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ALLOY DEVELOPMENT AND PROCESSING OF FeAl: AN OVERVIEW

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Abstract

In the last few years, considerable progress has been made in developing B2-phase FeAl alloys with improved weldability, room-temperature ductility, and high-temperature strength. Controlling the processing-induced microstructure is also important, particularly for minimizing trade-offs in various properties. FeAl alloys have outstanding resistance to high-temperature oxidation, sulfidation, and corrosion in various kinds of molten salts due to formation of protective Al_2O_3 scales. Recent work shows that FeAl alloys are carburization-resistant as well. Alloys with 36 to 40 at. % Al have the best combination of corrosion resistance and mechanical properties. Minor alloying additions of Mo, Zr, and C, together with microalloying additions of B, produce the best combination of weldability and mechanical behavior. Cast FeAl alloys, with 200 to 400 μm grain size and finely dispersed ZrC, have 2 to 5% tensile ductility in air at room-temperature, and a yield strength > 400 MPa up to about 700 to 750°C. Extruded ingot metallurgy (I/M) and powder metallurgy (P/M) materials with refined grain sizes ranging from 2 to 50 μm , can have 10 to 15% ductility in air and be much stronger, and can even be quite tough, with Charpy impact energies ranging from 25 to 105 J at room-temperature. This paper highlights progress made in refining the alloy composition and exploring processing effects on FeAl for monolithic applications. It also includes recent progress on developing FeAl weld-overlay technology, and new results on welding of FeAl alloys. It summarizes some of the current industrial testing and interest for applications.

IRON- AND NICKEL-ALUMINIDES ARE among the most attractive ordered intermetallics for large scale

commercial applications because their constituent elements are relatively abundant [1,2]. While the iron-aluminides have tended to be less ductile at room-temperature and weaker at high-temperatures compared to the nickel-aluminides [3,4], they are more attractive than nickel-aluminides from a cost standpoint, because their raw or scrap constituents are more abundant and cheaper. Both kinds of aluminides are oxidation and corrosion resistant because they form adherent, protective (compact) Al_2O_3 scales at high-temperatures [5]. The iron-aluminides, particularly FeAl alloys, are resistant to a wider range of high-temperature corrosion environments, including oxidation, sulfidation, and corrosion in molten nitrate and carbonate salts [5-7]. However, FeAl alloys have been plagued by poor mechanical properties at ambient and elevated temperatures, as well as by fabrication difficulties, including inadequate weldability.

Fe_3Al and FeAl ordered intermetallics have been the subject of considerable scientific study and some alloy development since the 1950s, especially Fe_3Al . Recent progress in developing Fe_3Al alloys has been reviewed and summarized, particularly efforts at the Oak Ridge National Laboratory [8-10]. There had been less overall effort on developing FeAl with useful properties, until the work begun by Liu et al. [11] in the early 1990's. Data from efforts at ORNL to develop corrosion-resistant FeAl alloys with improved weldability, room-temperature properties and high-temperature strength were not published until a few years ago [12-14]. The recent development of the Exo-Melt™ process for melting nickel- and iron-aluminides in a way that takes advantage of their very large exothermic heats of reaction, has created an opportunity for commercialization that did not exist before [2,15,16]. This paper overviews primarily the work that has been done at ORNL to further improve the base FeAl alloy composition that was found to have the

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best combination of weldability and mechanical properties. This paper also summarizes the large effects that processing-induced microstructures have on room-temperature mechanical properties, especially impact toughness. Finally, some potential applications are discussed.

Corrosion Resistance

The resistance of FeAl alloys to various kinds of corrosion or metal wastage is usually the primary reason for considering these new materials as alternates for standard commercial Fe-Cr or Fe-Cr-Ni stainless steels and alloys. Several recent papers have reviewed general oxidation/corrosion resistance of iron-aluminide alloys at high-temperatures or in aqueous environments at room-temperature [5,17]. Therefore, we will highlight only the important or new data on the optimized FeAl alloys that are being developed.

These current FeAl alloys based on 36 to 38 at. % Al were developed because FeAl alloys with > 30 at. % Al showed excellent resistance to molten, highly oxidizing sodium/potassium nitrate salts at 650°C [7,11]. The corrosion resistance of FeAl alloys in such nitrate salts does not appear to be affected by minor alloying additions [7]. Recently, cast FeAl was also found to have outstanding resistance to highly oxidizing molten sodium chloride/carbonate salts at 900°C (see Fig. 1) [18].

It is well known that Fe₃Al alloys are very resistant to sulfidation at 800°C and above [5,6,19], and FeAl alloys show similar excellent sulfidation resistance (see Fig. 2). With higher aluminum contents, the FeAl alloys

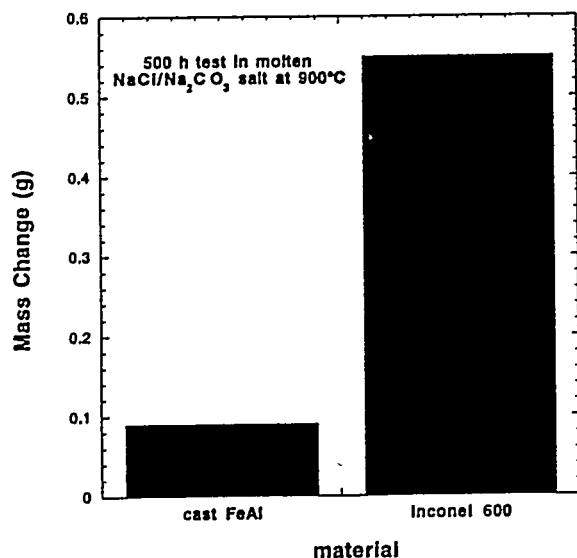


Fig.1. Mass changes of cast FeAl (FA-385) and Inconel 600 coupons exposed to molten NaCl - Na₂CO₃ salt at 900°C for 500 h [18].

