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DEVELOPMENT AND COMMERCIALIZATION STATUS OF Fe_3Al-BASED INTERMETALLIC ALLOYS

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Abstract

The Fe_3Al-based intermetallic alloys offer unique benefits of excellent oxidation and sulfidation resistance at a potential cost lower than many stainless steels. Such benefits of Fe_3Al-based alloys have been known since the 1930s. However, development of these materials has been limited by at least two major issues: poor room-temperature (RT) ductility and low high-temperature strength. Recent understanding of environmental effects on RT ductility of these alloys has led to progress toward taking commercial advantage of good properties of Fe_3Al-based materials. The cause of low ductility appears to be related to hydrogen formed from the reaction of aluminum in the alloy with moisture in the air. The environmental effect has been reduced in these intermetallic alloys by two methods. The first deals with producing a more hydrogen-resistant microstructure through thermomechanical processing, and the second has dealt with compositional modification.

The alloys showing reduced environmental effect have been melted and processed by many different methods. The material has been prepared both in the laboratory and at commercial vendors. The laboratory and commercial heats have been characterized in terms of their tensile, impact, creep, and fatigue properties. Tests have been conducted in both air and controlled environments to quantify environmental effects on these properties. These materials have also been tested for their aqueous corrosion response in various media and their resistance to stress corrosion cracking. Oxidation and sulfidation data on these alloys have been generated over a range of compositions, and effects of minor alloying elements on cyclic oxidation resistance have also been investigated.

Several applications have been identified for the newly developed iron aluminides. Commercialization status of these alloys is described.

Introduction

Iron aluminides have been of interest since the 1930s when their excellent oxidation resistance was first noted (1,2). Since that observation, research has continued on these materials to improve their room-temperature (RT) ductility and strength above 600°C. Especially noteworthy are the studies conducted at the Naval Ordnance Laboratory in the 1950s (refs. 3-6); Ford Motor Company in the 1950s and 1960s (refs. 7-9); Iowa State University from 1965 through 1975 (refs. 10-15); and, more recently, the work at Pratt & Whitney (16), TRW (17), and the Oak Ridge National Laboratory [ORNL] (18-23). All of these studies, as well as less extensive studies in other laboratories, have resulted in significant contributions to the understanding of the fabrication and mechanical properties of iron aluminides, as well as selected demonstrations of the excellent corrosion resistance of these alloys. Several excellent reviews have been written on the progress made on the iron-aluminide alloys (24-29). The purpose of this paper is to report on how the recent insight into the causes for low ductility of these materials has helped in their development for several potential applications. Specifically, this paper will describe the environmental effect; effect of composition and heat treatment on environmental effect, melting, processing and mechanical properties of these alloys; and will give one example of a prototype part.

Phases and Crystal Structure

Studies of phase relationships in the Fe-Al system have confirmed (30,31) the following equilibrium phases near the Fe_3Al composition: a disordered solid solution (α), an Fe_3Al with an imperfectly ordered B2 structure, an ordered Fe_3Al with the $D0_3$ structure, and the two phase regions of $\alpha + D0_3$ and α + B2. The $D0_3$ and B2 crystal structures are derivatives of the body-centered-cubic structure. The transition temperature (T_c) between $D0_3$ and B2 in the binary Fe_3Al alloys is approximately 550°C.

Environmental Effects

Fe_3Al alloys have been known to have poor ductility at RT. However, recently, it has been pointed out (32-35) that the interaction of H_2O vapor in the air with aluminum in the alloy is the major contributor to the low RT ductility of Fe_3Al and FeAl alloys. The extent of the environmental effect on binary Fe_3Al (28% Al) [*unless otherwise noted, all compositions will be given in atomic percent*] is illustrated in Table I (34). The mechanism proposed (32-35) for the environmental effect is similar to that observed in aluminum and its alloys (36,37). The embrittlement is thought to involve the chemical reaction (37):

$$2\ Al + 3\ H_2O \rightarrow Al_2O_3 + 6\ H \qquad (1)$$

Because iron-aluminide alloys contain relatively large concentrations of aluminum, it is postulated that the aluminum reacts with the water vapor in air, producing alumina and chemisorbed atomic hydrogen, which induces classic hydrogen embrittlement at crack tips, where fresh surfaces are created due to stress concentrations. Additional work is needed to understand further details of environmental embrittlement in iron aluminides. However, it is clear that the low ductility commonly observed at RT is the result of an extrinsic factor (namely, moisture-induced environmental embrittlement).

