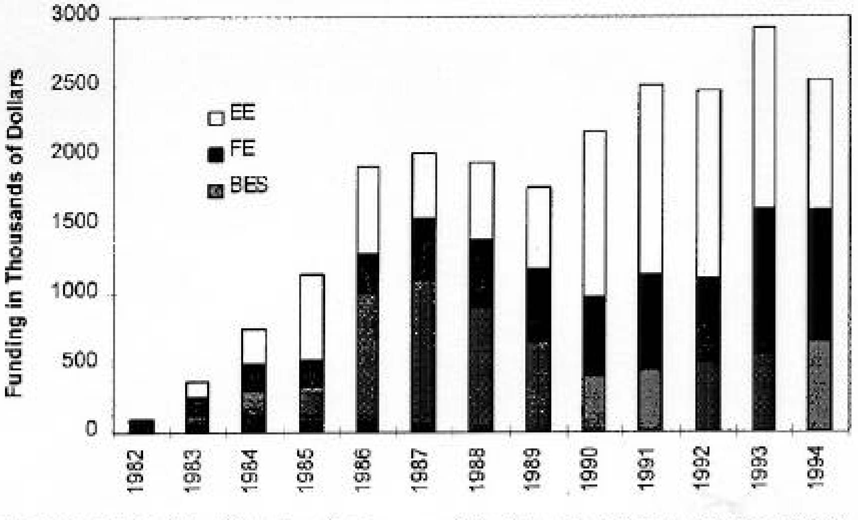

FIGURE 2-1 Profile of funding (in then-year dollars) by Basic Energy Sciences (BES), Energy Efficiency (EE), and Fossil Energy (FE) offices of DOE. Source: ORNL.

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[1] Figure taken fromIntermetallic Alloy Development: A Program Evaluation, National Research Council, Publication NMAB-487-1, National Academy Press, Washington, D. C. (1997)

Development of new materials or systems normally requires many years of funding at a level comparable with the funding shown in the table, followed by very rapidly escalating costs for demonstration, pilot plant testing, and capital investment. The current and potential applications of intermetallic alloys are being realized without those high costs because the Oak Ridge investigators have continued to work with the industrial partners to ensure that retrofitting with the new alloys can take place. Commercialization requires that materials producers, welding materials suppliers, and customers be available. This requires considerable effort on the part of the materials developers to ensure that the market can be developed and this effort must take place over many years.

Though development of basic knowledge is primarily the role of the Office of Science, there remains a need to acquire fundamental information to support the transition to commercial application. Examples of such basic knowledge include thermochemical data to enable welding and casting models for specific applications, identifying effects of microstucture and alloying elements on properties, alloy development, and environmental effects on materials performance in the industrial service environment. This information had to be developed as part of the intermetallic alloy R&D program before the applications described above could be successfully pursued. The EERE AIM program funded this work to ensure the success of the effort. Table 2 outlines year-by-year the funding that went into the development of intermetallic alloys. EERE has provided roughly 48% of the overall funding for intermetallic alloy development.

What Would Have Happened Had There Been No DOE Role

Intermetallic alloys required decades of basic and goal-oriented applied research before they were ready for industrial application. It is doubtful that industry alone would have been able to allocate sufficient resources for such a long-term sustained research program on a single class of materials. However, through the DOE-sponsored R&D and cost-shared programs with industry, intermetallic alloys are now being commercialized in IOF industries. As a result, approximately 38 trillion Btu/yr, with an estimated energy cost saving of nearly 125 million dollars/yr, will be saved by 2020 via just two general applications of nickel aluminides. With several more applications of aluminides in testing, plus the potential of other intermetallic alloys, these impacts are likely to become very substantial across several IOF industries. Without IMF and other DOE programs, intermetallics would not today be available and fabricable into usable industrial components.

The risk involved in materials research projects such as the work on intermetallic alloys is much higher than what industry can afford with respect to both funding and time. The markets in most of the IOF industries are very competitive, and the future will be even more so as these markets are increasingly affected by globalization. Consequently, R&D funds within industry are quite limited, and almost all of the industrial funding goes to near-term, low-risk development. These near-term projects produce incremental improvements in energy efficiency, usually for legacy materials, that fall very short of industry-identified performance targets required to compete in tight markets. New, advanced materials are needed to address these performance targets. In addition, the transition from proof of feasibility to commercialization requires extensive follow-on and sustained support by the technology developers, as exemplified by the two successful cases of nickel aluminide presented earlier.

Table 2

YEAR	**EERE MATERIALS ($k)***	**TOTAL DOE MATERIALS FUNDING ($k)**
1981	0	10.8
1982	0	164.6
1983	124	494
1984	297	944
1985	939.5	1795
1986	927	2525
1987	691.5	2446
1988	840	2479
1989	790	2462
1990	1326	2680
1991	1430	2946
1992	1363	3017
1993	1467	3444
1994	1088	3041
1995	1000	2864
1996	800	2536
1997	1540	2986
1998	1439	2742
1999	1150	2247
TOTAL	**20,005**	**41,823.4**

***** *Ni_3Al heat treat fixtures: estimated DOE funding $1M; industrial in-kind cost-share $5M.*
Ni_3Al steel transfer rolls: estimated DOE funding $2M; industrial in-kind cost-share $2M.
Iron aluminide ethylene cracking tubes: estimated DOE funding $1.5M; industrial in-kind cost-share $10M.
(Additional applications are continuing to be explored.)