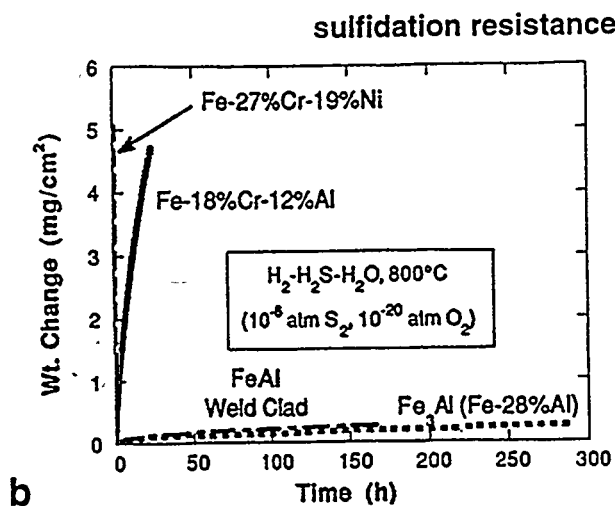
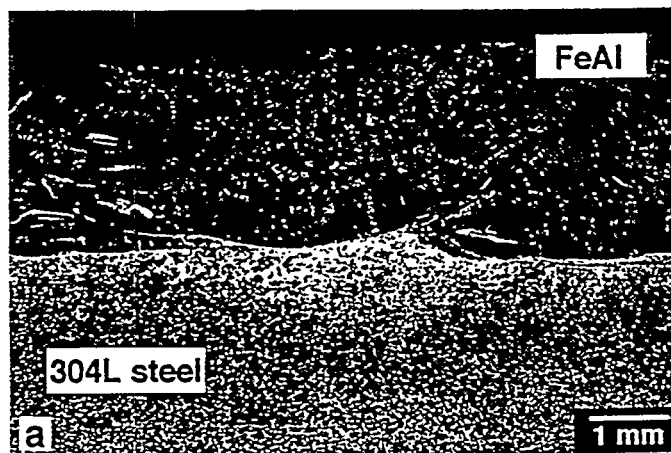


Fig. 2. (a) Multi-pass FeAl weld-overlay made on type 304L austenitic stainless steel, and (b) sulfidation resistance of a specimen made from the FeAl overlay and tested at 800°C with other materials, including Fe-Cr-Ni, FeCrAl, and Fe₃Al alloys.

also do not show the adverse effects of chromium on sulfidation resistance found in Fe₃Al type alloys [19]. Although both FeAl and Fe₃Al alloys resist the sulfidizing and oxidizing conditions found in simulated coal gasification or combustion environments at 650°C, FeAl alloys retain their corrosion resistance when HCl is introduced into that combustion environment [20]. Recent data on an P/M Fe-40Al alloy show that its sulfidation resistance at 700°C is better than premium high-performance materials like MA 956 alloy [21].

Finally, with regard to carburization resistance, both Fe₃Al and FeAl alloys are thought to have potential comparable to the excellent carburization resistance demonstrated recently by Ni₃Al alloys at 1000°C [5], and attributed to their protective Al₂O₃ scales [2]. Very recent tests of cast FeAl alloys (36 to 38 at. % Al) show that they too possess outstanding carburization resistance at 1000°C (atmosphere simulating ethylene pyrolysis) (see Fig. 3) [22].

Data of Maziasz (ORNL) and Smith (INCO), 1996

Carburization Resistance of FeAl (INCO data)

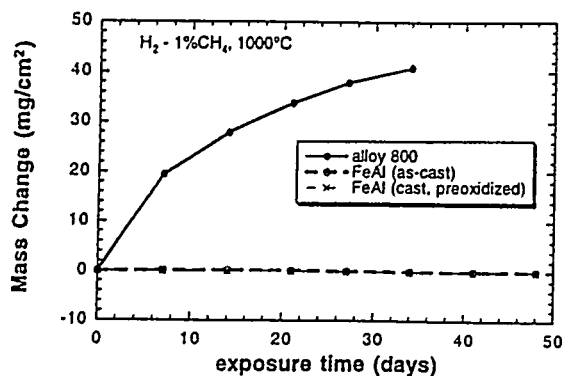


Fig. 3. Carburization testing of cast FeAl (FA-385M21 and FA-386M2) alloys, with and without preoxidation (1 h at 1200°C), at 1000°C in an atmosphere simulating ethylene pyrolysis [22].

To summarize, FeAl alloys have outstanding resistance to a variety of aggressive high-temperature gaseous or molten salt environments. Moreover, they appear to be resistant to combined environments, although more testing is needed. FeAl alloys even show some resistance to dissolution in molten aluminum at 775-800°C [12], and may be resistant to other kinds of molten metals. Clearly, there is a need to broaden the scope of high-temperature corrosion testing of FeAl alloys. Those kinds of data will then help define many of the new applications, with some idea of how much better FeAl alloys are relative to conventional steels or Fe-Cr-Ni alloys.

Weldability

Weldability is essential for almost any engineering material application, and is often a critical issue for new, advanced materials. Weldability became such an issue for FeAl when an improved Fe-36Al alloy containing Mo, Zr, and B (FA-362, with good room-temperature ductility and high-temperature strength) was found to hot-crack badly during autogenous welding tests [11,23]. FeAl developmental alloy compositions and designations are given in Table I. The Sigmajig test, developed at ORNL to quantitatively measure the threshold applied stress for hot-cracking of thin-sheet specimens [24], revealed that high levels of boron (about 0.24 at. %) were the cause of the hot-cracking [11,23]. Similar FeAl alloys without any boron (i.e. FA-372), or with carbon instead of boron (i.e. FA-385 and FA-386), were much more resistant to hot-cracking (see Fig. 4).