Methods of Reducing Environmental Effect

Stopping Generation of Hydrogen

Elimination of moisture coming into contact with the iron-aluminide alloy is one obvious method of stopping hydrogen generation. Table I has shown that if the moisture content from the environment is reduced by evacuating the air surrounding the specimen, high-ductility values are possible. The major reason for

Table I Effect of Test Environment on Room-Temperature (RT) Tensile Properties of Binary Fe_3Al (28% Al)[a]

Test Environment	Elongation (%)	Strength (MPa)	
		0.2% Yield	Ultimate Tensile
Air	3.7	279	514
Vacuum (10^{-4} Pa)	12.4	316	813
Oxygen[b]	11.7	298	888
H_2O vapor[c]	2.1	322	439

[a]Specimens were annealed at 850°C for 1 h followed by a five-day treatment at 500°C to stabilize $D0_3$ at RT. All of the tests were at a strain rate of 3.3×10^{-3} s^{-1}.
[b]Chamber was evacuated to 10^{-4} Pa, then oxygen was leaked in to a partial pressure of 6.7×10^4 Pa.
[c]Air saturated with water vapor was leaked into the vacuum chamber.

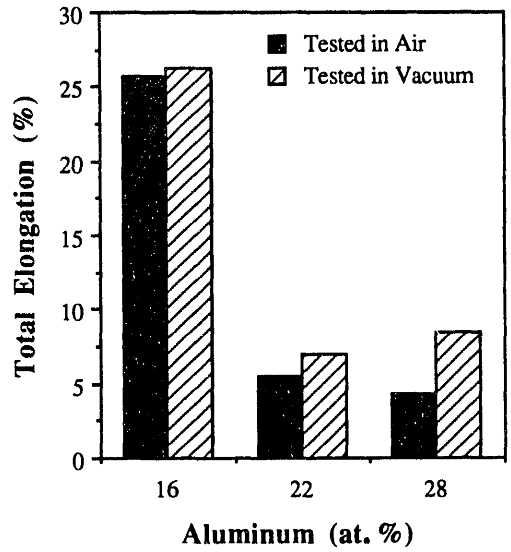

Figure 1. Effect of aluminum content on minimizing environmental effect in binary Fe-Al alloys. All specimens were tested after a 1-h treatment at 700°C followed by air cooling.

high ductility is prevention of hydrogen generation. Evacuating the chamber followed by introducing dry oxygen also results in high ductility. This observation again suggests that as long as moisture is eliminated, ductility is high.

An oil film on the specimen surface can prevent the contact of moisture in air with the aluminum on the alloy surface and, thus, reduce or eliminate the hydrogen embrittlement. An example of its effectiveness is shown in Table II for an Fe-28 Al-2 Cr alloy. Note that the specimen tested with oil had nearly double the ductility.

Table II Effect of Oil Film on Room-Temperature Tensile Properties of a Ternary Fe-28% Al-2% Cr Alloy

Surface Treatment[a]	Elongation (%)	Strength (MPa)	
		0.2% Yield	Ultimate Tensile
Bare	6.4	537	810
Coated with oil[b]	17.8	519	1018

[a]Tensile tests were conducted in air at a strain rate of 3.3 x 10-3 s^{-1}.
[b]Mineral oil.

These methods of stopping the hydrogen generation are acceptable for research studies, however, their practical applications are limited because: (1) the alloy will be used in air and not in vacuum and (2) for most of the applications, oil coating on the surface will not be acceptable.

