EFFECT OF THERMOMECHANICAL PROCESSING ON MECHANICAL

PROPERTIES OF Fe-16 at. % Al ALLOY*

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Abstract

An iron-aluminum alloy containing 16 at. % Al, which is essentially free from environmental effect on its ductility, has been developed. This alloy has over 20% elongation at room temperature. This paper presents in detail the effect of vacuum versus air melting on the properties of Fe-16 at. % Al alloy. The comparative results have shown air-induction melting to produce lower room-temperature ductility for the identical processing steps. Additional processing steps required to improve the ductility of air-melted material are also identified.

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*Research sponsored by the U.S. Department of Energy, Office of Fossil Energy, Advanced Research and Technology Development Materials Program [DOE/FE AA 15 10 10 0, Work Breakdown Structure Element ORNL-2(H)], under contract DE-AC05-84OR21400 with Martin Marietta Energy Systems, Inc.

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Introduction

The oxidation and sulfidation resistances of iron aluminides are the primary driving forces for their development (1). There are many old references in the literature which suggest that the failure to commercialize Fe₃Al-base materials has primarily been due to their poor room-temperature ductility and low strength at temperatures exceeding 600°C (ref. 2). The environmental effect associated with the formation of hydrogen from the reaction of aluminum in the alloy with moisture in air has been identified as the primary cause for the poor ductility of Fe₃Al-base alloys (3). The reduction of aluminum content to 16 at. %, which results in a primarily disordered alloy, has been observed to essentially eliminate the environmental-related embrittlement. The purpose of this paper is to describe the development of an Fe-16 at. % Al-base alloy and present results on the effects of melting practice on its properties.

Alloy Identification

The FAP and FAPY are two versions of low-aluminum (16 at. %) alloys developed to alleviate the problems of environmental effects and low room-temperature ductility with Fe₃Al-base alloys.

The alloy compositions are given in Table I. Both alloys are disordered α -alloy with the only difference being that FAPY contains yttrium. The FAP and FAPY alloy compositions were designed (4) after a systematic study (5) of 12 alloy compositions.

Table I Chemical Compositions of Two Fe-16 at. % Al Alloys (FAP and FAPY)

Element	Alloy (%)			
	FAP		FAPY	
	Atomic	Weight	Atomic	Weight
Al	16.12	8.46	16.12	8.46
Cr	5.44	5.50	5.44	5.50
Zr	0.11	0.20	0.11	0.20
C	0.13	0.03	0.13	0.03
Mo	1.07	2.00	1.07	2.00
Y	--	--	0.06	0.10
Fe	77.13	83.81	77.07	83.71

Each element of the alloy has a specific role which is described below:

Aluminum: The aluminum content is controlled to eliminate the environmental effect. This is accomplished through minimization of hydrogen generation because of lower aluminum activity than in Fe₃Al. It is also accomplished through the transfer of ordered structure of Fe₃Al to a disordered structure, which is known to be less susceptible to hydrogen embrittlement (4).

Chromium: The chromium addition to the alloy is made to gain resistance to aqueous corrosion.

Molybdenum: Molybdenum is added to the alloy to protect against pitting corrosion.

Zirconium and Carbon: These elements are added to form high-melting-point zirconium-carbide particles to provide grain refinement during solidification.

Yttrium: Small additions of yttrium are made to enhance high-temperature oxidation resistance.

This paper presents methods of alloy preparation and processing and tensile data.

Melting

The melting of Fe-16 at. % Al alloys has been found to be most successful when advantage is taken of the exotherm (6) that occurs during the formation of FeAl (see Figure 1). The proper use of the exotherm saves energy, provides better alloy chemistry control, reduces melting time, and minimizes slag formation. The specific furnace loading required to take advantage of the exotherm during the melting of FAP and FAPY alloys is shown in Figure 2. The iron content of the alloy is divided between the top and bottom of the melt crucible. The ratio of iron to aluminum on top of the crucible is chosen to equal 50:50 at. %. The remaining iron is put at the bottom, and the alloying elements are sandwiched between the top and bottom. When the power is turned on, the entire furnace load is heated, and when the temperature reaches 660°C, the aluminum melts. The molten aluminum reacts exothermally with the iron setting next to it at the crucible top. The FeAl liquid at approximately 1500°C flows down, dissolves the alloying elements, and raises the temperature somewhat for pouring. This furnace-loading sequence has been successfully used commercially by Sandusky International for melting 2500-kg heats of nickel-aluminide alloy containing approximately the same amount of aluminum as is present in the FAP and FAPY alloys.

The crucible selection for the melting of high-aluminum alloys is also important. Figure 3 shows the free energy of formation for Al_2O_3 and MgO as a function of temperature (7). Based on this figure, the aluminum in the FAP and FAPY alloys will reduce MgO of the magnesia crucible at temperatures $\geq 1600^\circ\text{C}$. This type of reduction of MgO will promote magnesium pickup by the alloy and erosion of the crucible (8). The effects of magnesium on properties of FAP and FAPY alloys are not yet known. However, given the choice, the Al_2O_3 crucible should be preferred over the magnesia crucible.