From previous Sigmajig studies of 300 series steels, threshold hot-cracking stress values above 100 MPa are considered good, and values of 280 MPa have been

Table I. FeAl Alloy Compositions

Alloy	Composition (at.%)							
	Al	Mo	Zr	C	B	Cr	Nb	Ti
FA-324	35.8							
FA-328	35.8		0.05		0.24	5		
FA-350	35.8		0.05		0.24			
FA-362	35.8	0.2	0.05		0.24			
FA-372	35.8	0.2	0.05					
FA-384	35.8	0.2	0.05			2		
FA-385	35.8	0.2	0.05	0.13				
FA-386	35.8	0.2	0.05	0.24				
FA-387	35.8	0.2			0.24			
FA-388	35.8	0.2		0.25				
FA-385M1	35.8	0.2	0.05	0.13	0.01			
FA-385M2	35.8	0.2	0.05	0.13	0.021			
FA-385M3	35.8	0.2	0.05	0.13		2		
FA-385M4	35.8	0.2	0.05	0.13			0.5	
FA-385M5	35.8	0.2	0.05	0.13		2	0.5	
FA-385M6	35.8	0.2	0.05	0.25		2	0.5	
FA-385M7	35.8	0.2	0.1	0.25		2	0.5	
FA-385M8	35.8	0.2	0.05	0.13		2	0.5	0.05
FA-385M9	35.8	0.2	0.05	0.25		2	0.5	0.05
FA-385M10	35.8	0.2	0.05	0.13		2	0.5	0.05
(+ 0.5 Ni + 0.3 Si + 0.016 P)								
FA-385M11	35.8	0.2	0.05	0.13		2	0.05	
(+ 0.25 W)								
FA-328M11	35.8	0.25	0.1	0.4	0.015	5	0.15	0.3
(+ 0.5 V + 1 Mn + 0.2 Si + 0.06 P)								
FA-385M21	36	0.2	0.05	0.13	0.04			
FA-386M1	36	0.2	0.07	0.28	0.02			
FA-386M2	38	0.2	0.07	0.28	0.02			
FA-30-M1	30	0.2	0.05	0.22	0.021			
FA-30-M2	30	0.2	0.05	0.22	0.021			0.05
FA-30-M3	30	0.48	0.05	0.22	0.021	2		0.05

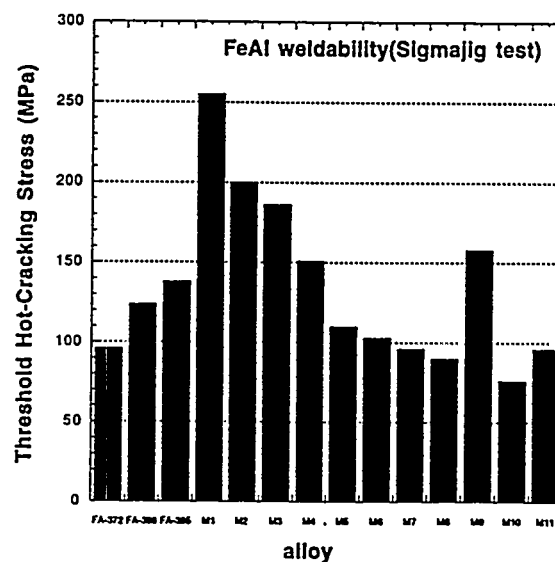


Fig. 4. Threshold hot-cracking stresses measured on 0.76 mm thick sheet specimens of various FeAl alloy compositions using the Sigmajig apparatus. Alloy compositions are given in Table I. The FA-385M1 and FA-385M2 alloys with the highest threshold stresses contain microalloying additions of boron.

measured on the most weldable steels. The two FeAl alloys containing added carbon had threshold hot-cracking stresses of 120 to 140 MPa, while the boron-free alloy was marginal at just below 100 MPa. Microalloying additions of boron (100 and 210 appm, FA-385M1 and FA-385M2, respectively, see Table I) were made to the FA-385 alloy composition, and hot-cracking resistance was dramatically improved, with threshold stresses of 200 to 250 MPa. For perspective, the most weldable Fe₃Al type alloy has a threshold hot-cracking stress of about 170 MPa, and other modified Fe₃Al alloys have values of 100 to 140 MPa [25]. Other modified FeAl alloys with good hot-cracking resistance were alloys containing additions of Cr, Nb, or Cr + Nb + Ti (Fig. 4).

Another issue related to producing sound, crack-free welds is cold-cracking. Such cracking occurs during final cooling after welding, and is accompanied by audible acoustic emission. Such cracking occurs perpendicular and adjacent to the weld on a SigmaJig test specimen, rather than along the center line of the weld [23]. Cold-cracking is related to the environmental embrittlement effects of hydrogen from moisture in the air, as well as to the inherent ductility of the alloy. As with FeAl weld-overlays, some success has been achieved with pre-heat (up to 350 to 400°C) prior to welding, but current efforts are focussed on welding FeAl alloys which have been processed to have much better ductility in air at room temperature (see following sections).

Tensile Properties at Room-Temperature

Earlier FeAl alloy development work by Liu et al. showed that both processing (extruded rod compared to rolled sheet) and minor alloying additions (mainly B) dramatically affected the room-temperature ductility of FeAl alloys in air [11], as shown in Fig. 5. Ductility of FeAl alloys in air tends to be quite low due to hydrogen-induced embrittlement from moisture in the air at room temperature [26,27], and boron additions improve resistance to such embrittlement [28]. Moreover, significant refinements in grain size also improve the ductility of FeAl in air [9,11,12,29]. The combination of these two effects was found in the hot-extruded FA-362 alloy (Fig. 5) [11], with almost 12% total elongation in air, compared to the hot-rolled FA-385 alloy, with a much coarser grain-size, no boron, and only about 3% elongation.