Reducing Environmental Embrittlement

There are several potential methods of improving the RT ductility through reduction in the environmental-embrittlement effect. Each of the methods is described below:

Control of Composition. The aluminum variation from 16 to 28% in the Fe-Al binary alloys (Fig. 1) showed that the ordered alloy (28% Al) was the most sensitive, and the disordered alloy (16% Al) was the least sensitive to the environmental effect. Furthermore, the disordered binary alloy possessed the highest ductility at over 25%.

Chromium addition was found to have a strong beneficial effect on improving the ductility of the ordered alloy (28% Al) (Fig. 2). Further additions of carbon or zirconium/carbon for grain refinement showed no additional benefits. The ductility of the disordered alloy, in which the environmental effect (Fig. 1) was substantially reduced, was not affected by the chromium addition. The two-phase alloy containing the α-solid solution and the ordered $D0_3$ phase showed significant improvement in ductility with the chromium addition. This alloy also showed further improvement in ductility with the addition of grain refiners such as zirconium/carbon. One possible reason for such improvement may be the more uniform distribution of the ductile α-solid solution around each of the ordered-phase particles. Several possible reasons for the improvement in ductility with chromium additions have been suggested by McKamey et al. (29).

Microstructural Modifications. The environmental effects can also be reduced by minimizing hydrogen diffusion from the surface to the specimen interior through microstructural modification (38,39). The microstructures and the associated ductilities for an Fe_3Al alloy containing 5% Cr, 0.1% Zr, and 0.04% B are shown in Fig. 3. The highly elongated structure, with minimum transverse grain boundaries, is the most resistant to hydrogen diffusion, resulting in its highest ductility. The increased recrystallization increased the number of transverse boundaries, which increased the hydrogen diffusion and resulted in lower ductility values for ductility [values marked in the upper right-hand corner of Fig. 3(*b-d*)].

Control of Ordered Structure. The environmental effect in Fe_3Al has been shown (40) to be dependent on the crystal structure at RT. The $D0_3$ structure is more environmentally sensitive than the B2 structure. This has been demonstrated by the data on a binary and ternary alloy tested in both $D0_3$ and B2 conditions (see Table III). Note that having the $D0_3$ structure can reduce the ductility to nearly half of that in the B2 condition.

Maximization of RT Ductility

A combination of methods described above can be used to maximize the RT ductility of Fe_3Al alloys. Note, however, that the gain in ductility from thermomechanical processing and control of

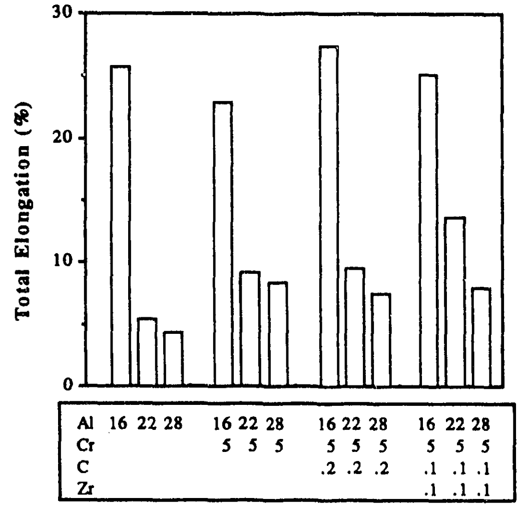

Figure 2. Effect of alloying additions in atomic percent to reduce the environmental effect of Fe-Al alloys. All of the specimens were tested in air after a 1-h treatment at 700°C followed by air cooling.