The most effective method of adding yttrium to the melt is to use an Al-Y eutectic, which melts at approximately 600°C and is commercially available (9).

Using the process described above, two 15-lb heats of the FAPY alloy were melted, one in air and one in vacuum. In each case, the melt was cast into 75-mm (3-in.) -diam ingots. Both ingots were hot extruded at 1100°C through a die with a hole of 18 mm (3/4 in.) diam, which resulted in a reduction ratio of 16:1. Tensile test bars were machined from each of the extruded bars. Specimens were annealed at 800°C for 1 h followed by air cooling. Tensile tests were conducted in air from room temperature to 600°C.

Results

Data from Table II are plotted in Figure 4. The most significant observation is the lower ductility of the air-induction-melted (AIM) material as compared to the vacuum-induction-melted (VIM) material at room temperature. The AIM material also showed more scatter in the data at room temperature as evidenced by the values of 1 and 9% for the two tests. At $\geq 50^\circ\text{C}$, the ductility values of the AIM material matched those for the VIM material. Microstructural analyses were conducted on the AIM and VIM materials to determine the cause(s) of lower room-temperature ductility of the AIM material.

Figure 1 - Extent of exothermic reaction in aluminum binary systems with the transition metals iron, cobalt, nickel, and copper [German (6)].

Figure 2 - Furnace-loading sequence to take advantage of the exotherms in Figure 1 for melting of nickel and iron aluminides.

Figure 3 - Comparison of standard free energy of formation as a function of temperature to reveal the relative stability of MgO and Al₂O₃ during melting of the alloys (7).

Table II Tensile Properties of Hot-Extruded and Annealed Bars^a of FAPY
Alloys Melted in Air and Vacuum^b

Test temperature (°C)	Strength (MPa)		Total elongation (%)	Reduction of area (%)
	Yield	Tensile		
Air melted (Heat 15633)				
23	485	523	1.13	0.80
23	520	636	9.09	9.55
50	495	644	26.13	64.00
100	442	590	24.27	64.83
200	367	572	22.96	56.49
600	272	276	63.40	89.97
Vacuum melted (Heat 15634)				
23	514	656	21.40	52.44
100	446	612	24.00	67.41
200	361	591	24.03	64.42
600	265	269	65.55	91.31

^aSpecimens annealed for 1 h at 800°C followed by air cooling prior to testing.

^bAll of the tests were made in air at a strain rate of $3 \times 10^{-3} \text{ s}^{-1}$.

Figure 4 - Comparison of tensile properties as a function of test temperature for air-induction-melted (AIM) and vacuum-induction-melted (VIM) materials (FAPY) tested in the hot-extruded and annealed condition (1 h at 800°C followed by air cool).

The optical microstructures of the AIM and VIM materials are shown in Figure 5, and the grain size measurements in the longitudinal and transverse sections are listed in Table III. This table shows that the grain sizes of the extruded bars from the AIM and VIM ingots are nearly the same, except that near-edge sections of the AIM material in the transverse orientation showed nearly twice the grain size of the VIM material. Since yttrium is the strongest deoxidizer element, it is expected that a larger fraction of it will be oxidized during air melting as opposed to vacuum melting.

The low-magnification scanning electron micrographs (SEM) of the room-temperature-tested specimens of the AIM and VIM materials are shown in Figure 6. The higher magnification SEM photographs are shown in Figures 7 through 10. These figures show the following features: (1) the AIM specimen failed with no reduction of area as opposed to the VIM material which showed reduction of area and extensive surface deformation, (2) the AIM specimen fracture surface consisted of cleavage with some intergranular features, and (3) the VIM specimen showed cleavage with significant ductile tearing at the center of the fractured surface but only very limited ductile tearing near the specimen edge.

(a) 94-0875-2

(b) 94-0876-2

Figure 5 - Comparison of optical microstructures of air-induction-melted (AIM) and vacuum-induction-melted (VIM) materials (FAPY) in hot-extruded and annealed condition (1 h at 800°C followed by air cool).

Table III Grain Size (mm) of Vacuum- and Air-Melted and Extruded Bar^a of FAPY Melted in Vacuum or Air

Melt practice	Orientation		
	Longitudinal center	Transverse	
		Center	Near edge
Vacuum	67	56	42
Air	77	57	92

^aThe extruded bar was annealed for 1 h at 800°C prior to grain-size measurement.

The sections of room-temperature-tested specimens from the AIM and VIM materials were also subjected to microprobe analysis. The backscattered electron images of the AIM and VIM specimens are shown in Figures 11 and 12. Both specimens show the presence of precipitates. The precipitates in the VIM material are of essentially the same size and are nearly uniformly distributed, whereas precipitates in the AIM material are of elongated shape and are present as stringers. The elemental maps for iron, zirconium, and chromium for the AIM and VIM materials are shown in Figures 13 and 14. These figures show that the precipitates seen in Figures 11 and 12 are zirconium-rich and free of iron. The AIM material showed segregation of chromium at the grain boundaries [see Fig. 13(c)]. The stringers of zirconium- and chromium-rich particles at the grain boundaries are considered responsible for the mixed mode cleavage and grain-boundary-dominated fracture in the AIM material. Since the matrix and grain-boundary strength can both be reduced in the AIM material by the precipitates, the lower ductility values (Table II) are not surprising. In fact, if these precipitates are responsible for low ductility, their variation along the extrusion length could be responsible for the observed scatter in the room-temperature data for the AIM material.