To attempt to separate processing/microstructure effects from alloying effects, ductility in ambient air was measured for the weldable FA-385 base alloy in different processing conditions, including hot-rolled sheet, hot-extruded bar, as-cast ingots, and P/M material consolidated by direct hot-extrusion [12] (Fig. 6a). The as-rolled sheet was very brittle in air, but had over 10% ductility in oxygen, suggesting a very strong environmental effect. Heat-treatment of that same sheet for 1 h at 900°C

improved the ductility in air to about 2% and the ductility in oxygen to 15%. Heat-treatments in air at 700°C and above produce thin oxide films that lessen the embrittling effect of hydrogen [9]. Hot-extruded FA-385, with a recrystallized grain size of about 50 µm, had about 8% ductility in air, and 12.5% in oxygen, with clearly much better resistance to environmental embrittlement. Cast FeAl, even with a much coarser grain size of 260 µm, still had 2% ductility in air. Finally, P/M FeAl with a 9 µm grain size had over 9% ductility, and had the best resistance to environmental embrittlement of this group.

The strength of FeAl in ambient air is only meaningful when the material has some ductility (Fig. 6b). Brittle FeAl, like the hot-rolled sheet with 0.1% elongation, fractures just about at the yield stress (YS). FeAl alloys show high work-hardening and almost no necking at room-temperature, so that all of the measured elongation is uniform elongation, and the materials fracture at their ultimate tensile strength (UTS). The FA-385 base alloy, when processed to have some ductility, also has a YS (375 to 425 MPa) that does not depend strongly on fabrication history, except for the P/M FeAl which is stronger (500 MPa). This preliminary comparison demonstrates dramatically that fabrication conditions and processing-induced microstructure have large effects on room-temperature ductility of FeAl for a given alloy composition. Therefore, alloying effects on room temperature mechanical properties need to be considered within the context of processing history. For the remainder of this section, we will separate cast, hot-extruded I/M, and hot-extruded P/M processing effects, and compare alloying effects within those categories.

CAST MATERIAL - An earlier study of cast Fe₃Al alloys (Fe-28Al) with the DO₃ structure had shown low ductility and strength in air [30]. By contrast, the same Fe₃Al alloys showed much better ductility when wrought-processed to retain the B2 phase [8,31,32]. Studies of environmental embrittlement on fatigue-crack-growth in Fe₃Al alloys also suggested that the B2 phase had better resistance to environmental embrittlement than the DO₃ phase [9,33,34]. Therefore, the ductility of as-cast FeAl (Fe-36Al) alloys that were entirely B2 phase (and non-magnetic) and showed the best weldability (FA-385, -385M1 and -385M2) was measured in air at room-temperature (Fig. 7). Several Fe-30Al alloys with minor alloying additions similar to the Fe-36Al alloys, but with a mixture of B2 and DO₃ ordered phases (hence somewhat ferromagnetic) were also included for comparison.

The base as-cast Fe-36Al alloy (FA-385) showed about 2.5% ductility in air, and about 8% ductility in oxygen (Fig. 8), with a YS of a little less than 400 MPa (Fig. 9). Microalloying with 100 to 210 appm boron increased the ductility in air, but decreased the ductility in oxygen because those boron-doped alloys were also stronger. The boron-doped alloys showed more resistance

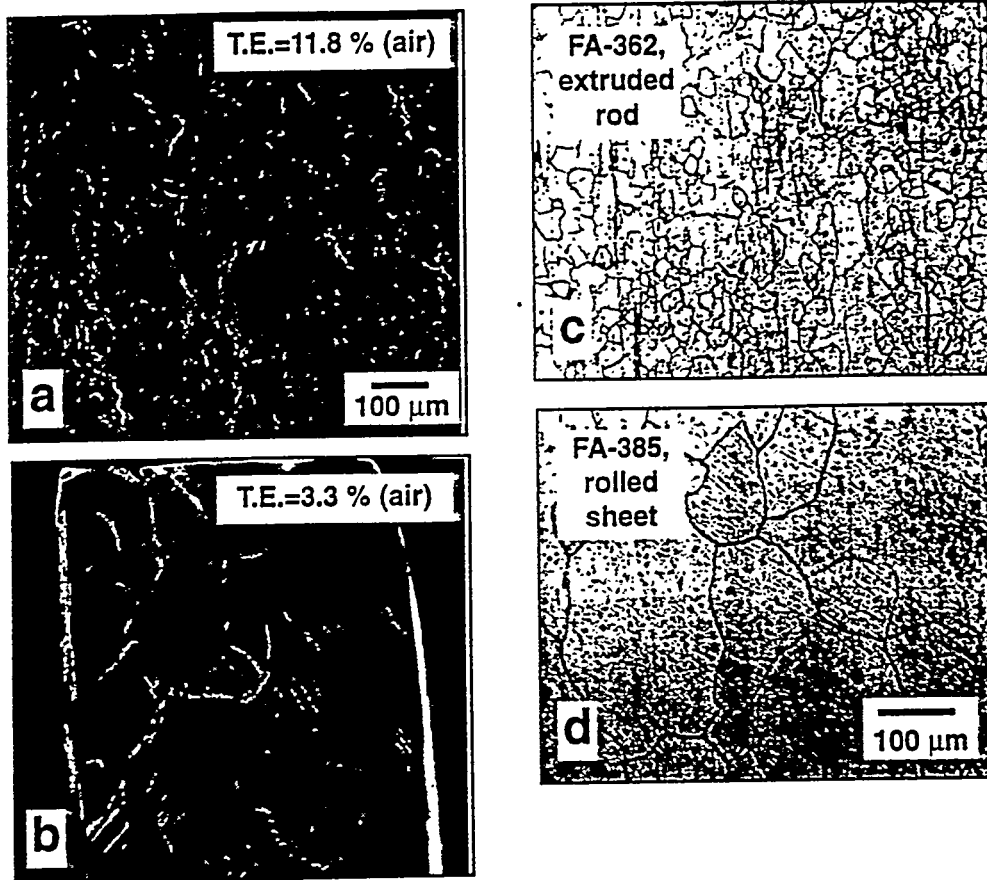


Fig. 5. Fracture surface (SEM), (a) and (b), and longitudinal grain size (metallography), (c) and (d), of two FeAl alloys tensile tested at room temperature in air. The FA-362 alloy (Fe-36Al + Mo, Zr, B) hot-extruded at 900°C has significantly refined grain size and fracture features, and high ductility of 11.8%. The FA-385 alloy (Fe-36Al + Mo, Zr, C) hot-rolled at 900 to 1000°C has coarser grain size and fracture features, and only 3.3% ductility.