The first two nickel aluminide furnace rolls were installed at the Burns Harbor plant in January 1994 and are still in excellent condition. However, for Bethlehem Steel to replace existing rolls with the nickel aluminide rolls has required many years of applications engineering. This included casting of the trunnions that are connected to each end of the roll, developing welding procedures to attach the trunnions, and perfecting the centrifugal casting of rolls at Sandusky International. It also takes a long time to prove that something will last a long time in the industrial environment. Without the continued engagement of Oak Ridge National Laboratory with Sandusky International and Bethlehem Steel for the past seven years to solve engineering problems and develop new materials/designs/fabrication methods as they arose, the current success could not have been realized.

Oak Ridge National Laboratory and Delphi signed a CRADA in 1992 to test nickel aluminide for use as carburizing trays and fixtures. If the effort had ended with completion of the CRADA in 1995, there would be no success today. Delphi recently held a "Success Event", celebrating the commercialization of nickel aluminide heat-treating fixtures, at which Donald Albrecht, Delphi's Director of Manufacturing Engineering, stated that fixture life has been more than doubled, "dramatically enhancing manufacturing productivity."[2] Paul J. Tosch, President of Delphi Saginaw Steering Systems and Vice President of Delphi Automotive Systems, also has said, "The benefits to be realized [from the employment of nickel aluminides] for Delphi and other industries are significant and worthy of the investment"[3] But, casting and welding procedures had to be successfully developed and long-term testing of the trays and fixtures in the carburizing furnace was required. It was also a task to show that the trays and fixtures could be made much less massive because of their superior high temperature strength. This program demonstrated once more that it takes a long time to prove that something will last for a long time in the industrial environment. Successes like this are seldom realized after completion of a three year project–continued sustained engagement over many years is essential.

Industry is unable to make use of advances emerging from the basic materials research sponsored by the DOE Office of Science and other agencies without programs like IMF that fill the gap between these basic materials research advances and industrial materials technology needs, and then follow-through with the kind of long-term continued involvement and commitment described in the previous paragraphs. Without IMF's dedicated advanced materials research, industry is left with incremental improvements in materials technology. With IMF, industry can enter new paradigms of energy efficiency with the help of advanced materials like intermetallic alloys and, therefore, set the worldwide industrial standard for energy and production efficiency.

Energy Impacts Matrix

Intermetallic alloys are in the early stages of commercialization. Currently, one application is in commercial use (nickel aluminide heat-treating fixtures at Delphi, Saginaw) and various others are under test at industrial sites (e,g., nickel aluminide transfer rolls for the steel industry at Bethlehem Steel). As quoted earlier, management at Delphi have hailed these new heat-treating fixtures as "dramatically enhancing manufacturing productivity." Table 3 lists net cumulative energy savings estimates, as well as non-energy savings benefits for each of these applications, as well as another application that is in an earlier stage of commercial testing. As additional applications enter the pipeline and develop in maturity over the coming years, and as other IOF

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[2] "Delphi Adopts Ni_3Al For Heat Treat Fixtures," Advanced Materials & Processes, ASM International, vol. 159, no. 6, p. 9, (June 2001).

[3] Press Release, Delphi "Success Event" (June 2001).

industries identify specific applications of intermetallics, the overall energy impact of the development of industrially-applicable intermetallic alloys and related materials will grow substantially beyond the initial impacts shown in the table.

Table 3

Intermetallic Alloy Application	Net Annual Energy Savings (2020)	Non-energy Benefits	Knowledge Benefits
Nickel aluminide heat treating fixtures	**Increased productivity allows use of 2 rather than 3 new furnaces at Delphi, saving 60 MBtu/year** **30 trillion Btu/yr ($100 million/yr)**	Improved productivity (reduced downtime since trays last more than twice as long because of better resistance to carburization and oxidation) Improved environmental performance	Nickel aluminide alloy design (compositions) Casting technology and materials properties Joining and weld repair technology
Nickel aluminide transfer rolls for steel furnaces	**8 trillion Btu/yr ($25 million/yr)**	Improved productivity (reduced downtime because of better resistance to oxidation) Improved quality (less scratching of steel plates) Improved environmental performance, including reduction in emissions of combustion-related pollutants	Improved knowledge of the performance of these alloys and their benefits in steel reheat furnace operations Development of manufacturing and coating methods for intermetallic components
Iron aluminide and other advanced material tubes for ethylene cracking	**250 trillion Btu/yr ($1.25 billion/yr)**	Improved productivity through fewer and shorter decoking operations, fewer scheduled and unscheduled down times Improved environmental performance through more efficient use of energy and more efficient process operations	Improved knowledge of the behavior of alloys that survive under the severe operating environment of ethylene processing Improved alloys including intermetallic and metallic compositions Development of alternate fabrication techniques for producing tubes of different alloy compositions

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