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DEVELOPMENT OF DUCTILE Fe_3Al-BASED ALUMINIDES

C. G. McKamey, V. K. Sikka, and G. M. Goodwin

Oak Ridge National Laboratory
P.O. Box 2008
Oak Ridge, TN 37831-6115

ABSTRACT

Iron aluminides based on Fe_3Al are of interest because of their excellent oxidation and corrosion resistance, especially in sulfur-bearing atmospheres. Work at Oak Ridge National Laboratory (ORNL) has centered on developing Fe_3Al-based alloys with improved ambient temperature ductilities and increased strengths at temperatures of 600-700°C. Ambient temperature brittleness in this system is not "inherent", but is caused by atomic hydrogen which is produced by an environmental reaction between aluminum in the alloy and water vapor in the atmosphere. Great strides have been made in understanding this embrittlement phenomenon, and the production of alloys with room temperature ductilities of over 10% and tensile yield strengths at 600°C of as high as 500 MPa is now possible through modifications in alloy composition and control of thermomechanical processing techniques. Creep rupture lifes of over 200 h at 593°C (1100°F) and 207 MPa (30 ksi) can also be produced through control of alloy composition and microstructure. This paper summarizes our present efforts to improve the tensile and creep rupture properties and gives the status of efforts to commercialize Fe_3Al-based alloy compositions.

INTRODUCTION

Iron aluminide alloys have excellent corrosion resistance, low material cost, and a lower density than stainless steels. Their tensile strengths also compare favorably with many ferritic and austenitic steels. However, limited ductility at ambient temperatures and a sharp drop in strength above 600°C have been major deterrents to their acceptance for many structural applications. Recent studies have demonstrated that improved engineering ductility (to 10-15% in Fe_3Al) can be achieved in wrought Fe_3Al-based iron aluminide alloys through control of composition and microstructure.[1-4] Accompanying this improvement has been an increased understanding of the causes for ambient temperature embrittlement in this system.[5-6] Because of these advances, iron aluminide alloys are again being considered for structural uses, especially for applications where their excellent corrosion resistance is manifested.

In past years, several efforts have been undertaken to understand and improve the metallurgical properties of iron aluminides with the aim of producing strong, ductile, corrosion resistant materials for structural applications.[7-11] Although iron and aluminum form several intermetallic phases, the iron-rich iron-aluminide compositions appear to have the most potential for structual applications. The current interest of the Fossil Materials Program centers on compositions near the Fe_3Al ordered phase, as well as disordered compositions of about 16 at.% Al. Since many other aspects of the iron-aluminide alloy development program will be discussed as part of this proceedings (including as-cast properties, weldability, corrosion resistance, fracture behavior, and corrosion resistance), this paper will only discuss, in general terms, the recent progress that has been made on improving tensile and creep-rupture properties of Fe_3Al-based iron aluminides, as well as the current status of commercialization of alloys developed at ORNL.

DISCUSSION OF CURRENT ACTIVITIES

Maximization of Room Temperature Mechanical Properties

The mechanical properties of iron aluminides are very sensitive to many factors including aluminum content, order (type, amount, size of ordered domains), heat treatment, test temperature, alloying additions, environment, microstructure, and defects. At room temperature (RT), compositions below approximately 20 at.% Al fail in a ductile manner by void nucleation and coalescence and exhibit tensile ductilities of over 20%.[12] With the onset of the ordered $D0_3$ and B2 structures, however, ductility decreases rapidly. At temperatures below the ductile-brittle transition, the ordered FeAl and Fe_3Al phases characteristically exhibit limited tensile ductility and brittle fracture. Until recently, the poor ductility of Fe_3Al-based compositions was assumed to reflect an intrinsically low cleavage strength,[13] in combination with a retardation of cross-slip due to the ordered phase.[14] However, recently, Liu et al.[5-6] showed that the ductilities of both FeAl and Fe_3Al are significantly decreased through the reaction of water vapor in the test environment with aluminum in the alloy. This reaction releases atomic hydrogen which induces hydrogen embrittlement. These results indicate that Fe_3Al and FeAl are intrinsically much more ductile than previously realized, and that the low ductility commonly observed at RT is the result of an extrinsic factor, namely moisture-induced environmental embrittlement.

Because of the great potential of iron aluminides for use in applications where its superior sulfidation and oxidation resistance can be exploited, efforts are being pursued to minimize or eliminate the environmentally-produced hydrogen embrittlement in this system. Results to date indicate that improved RT tensile ductility can be produced in ordered iron aluminides by controlling the (1) composition, (2) ordered phase structure, (3) grain size and shape, and (4) surface conditions.