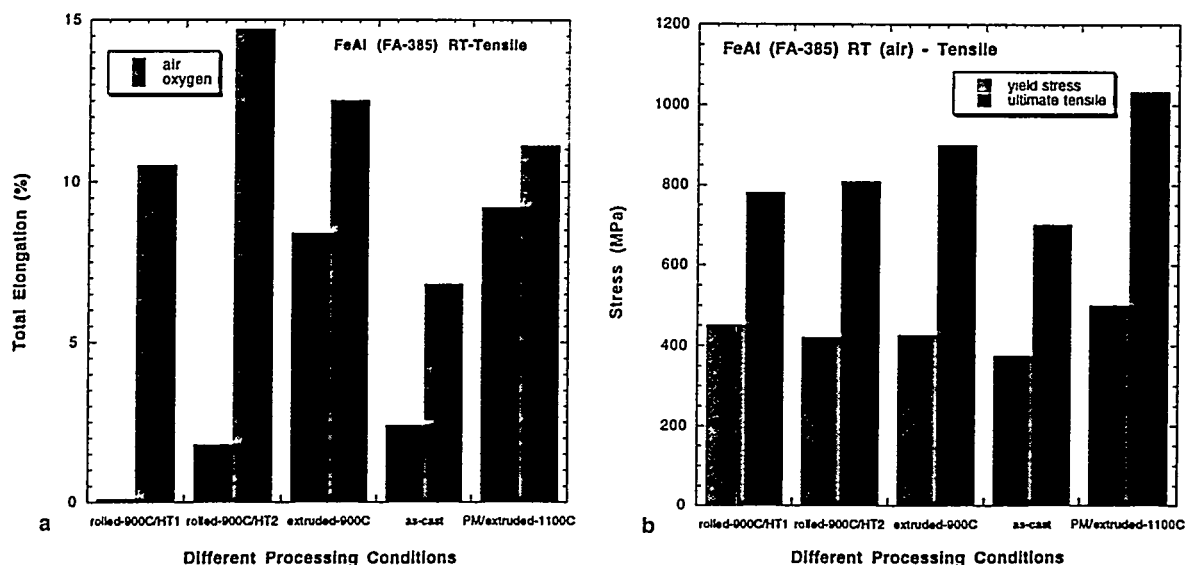


Fig. 6. (a) Ductility and (b) yield and ultimate tensile strength of FeAl (FA-385) alloy tensile tested at room-temperature in air (and oxygen for ductility) in various fabrication conditions. Sheet hot-rolled at 900°C and then heat-treated for 1 h at 900°C (HT1) or 1 h at 1000°C (HT2), I/M rod hot-extruded at 900°C, as-cast material, and P/M rod hot-extruded at 1100°C cover the range of different fabrication conditions for the same alloy.

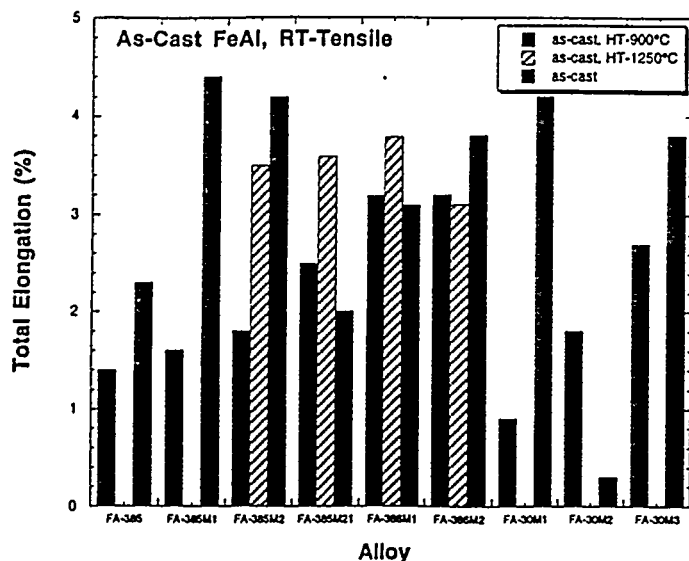


Fig. 7. Tensile ductility for various heats of as-cast FeAl alloys tested in air at room-temperature. Specimens were either stress-relieved after machining for 1 h at 750°C, or heat-treated in air for 1 h at 900°C or 1250°C. The alloys included Fe-36Al alloys with and without boron (FA-385, -385M1, -385M2), Fe-36 and -38Al alloys with more boron or more C and Zr (FA-385M21, -386M1, -386M2), and Fe-30Al alloys (FA-30M1, -30M2, -30M3).

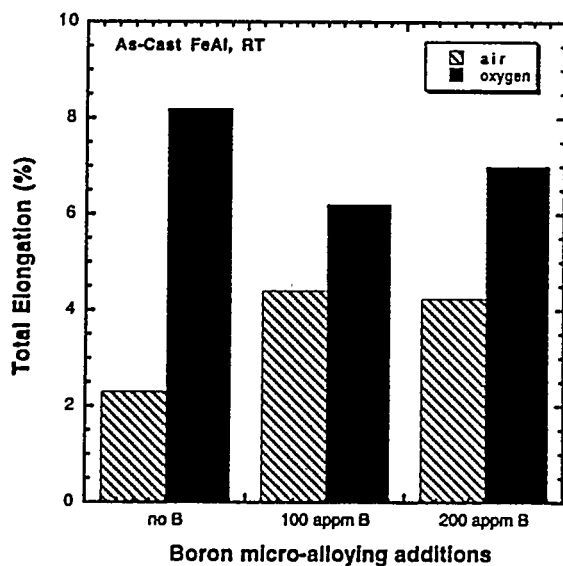


Fig. 8. Effect of boron microalloying additions on environmental embrittlement of as-cast FeAl (FA-385), determined by tensile testing at room-temperature in air and in oxygen.