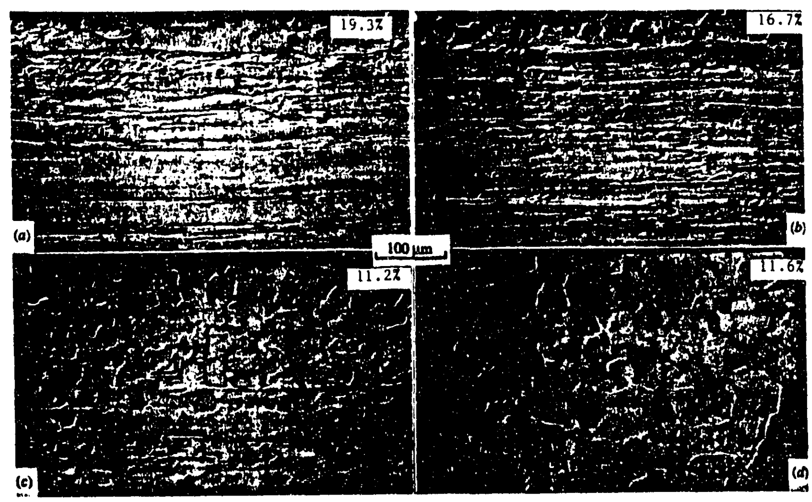

Figure 3. Optical micrographs of the oil-quenched, Fe_3Al-based alloy annealed for 1 h at: (*a*) 700°C, (*b*) 750°C, (*c*) 800°C, and (*d*) 900°C. Room-temperature values of total elongation for each microstructure are marked on the micrographs.

Table III Effect of Crystal Structure on Room-Temperature Tensile Properties of Fe-28 at. % Al and Fe-28 at. % Al-5 at. % Cr[a]

Alloy	Heat Treatment[b]	Crystal Structure	Elongation (%)	Strength (MPa)	
				0.2% Yield	Ultimate Tensile
Fe-28 Al	800°C/1 h/AC	B2	3.90	366	600
Fe-28 Al	800°C/1 h/AC plus 500°C/96 h/AC	$D0_3$	2.40	326	456
Fe-28 Al-5 Cr	800°C/1 h/AC	B2	7.04	533	558
Fe-28 Al-5 Cr	800°C/1 h/AC plus 500°C/96 h/AC	$D0_3$	4.06	265	423

[a]All of the tests were done in air at a strain rate of 6.7×10^{-4} s^{-1}.
[b]AC = air cooled.

crystal structure is of a temporary nature and will be lost under certain component fabrication or operating conditions. The more stable and reproducible improvement in ductility is produced by controlled alloy chemistry. Further chemistry modifications are still needed to improve the ductility of Fe_3Al-based alloys. The Fe-16% Al-based alloys offer compositions with RT ductility values exceeding 25% with a greatly reduced environmental effect.

Commercialization of Iron Aluminides

Alloy Compositions

Based on the concepts discussed above, five alloy compositions have been identified for commercialization at ORNL (see Table IV).

Melting

Air melting is feasible (41-43) for iron-aluminide alloys. Reasonable, but not exceptional, care is needed in treating the melt charge and the selection of crucible material. Both the iron and aluminum need to be dried to minimize the generation of hydrogen. Because of the reaction of aluminum with moisture, a large amount of hydrogen can be generated and dissolved in molten metal. It is the rejection of this hydrogen during solidification that causes the gas porosity in iron aluminides. Typical hydrogen levels in the Fe_3Al alloy melted in air can be in the range of 3 to 4 ppm. The hydrogen level of the alloy can be further reduced to approximately 2 ppm by blowing argon through the melt. Vacuum melting of the alloy can yield hydrogen levels of approximately 1 ppm.

The high aluminum content of the Fe_3Al-based alloys allows excellent protection through the formation of a protective aluminum oxide slag. The aluminum oxide formation yields (41) low levels of oxygen and nitrogen in the melt (see Table V) and also provides nearly 100% recovery (41) of most of the alloying elements. Vacuum melting and electroslag-remelting processes further reduce the oxygen and nitrogen contents of the Fe_3Al-based alloys (see Table V).

Melting in a magnesia crucible showed a pickup of 20 ppm of magnesium as opposed to 10 ppm observed in the Al_2O_3 crucible. The higher magnesium level, especially when segregated, can cause hot-workability problems (41). The magnesium levels can be reduced to 10 ppm by the vacuum-arc-remelting process.