Studies have shown that the ductility of the Fe_3Al-based aluminide can be substantially improved by increasing aluminum content from 25 to 28 at.% (ref. 15) and by adding chromium at a level of 2-6% (ref. 16). The increase in aluminum concentration sharply decreases the yield strength of the aluminide by eliminating the hardening effect of precipitated α phase.[15] The beneficial effect of chromium has not been totally explained but studies suggest several possible mechanisms. These include (1) enhanced cleavage strength and partially suppressed cleavage fracture[16]; (2) reduced APB energies, which increases the separation between the four superpartial dislocations and thereby allows easier dissociation of the superdislocations and easier cross-slip[16]; and (3) reduced environmental embrittlement due to some as-yet unexplained effect on the water vapor/aluminum reaction.[17,18]

Fe_3Al alloys can be improved further by thermomechanical treatment and alloying with other elements to improve strength.[19,20] Stress relief heat treatment below the recrystallization temperature, followed by oil quenching, has produced dramatic increases in ductility which is believed to be due to three factors: (1) Oil quenching after heat treatment leaves a residue of oil in any cracks present on the specimen surface and thus minimizes the entrance of hydrogen during stressing. (2) Heat treating at 700-750°C relieves stress in the sheet but maintains the elongated grain morphology of the rolled microstructure. This unrecrystallized microstructure provides a minimum of transverse cleavage planes, thereby disrupting the fracture path and making it more difficult for hydrogen to enter.[21,22] (3) Heat treatment at 700-750°C followed by rapid quenching maximizes the retention of B2 ordered phase and minimizes the amount of $D0_3$ structure, which has been shown, through tensile and fatigue tests, to be more environmentally sensitive than the B2 structure in the Fe_3Al-based compositions.[21,23]

The additions of Mo, Nb, and Zr not only improve strength, but produce grain refinement and increases in recrytallization temperature. Table I shows the effect of adding small amounts of these elements on the RT tensile properties of the FA-129 composition. With the addition of up to 0.8% Mo and 0.05% Zr, along with 0.03-0.05% C to promote carbide precipitate formation, the RT tensile strength of FA-129 doubles. However, as has been previously observed for Fe_3Al-

Table I. Tensile properties of Fe_3Al-based compositions

Alloy[a] FA-	Nominal Composition (at.%)	RT[b] Strength[c] (MPa)		RT[b]	600°C[b] Strength[c] (MPa)		600°C[b]
		YS	FS	El. (%)	YS	FS	El. (%)
129	Fe-28Al-5Cr-0.5Nb-0.2C	384	930	16.9	417	520	38.3
	<u>Fe-28Al-5Cr-0.005B</u>						
168B	-0.5Nb-0.8Mo-0.05Zr-0.03C	699	958	10.7	599	621	34.7
176B	-0.5Nb-0.4Mo-0.025Zr-0.05C	569	929	11.8	536	546	41.3
179	-0.5Nb-0.6Mo-0.025Zr-0.05C	658	985	10.0	554	565	33.8
180	-0.5Nb-0.8Mo-0.025Zr-0.05C	610	900	8.4	538	571	34.7
183	-0.3Nb-0.8Mo-0.025Zr-0.05C	585	893	9.2	442	476	51.8
184	-0.1Nb-0.8Mo-0.025Zr-0.05C	435	705	7.0	430	462	52.2

[a]Except where noted, all alloys were heat treated 1h at 700°C before punching specimens, then specimens were annealed for 1 h at 700°C.
[b]Tested in air at a strain rate of 3.3×10^{-3}/s.
[c]YS=0.2% yield strength, FS=fracture strength.

based alloys,[19] RT ductility tends to decrease with any addition which increases strength. Note in Table I that the RT tensile strength of FA-168B is almost double that of FA-129, but the ductility has decreased to near 10%. A compromise between RT strength and ductility must therefore be forged.

Improvements in High Temperature Properties

With increasing temperatures above about 600°C, the strength of iron aluminides decreases drastically, while the elongation increases. Along with low RT ductility, the decrease in strength above 600°C is a factor which has restricted the use of these materials for structural applications at these high temperatures where their excellent corrosion resistance would be very beneficial. Studies are underway at ORNL to improve the high-temperature strength properties through macro- and microalloying additions and improved fabrication techniques. The results of past studies of the effect of alloying on the properties of iron aluminides have been reviewed previously.[3,24] High temperature tensile and creep strengths have been shown to be improved with additions of elements such as Ti, Mo, Zr, Hf, or Nb, but at the expense of room temperature ductility.[1-3,25,26] With boron or carbon, these elements also form precipitates which are potent strengtheners and grain size refiners.[1-3] With the addition of several elements together, synergistic effects on metallurgical properties become very important, while at the same time becoming more difficult to determine.