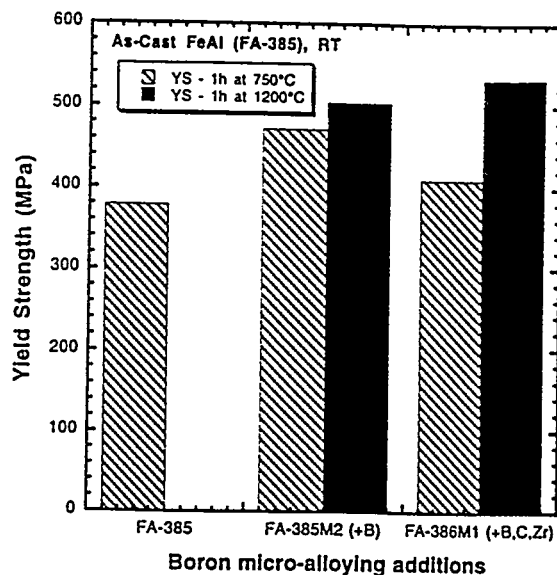


Fig. 9. Effects of microalloying additions and heat-treatment on yield strength of as-cast FeAl (FA-385) alloys tensile tested in air at room-temperature.

to environmental embrittlement because there was less difference between ductility in oxygen and air. Boron microalloying additions changed the fracture mode from intergranular (boron-free) to transgranular (100 to 210 appm B). The boron additions also affected the microstructure by refining the grain size (from 260 μm to 100 to 120 μm , see Fig. (10), and enhancing the precipitation of fine ZrC (Fig. 11). These microalloying effects are quite complex, because increasing the boron level from 200 to 400 appm (FA-385M21) alone lowers the ductility slightly, whereas increasing both the carbon and zirconium levels together keeps the ductility at 3 to 4% (alloys FA-386M1 and -386M2). When alloying additions similar to those found in the FA-385M2 alloy were made to an Fe-30Al base alloy, the result was about 4% ductility in air with a YS of close to 500 MPa (Fig. 7). However, a minor addition of titanium makes that alloy very brittle, whereas adding chromium and more molybdenum together with the titanium makes the properties similar to those of the FA-30M1 alloy.

Finally, heat-treatments have important effects on the room-temperature tensile properties of FeAl alloys. A heat-treatment of 1 h at 750°C relieves residual stresses caused by specimen machining, and preoxidizes the surface, which provides some protection from moisture-induced embrittlement during testing in air [9]. A heat-treatment of 1 h at 900°C has been found to improve the ductility of as-cast Fe_3Al alloys [30], and of hot-rolled FeAl sheet specimens (Fig. 6a) in ambient air. However, heat-treatments of 1 h at 1150 to 1250°C improve the high-temperature strength and creep-resistance of Fe_3Al and FeAl complex alloys [12,35]. As seen from Fig. 7, a heat-treatment of 1 h at 900°C either degrades (FA-385, -385M1, -385M2, FA-30M1, and -30M3) or has little

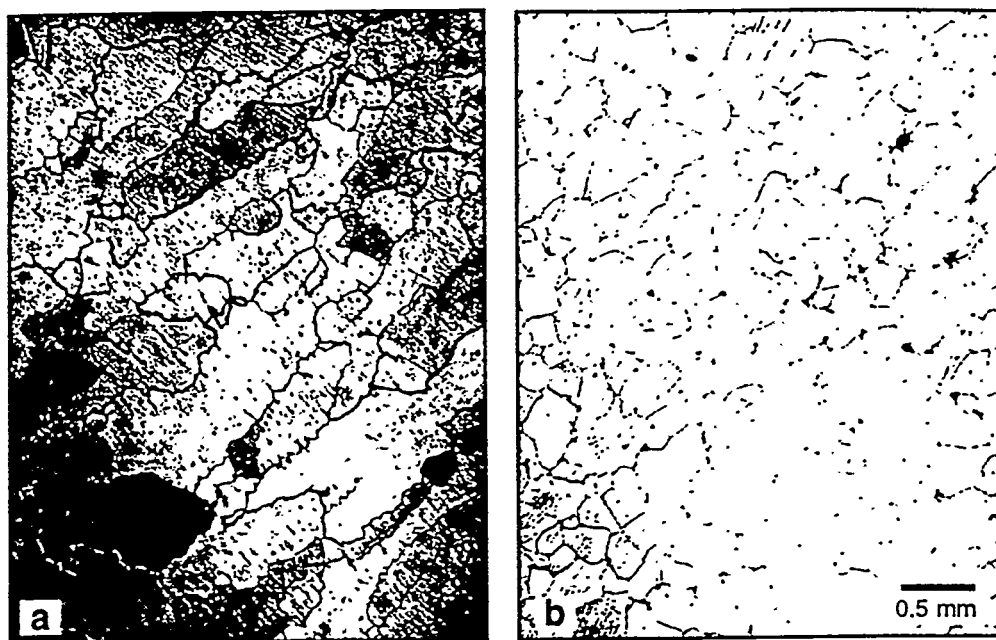


Fig. 10. Metallography to show grain sizes of as-cast FeAl alloys (a) without boron (FA-385), and (b) with 210 appm added boron (FA-385M2).