Casting

The Fe_3Al-based alloys are castable into shapes by both sand and investment-casting processes. The casting parameters such as type of sand, melt superheat, cooling rates, and post-cast treatments are not fully developed for sand castings. In the case of investment castings, issues such as shell material, use of grain refiner, shell temperature, melt superheat, cooling rates, and post-cast treatments need additional work. The low RT ductility in the as-cast condition is the primary concern in the handling and use of castings. Efforts are under way at ORNL to improve the cast ductility and to find answers to some of the questions raised above regarding the casting processes.

Processing

Fe_3Al-based alloys are hot workable with typical hot-working temperature ranging between 900 to 1100°C. The hot-worked material can be warm finished at temperatures as low as 650°C. The Fe-16% Al alloys can also be cold finished with intermediate anneals at 800°C. However, the Fe_3Al-based alloys are not cold workable.

Mechanical Properties

The tensile (44,45), creep (46), impact (47), and fatigue (44,48) properties of alloy compositions in Table IV have been reported previously. The total data set for tensile and creep properties have been analyzed to develop the average property curves and assembled into a data package (49). The average tensile property curves and 10^3-h creep-rupture values are plotted as a function of test temperature in Figs. 4 and 5. The impact properties of Fe-16% Al-based alloys (FAP and FAPY) and the Fe-28% Al alloy (FA-129) are compared in Fig. 6. Data in Figs. 4(*c*) and 6 show that the Fe-16% Al alloy has significantly higher tensile ductility and upper-shelf energies (USE) compared to Fe_3Al-based alloys. It should be pointed out that even with much higher tensile ductility and USE, the ductile-to-brittle transition temperature (DBTT) for Fe-16% Al-based alloy is still about 150°C. Efforts are needed to reduce the DBTT below RT.

Commercialization Status

Iron-aluminide alloys offer several advantages over the commercial materials to make them suitable for many applications (see Table VI). Several sizes of pilot heats (7 to 230 kg) of the

Table IV Compositions of Fe_3Al and Fe-16 at. % Al-Based Ductile Alloys

Element	Alloy (atomic percent)				
	FAS[a]	FAL[b]	FA-129[c]	FAP[d]	FAPY[e]
Al	28.08	28.03	28.08	16.12	16.12
Cr	2.02	5.03	5.04	5.44	5.44
B	0.04	0.04	--	--	--
Zr	--	0.08	--	0.11	0.11
Nb	--	--	0.51	--	--
C	--	--	0.20	0.13	0.13
Mo	--	--	--	1.07	1.07
Y	--	--	--	--	0.06
Fe	69.86	66.81	66.17	77.13	77.07

[a]Sulfidation-resistant alloy.
[b]High room-temperature (RT) tensile ductility.
[c]High-temperature strength with good RT ductility.
[d] Very high RT ductility.
[e]Very high RT ductility and high-temperature oxidation resistance.