However, the ductile-to-brittle transition temperature for the body-centered cubic material is known to be very sensitive to the inclusion content, size, and distribution. Data presented in this paper show that melting in air and using processing for the AIM material, similar to the VIM material, is not adequate to break down the precipitates in the FAPY alloy for it to be ductile at room temperature. Additional processing of the extruded bar, consisting of forging 50% at 1100°C followed by final rolling at 650°C to 0.76-mm (0.03-in.) -thick sheet, resulted in a significant increase in ductility of the AIM material (see Table IV). The VIM material, when subjected to the same processing steps as the AIM material, showed no change.

Summary and Conclusions

A newly developed melting method for Fe-16 at. % Al alloy (FAPY) was described. Tensile data on the AIM and VIM heats of FAPY after identical processing were presented. Optical, SEM, and microprobe analyses were carried out to explain the lower room-temperature ductility and more scatter in the data for the AIM material as opposed to the VIM material. Below are significant conclusions from the work:

1. The AIM material showed zirconium-rich particles as stringers as opposed to essentially equal size and nearly uniformly distributed particles in the VIM material.
2. The AIM material showed chromium-rich precipitates at the grain boundaries.
3. The presence of zirconium-rich particles as stringers and chromium-rich particles at the grain boundaries is considered responsible for lower room-temperature ductility of the AIM material.

(a) 3856

(b) 3855

Figure 6 - Low-magnification scanning electron micrographs of room-temperature tensile-tested specimens from: (a) air-melted and (b) vacuum-melted and extruded bar of FAPY.

(a) 3848

(b) 3853

Figure 7 - Fracture surfaces of room-temperature tensile-tested specimens from: (a) air-melted and (b) vacuum-melted and extruded bar of FAPY.

Figure 8 - Fracture surface details of room-temperature tensile-tested specimens from air-melted and extruded bar of FAPY: (a) 50 $\times$, (b) 200 $\times$, (c) 1000 $\times$, and (d) 2000 $\times$.

4. The inhomogeneous distribution of the zirconium- and chromium-rich precipitates in the AIM material may be responsible for scatter in its data.
5. Additional hot forging followed by warm working to break down the zirconium- and chromium-rich particles in the AIM material improved its room-temperature ductility to the same values as observed for the VIM material.

Acknowledgments

The author thanks K. S. Blakely and E. C. Hatfield for processing, R. H. Baldwin for tensile testing, C. R. Howell for analyzing data, E. C. George and C. G. McKamey for reviewing the manuscript, K. Spence for editing, and M. L. Atchley for preparing the manuscript.

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Figure 9 - Fracture surface details of room-temperature tensile-tested specimens from vacuum-melted and extruded bar of FAPY: (a) 50 $\times$, (b) 200 $\times$, (c) 1000 $\times$, and (d) 2000 $\times$.

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Figure 10 - Fracture surface details of room-temperature tensile-tested specimens from vacuum-melted and extruded bar of FAPY near the specimen center: (a) 50 $\times$, (b) 200 $\times$, (c) 1000 $\times$, and (d) 2000 $\times$.

(a) 3859

(a) 3860

(b) 3858

(b) 3861

(c) 3873

Figure 12 - Backscattered electron image photomicrographs for vacuum-induction-melted (VIM) material (FAPY) in hot-extruded and annealed condition (800°C for 1 h followed by air cool). (a) is at low magnification, and (b) is at high magnification.

Figure 11 - Backscattered electron image photomicrographs for air-induction-melted (AIM) material (FAPY) in hot-extruded and annealed condition (800°C for 1 h followed by air cool). (a), (b), and (c) are at increasingly higher magnifications.

(a) 3878

(b) 3875

(c) 3876

Figure 13 - (a) Zirconium, (b) iron, and (c) chromium maps for air-induction-melted (AIM) material (FAPY) in hot-extruded and annealed condition (800°C for 1 h followed by air cool).

(a) 3867

(b) 3868

(c) 3870

Figure 14 - (a) Zirconium, (b) iron, and (c) chromium maps for vacuum-induction-melted (VIM) material (FAPY) in hot-extruded and annealed condition (800°C for 1 h followed by air cool).

Table IV Tensile Properties of Extruded Bars of Air-Induction-Melted and Vacuum-Induction-Melted Materials After Additional Processing Consisting of Forging 50% at 1100°C Followed by Rolling at 650°C to 0.03-In.-Thick Sheet^a

Test temperature (°C)	Strength (MPa)		Total elongation (%)	Reduction of area (%)
	Yield	Tensile		
Air melted (Heat 15633)				
23	563	653	25.74	35.01
23	521	656	27.80	39.40
Vacuum melted (Heat 15634)				
23	559	665	21.68	23.49
23	562	662	21.00	29.04

^aSpecimens annealed for 1 h at 800°C followed by air cooling prior to testing.