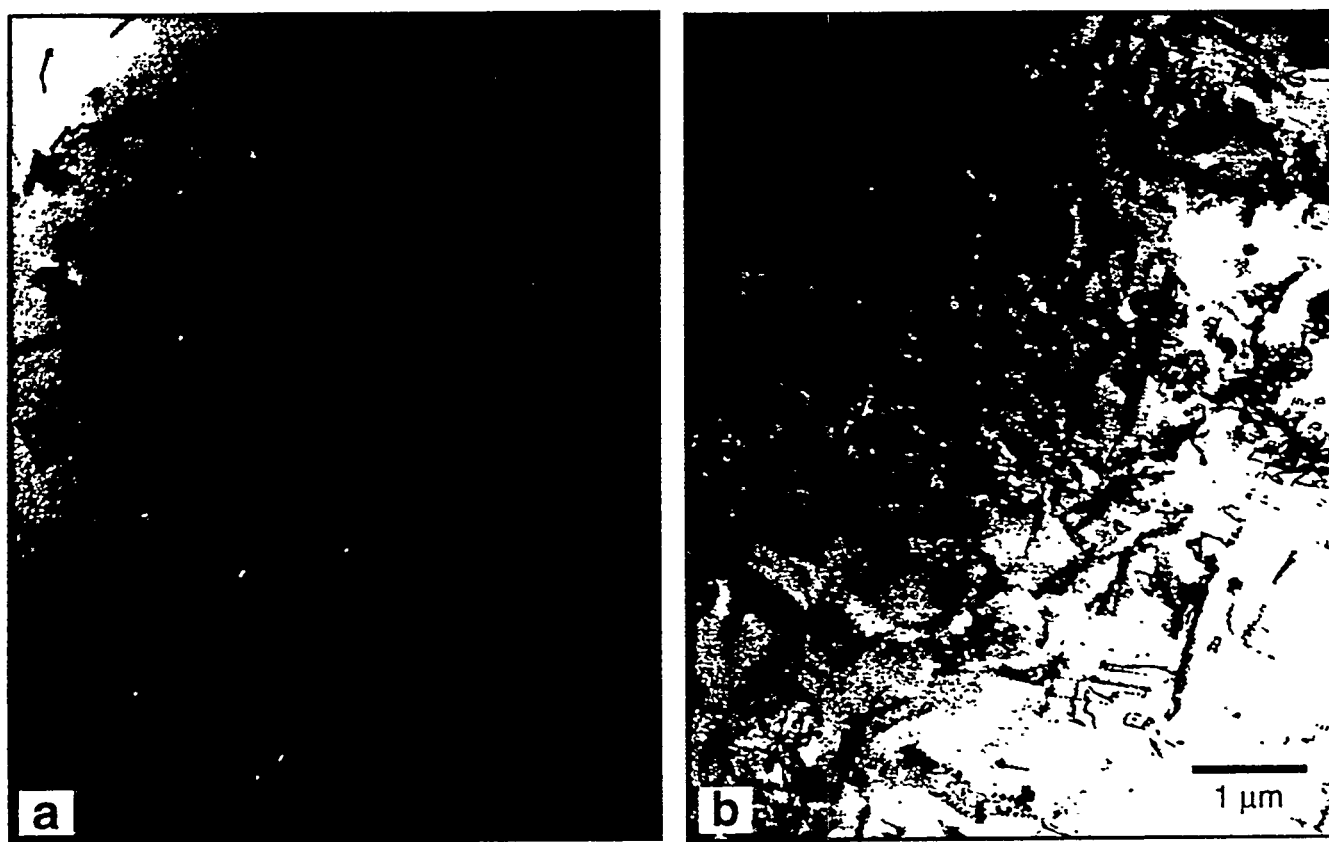


Fig. 11. TEM microstructures to show dislocation and ZrC precipitation along dislocations of as-cast FeAl (FA-385) alloys (a) without boron (FA-385), and (b) with 210 appm added boron (FA-385M2).

effect (FA-385M21, -386M1, and -386M2) on ductility for most of these alloys, but does improve the previously brittle FA-30M2 alloy. The heat-treatment of 1 h at 1200°C either does not change or improves (FA-385M21), the ductility of the higher aluminum FeAl alloys doped with boron and/or more carbon and zirconium. Heat-treatments of 1 h at 900 or 1200°C either boost the YS a bit, or have no effect, depending on the alloy. Fe-36 Al alloys with boron or more carbon and zirconium added have YS > 500 MPa after 1 h at 1200°C (Fig. 9), in addition to > 3% ductility.

HOT-EXTRUDED I/M MATERIAL - As in work by Liu et al. [11] on earlier Fe-36 Al alloys (i.e., FA-362), hot-extrusion at 900°C or above produces material with a significantly refined grain size. The weldable FA-385, -385M1 and -385 M2 alloys were cast as extrusion billets, and extruded at a 3:1 reduction ratio at 900°C. Extensive studies of hot-rolled sheet specimens of the various modified Fe-36Al alloys (FA-385M1 through -385M11, Table I) showed that the two boron-doped alloys had far better strength and ductility than any of the other alloys tested in air or oxygen [12]. Hot-extrusion at 900°C produced material with a uniform, recrystallized grain size of about 50 μm for the boron-free alloy, and about 35 μm for the two boron-doped alloys (Fig. 12a) [36]. TEM analysis reveals that these extruded materials have more dislocation networks, but none of the finer ZrC precipitates found in as-cast material.

The boron-free FA-385 alloy has about 8% ductility in air, and the boron-doped alloys have about 10% (12-15 % in oxygen) (Fig. 13a). The boron-free alloy exhibited mainly intergranular fracture, but the boron-doped alloys fractured by transgranular quasi-cleavage. All of these alloys had similar YS of 400 to 450 MPa, but had high UTS values of 800 MPa or more (Fig. 13b), due to their better ductility relative to cast material. The importance of these high values of uniform elongation is that these FeAl alloys can now be significantly deformed in air without cracking, as shown in Fig. 12b. Such behavior was impossible for hot-rolled sheet material, and is quite atypical of FeAl alloys.