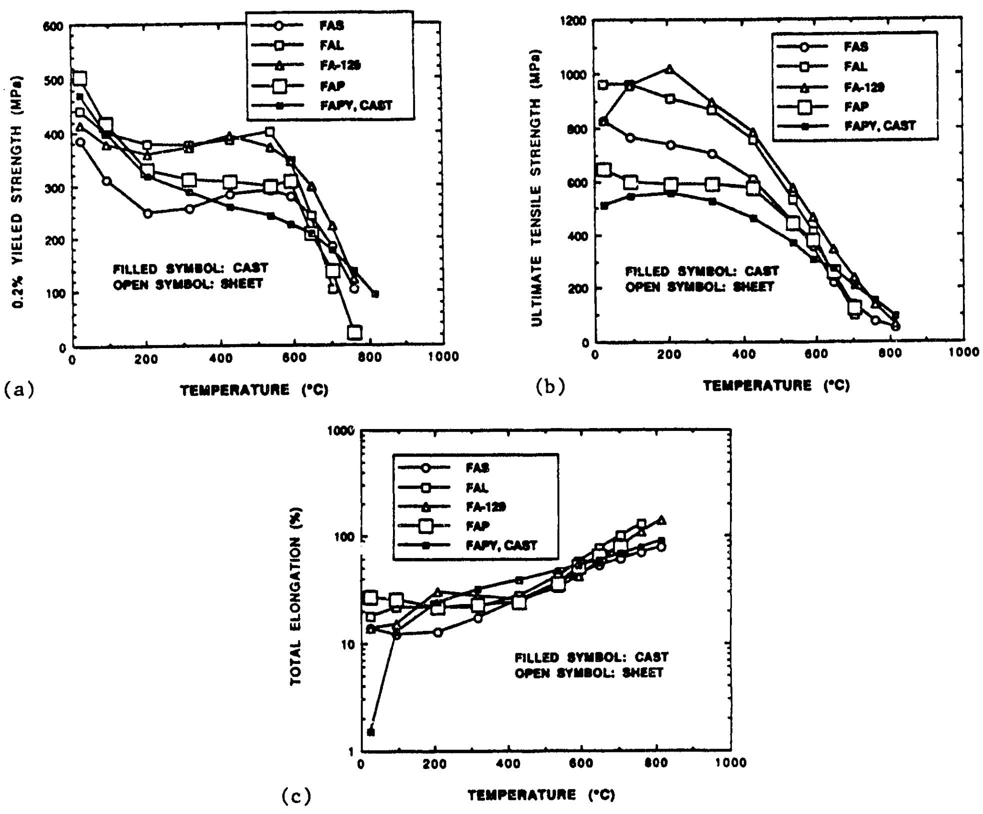

Figure 4. Average tensile properties of Fe-16 at. % Al-based alloy (FAP in wrought condition and FAPY in cast condition) and Fe-28 at. % Al-based alloy (FAS, FAL, and FA-129 in wrought condition): (*a*) 0.2% yield strength, (*b*) ultimate tensile strength, and (*c*) total elongation. Note the cast material has the lowest ductility.