Table I shows that the same elements added for RT strength also produce improved tensile strength at 600°C. In particular, the table shows that the maximum strengths are obtained for alloy compositions which include at least 0.5% Nb and 0.6% Mo, in combination with a small amount of zirconium. Using a combination of alloying and microstructural-modification techniques, alloys have been produced in experimental-size castings (450 mg melts) which have RT ductilities of over 10% and maintain a tensile strength of over 500 MPa to a temperature of 600°C.

Ordered alloys are generally considered to have higher creep resistance than random alloys due to the restricted movement of dislocations in the ordered crystal structure. However, the Fe_3Al binary alloy has been known for some time to have very poor high-temperature creep resistance compared to other ordered or iron-based alloys.[3,20,26-28] This discrepancy points out the lack of understanding about the actual mechanisms that cause creep in ordered alloys in general, and in the iron aluminides in particular. The importance of additional effects, such as non-stoichiometry, anti-phase domain boundaries, and the nature and structure of superdislocations and grain boundaries, and their role in deformation and fracture behavior, are unknown. Alloy FA-129 (Fe-28Al-5Cr-0.5Nb-0.2C, at.%), developed at ORNL, has good tensile strength (≥400 MPa) at temperatures to approximately 650°C, with a room temperature ductility of 15-20% (refs. 4, 29). However, for many applications its low creep-rupture strength (15-20 h life at 593°C and 207 MPa, see Table II) is not adequate.[20] The addition of molybdenum and a small amount of zirconium, in combination with the niobium already present, has resulted in a dramatic improvement in creep-rupture life to over 100 h (see Table II).[26,30]

A measure of the hot-crack susceptibility by the Sigmajig test is used as a means of comparing the weldabilty of the iron aluminides.[31] This test uses a preapplied transverse stress during autogenous gas tungsten arc welding of a 50 by 50 mm sheet specimen to determine the threshold cracking stress (TCS), above which hot-cracking is initiated in the fusion zone during welding. Using this test, the weldability of FA-129 was determined to be better than that of nickel aluminides and comparable to several other commonly-used alloys.[20] Its weldability has been improved still further by optimizing the welding process and parameters (e.g., using pre- and post-weld heat treatments to prevent cold-cracking).[32] The alloying additions used to improve

Table II. Creep-rupture at 593°C and 207 MPa and hot-crack susceptility as measured by the Sigmajig test for Fe_3Al-based alloys.

Alloy[a] FA-	Nominal composition (at.%)	Creep properties Life (h)	Elongation (%)	MCR (%/h)	TCS[b] [MPa (ksi)]
129	Fe-28Al-5Cr-0.5Nb-0.2C	16.8	68.9	1.5	=172 (25)
	Fe-28Al-5Cr-0.005B				
168B	-0.5Nb-0.8Mo-0.05Zr-0.03C	130.8	69.9	0.2	<<138 (20)
176B	-0.5Nb-0.4Mo-0.025Zr-0.05C	62.2	62.0	0.4	103-138 (15-20)
179	-0.5Nb-0.6Mo-0.025Zr-0.05C	119.5	72.9	0.2	131 (19)
180	-0.5Nb-0.8Mo-0.025Zr-0.05C	97.1	76.8	0.3	124 (18)
183	-0.3Nb-0.8Mo-0.025Zr-0.05C	41.2	58.6	0.8	<96 (14)
184	-0.1Nb-0.8Mo-0.025Zr-0.05C	19.0	65.0	1.7	>138 (20)

[a]Except where noted, all alloys were heat treated 1h at 700°C before punching specimens, then specimens were annealed for 1 h at 700°C.
[b]Threshold cracking stress for initiation of hot-cracking during gas-tungsten-arc welding.

mechanical properties have resulted in slightly reduced weldability, as well as lower room temperature tensile ductility. Further efforts have therefore been necessary to produce a Fe_3Al-based composition with an acceptable combination of room temperature ductility, high temperature strength, and weldability. By controlling the ratio of Nb, Mo, and Zr, and adding a small amount of carbon, alloys have been produced with over 10% RT tensile ductility, tensile strengths at RT and 600°C of over 500 MPa, a creep-rupture life of over 100 h at 593°C and 207 MPa, and acceptable weldability (for example, see properties of alloys FA-179 and -180 in Tables I and II). However, the amount of carbon added is also critical; although it appears to enhance weldability, too much carbon results in dramatic decreases in high-temperature creep resistance.[20]