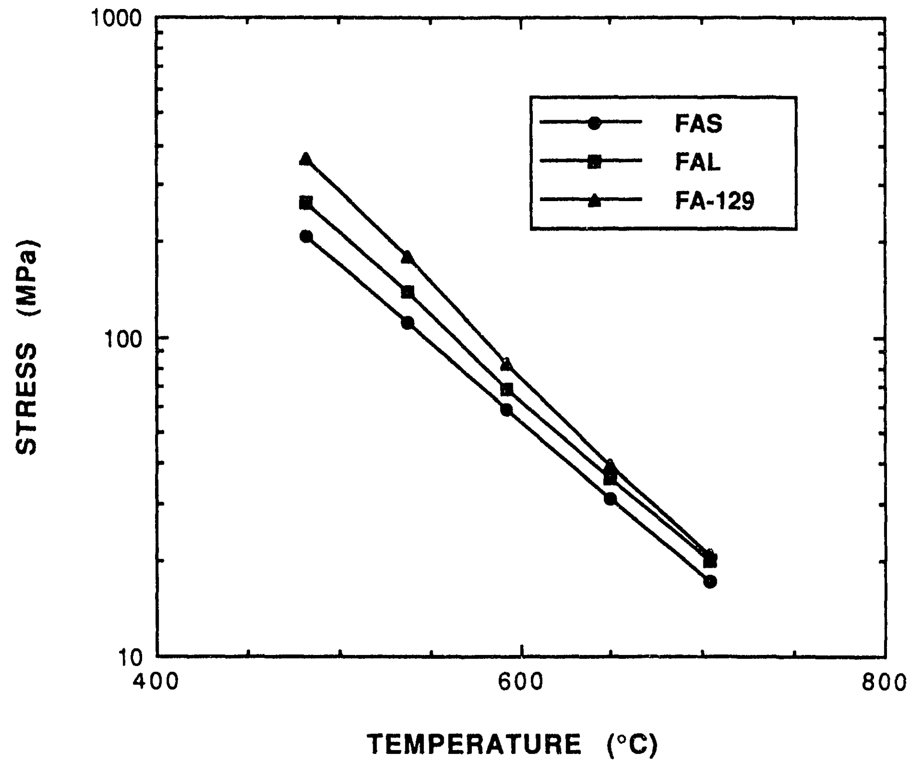

Figure 5. Average 1000-h rupture strength as a function of temperature for Fe-28 at. % Al-based alloy FAS, FAL, and FA-129 in the wrought condition.

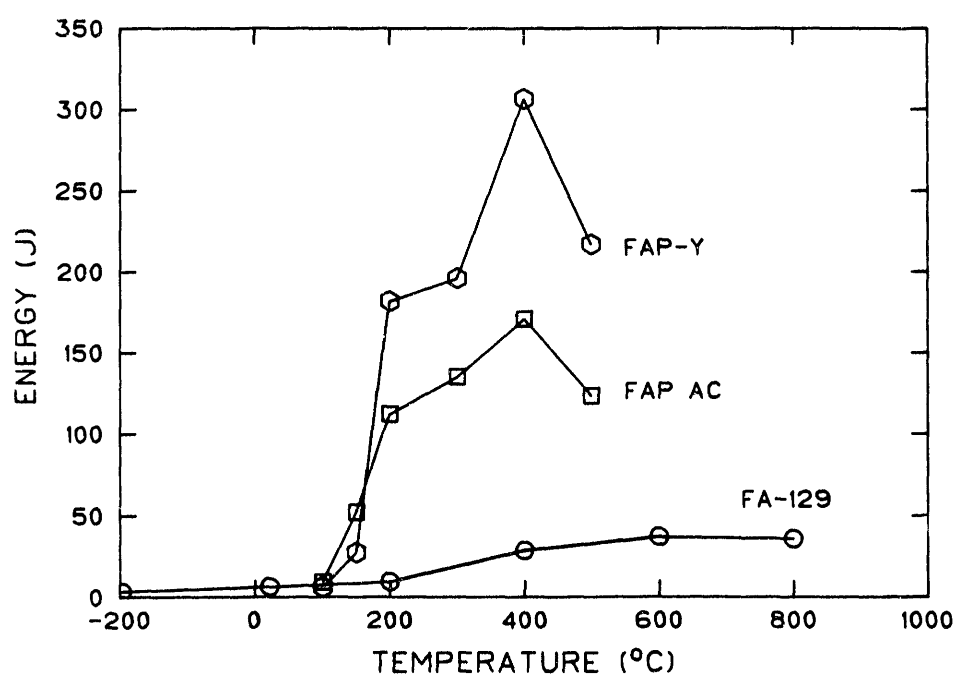

Figure 6. Charpy-impact properties of Fe-16 at. % Al-based alloys (FAP and FAPY) and Fe-28 at. % Al-based alloy FA-129 in the wrought condition. Note the significant improvement in upper shelf energy of the Fe-16 at. % Al-based alloys.

Table V Effect of Melting Practice on Range of Oxygen and Nitrogen Content Observed in Fe_3Al-Based Alloys

Melting Practice	Weight Percent	
	Oxygen	Nitrogen
Air Induction	0.0017 to 0.0040	0.0005 to 0.008
Vacuum Induction	<0.0007 to 0.0020	0.0001 to 0.0010
Vacuum-Arc Remelting	0.0010	0.0003
Electroslag Remelting	<0.0010 to 0.0014	0.0002

Fe_3Al-based alloys have been melted by commercial vendors (41). The 2000- and 3000-kg heats are the largest heats melted by Precision Rolled Products (50).

Table VI Potential Applications of Iron Aluminides

Application	Component System
Heating elements	Toasters, stoves, ovens, cigarette lighters, and dryers
Wrapping wire	Insulation wrapping for investment-casting molds
Regenerator disks	Automotive gas-turbine engines
Hot-gas filters	Coal-gasification systems
Tooling	Dies for superplastic forming of titanium-based alloys
Shields	Coal-fired power plants to protect the superheater and reheater tubes
Automotive	Exhaust manifolds, catalytic converters, and exhaust support hangers
Molten metals	Sensor sheathing material for molten aluminum, zirconium, and cadmium
Others	Components needing high-temperature sulfidation and oxidation resistance

The Fe-16% Al alloy has been cast by a commercial vendor into three different shapes. The alloy powder has been produced in commercial quantities by Ametek Specialty Metal Products and has been used to fabricate co-extruded tubing with iron aluminide on the outside and stainless steel on the inside (see Fig. 7). It has been commercially sprayed on several base materials.

The ORNL-developed alloy compositions (Table IV) have been licensed to four commercial vendors for manufacturing. The licensees are: (1) Ametek Specialty Metal Products Division for powder production, (2) Hoskins Manufacturing Company for heating elements, (3) Harrison Alloys for heating elements, and (4) Cast Masters for cast parts.

Additional work is needed to improve the ductility and strength of Fe_3Al-based alloys in both cast and wrought conditions. Efforts are also needed to develop welding wire, coated electrodes, and welding procedures. Additional physical and mechanical properties data need to be generated on commercially produced heats of alloys shown in Table IV and on new potential compositions with improved ductility and high-temperature strength.

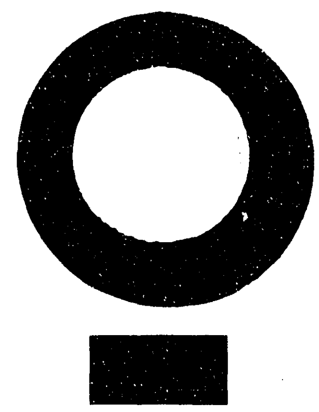

Figure 7. Photograph of co-extruded tubing of Fe-28 at. % Al alloy (FAS) on the outside of a 304 stainless steel tube. This piece was subjected to 500 h of thermal cycling between 900°C to room temperature. Each cycle was after 40 h.

SUMMARY AND CONCLUSIONS

Iron aluminides have been of great interest since the 1930s because of their excellent corrosion resistance. However, their commercialization has been hindered because of their extremely low ductility at RT. Recently, environmental embrittlement through the reaction of aluminum in the alloy with moisture in air has been identified as the cause for poor RT ductility. Methods of stopping or minimizing the embrittlement from hydrogen have been described. Data have been presented on alloy compositions, melting, casting, processing, properties, applications, and commercialization status. The following conclusions are possible from this work:

1. Eliminating H_2O by evacuating the environment around the test bar or minimizing hydrogen penetration with an oil film are two methods which can produce higher tensile ductility values at RT. However, these methods are useful for research studies and may not apply to practical applications.

2. Among the three methods of reducing environmental embrittlement and improving ductility, compositional modification is the best method. The chromium addition is beneficial in reducing the embrittlement of Fe_3Al-based compositions. The disordered alloy based on Fe-16% Al is not as sensitive to the environmental effect and possesses ductility of ≥25% at RT.

3. Care in the selection and pretreatment of melt stock, along with argon blowing through molten metal, can help to reduce hydrogen-related gas porosity in Fe_3Al-based alloys.

4. Use of an MgO crucible for melting can result in pickup of up to 20 ppm of magnesium in the alloy which, in the segregated form, can cause hot-workability problems. The use of an Al_2O_3 crucible can prevent magnesium pickup. If melted in an MgO crucible, secondary melting practices such as vacuum-arc remelting can reduce the magnesium content.

5. The Fe_3Al-based alloys can be processed by hot and warm working. However, the Fe-16% Al-based alloys can also be cold worked.

6. The Fe_3Al and Fe-16% Al-based alloys have several potential applications. Further work is needed to improve the RTtoughness, ductility, and high-temperature strength.

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