Commercialization Status of ORNL Alloys

Several properties of Fe_3Al-based iron aluminides make them attractive materials for structural applications. As well as the excellent oxidation and sulfidation resistance, low material cost, and low density mentioned earlier, they also have high electrical resistivity and soft magnetic characteristics similar to some nickel-based alloys. Also, the formation of Al_2O_3 on the surface by preoxidation can provide good chemical compatibility with many environments, including some aqueous environments. The major obstacle to commercialization has been the room temperature sensitivity to moisture-bearing atmospheres which results in hydrogen embrittlement. Not only does this sensitivity result in poorer mechanical properties under dynamic tensile loading, but also causes porosity during melting (especially air-melting), restricts fabrication procedures to temperatures above about 500°C, and produces cold-cracks during cooling of welds.

In order to take advantage of the unique properties of these materials, ways to minimize environmental embrittlement must be developed. Using the alloy development and thermomechanical processing techniques described above, several Fe_3Al-based alloys with RT ductilities of 10-20% and yield strengths of 500 MPa at temperatures up to 600°C have been produced in experimental-size castings (0.5-7 kg).[4,21,29] This improvement in properties makes Fe_3Al-based alloys more competitive with conventional austenitic and ferritic steels. Several alloy compositions developed at ORNL and licensed to commercial vendors for manufacturing are listed in Table III. The licensees are Ametek Specialty Metal Products Division for powder production, Hoskins Manufacturing Co. and Harrison Alloys for production of heating elements, and Cast Masters for production of cast parts. These and other vendors have produced various sizes of pilot heats, including heats of 2000 and 3000 kg melted by Precision Rolled Products.[33,34]

With these companies and others, several specific applications are being pursued,[21] all of which will result in reduced cost and lower use of strategic elements. These include:

Table III. Compositions of iron aluminide alloys currently being commercialized.

Element	Nominal composition (at. %)				
	FAS[a]	FAL[b]	FA-129[c]	FAP[d]	FAPY[d]
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]Good RT tensile ductility.
[c]Good high-temperature strength with good RT ductility.
[d]High RT ductility.

1. Heating elements for toasters, stoves, ovens, cigarette lighters, and dryers, where the iron aluminide could replace Nichrome wire. For this application, Fe_3Al has the advantages of improved oxidation resistance and significantly superior thermal cycling resistance.

2. High-temperature insulation wrapping wire for investment-casting molds. The wire needs to operate reliably at temperatures up to 1100°C in holding the insulation wrapped around the mold. Fe_3Al could replace the stainless steel wire currently used and should result in a lower rejection rate of investment-cast parts because of its improved oxidation resistance and longer life.

3. Regenerator discs in automotive gas-turbine engines. Discs are used to recover heat from the hot gases of gas-turbine engines in buses and trucks. This application could replace a nickel alloy containing cobalt and offers the advantages of reduced engine weight and longer life.

4. Hot-gas filters in the coal-gasification process. These filters are used to trap the particulate matter from the gas produced in the coal-gasification process.

5. Dies for superplastic forming of titanium-based alloys. The presence of nickel in the conventional die materials embrittles the titanium-based alloys. Iron aluminides could replace the nickel-base alloys and could provide better oxidation resistance, lower die cost, and longer life, and might result in improved quality of superplastically-formed parts.

6. Shields for protecting superheater tubing from oxidation and erosion in coal-fired power plants. The currently-used stainless steel shields must be replaced every two years. Iron aluminides could provide lower cost, longer life, and reduced down time to replace shields.

7. Exhaust manifolds, catalytic converters, mufflers, and exhaust support hangers for automotive applications. The iron aluminides could replace ceramics in the catalytic converters and the 400 series iron-chrome steels in the other applications. For these applications, they offer better oxidation and aqueous corrosion resistance, lighter weight with improved energy efficiency, longer component life, and lower cost.

8. Other applications such as sensors in molten metal processing systems, nickel-free alloy jewelry, and structural uses in highly oxidizing salts (such as nitrates) are being considered.

Additional work is needed to improve the ductility and strength of Fe_3Al-based alloys in both the as-cast and wrought conditions. Also, efforts are needed to develop welding wire, coated electrodes, and fluxes for welding procedures. A data base of physical and mechanical property data needs to be generated on those alloys being produced commercially (Table IV) and on the high-strength alloys currently being developed.

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