HOT-EXTRUDED P/M MATERIAL - Recently, it was discovered that P/M FeAl (nominally FA-385, but actually closer to the FA-386M2 composition in Table I, but without the boron) consolidated to be fully-dense by direct hot-extrusion at 950 and 1000°C had > 12% total elongation in air, high strength (YS - 600 to 670 MPa, UTS > 1100 MPa), and a *ductile fracture mode* [12,36,37] (Figs. 14 and 15). In oxygen, these alloy have 20 to 30%

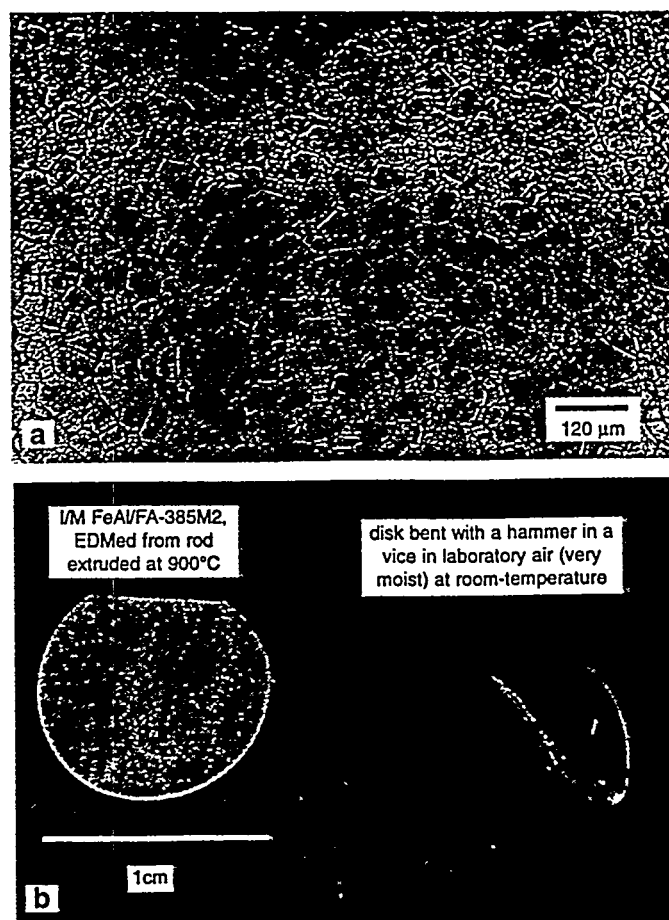


Fig. 12. (a) Metallography showing the refined and recrystallized grain structure of boron-doped FeAl alloy (FA-385M2), and (b) deformation of a small disc of that material in air at room-temperature without cracking.

ductility. Hot-extrusion at a 12:1 reduction ratio at 950 and 1000°C appears to be just above the recrystallization temperature of this FeAl alloy, which produces an ultrafine (2 to 5 μm) equiaxed grain structure (Fig. 16a). However, such good mechanical behavior appears to involve more than just grain-size refinement. The surfaces of the original powder particles had a thin Al_2O_3 film that remains even after significant deformation of the particles during consolidation at 950 and 1000°C. That oxide film still remains as an "envelope" around the many fine grains found within the prior powder particles after hot-extrusion. During tensile testing, those boundaries separate while material inside those particles fractures by microvoid coalescence (Fig. 15). The ultrafine FeAl grains also contain finely dispersed ZrC precipitates (Fig. 17a).

The same FeAl alloy extruded at 1100°C, by comparison, had a coarser grain size (9 to 10 μm), coarser intragranular ZrC and finely dispersed dislocation loops, and coarser oxides along prior powder particle boundaries

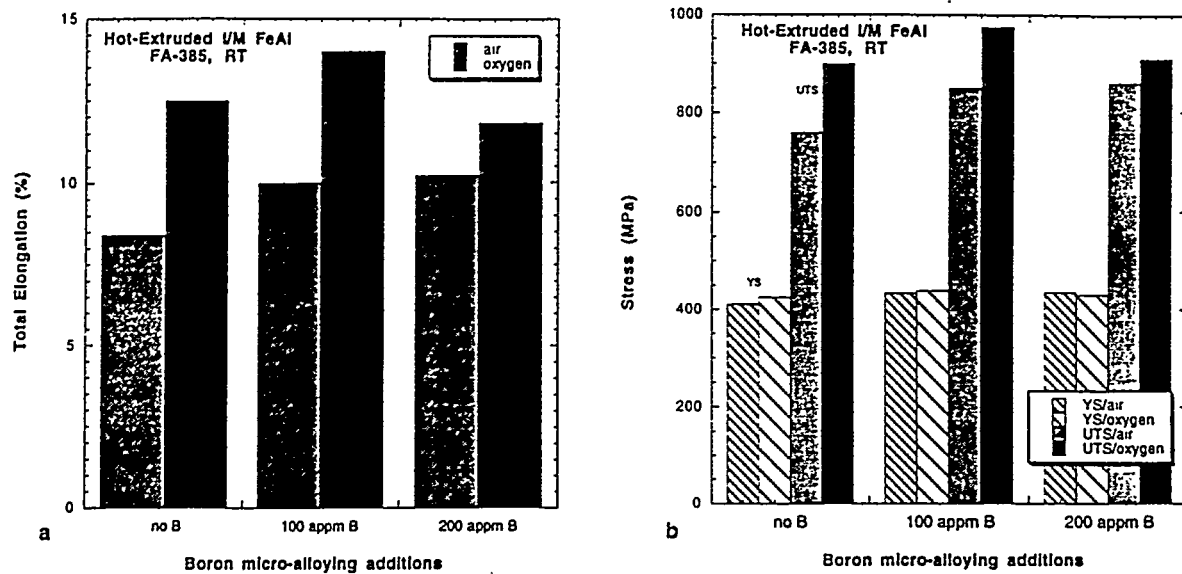


Fig. 13. Tensile properties at room-temperature of hot-extruded I/M FeAl alloys with (FA-385M1 and -385M2) and without (FA-385) boron additions tested in both air and oxygen. (a) total elongation, and (b) YS and UTS.

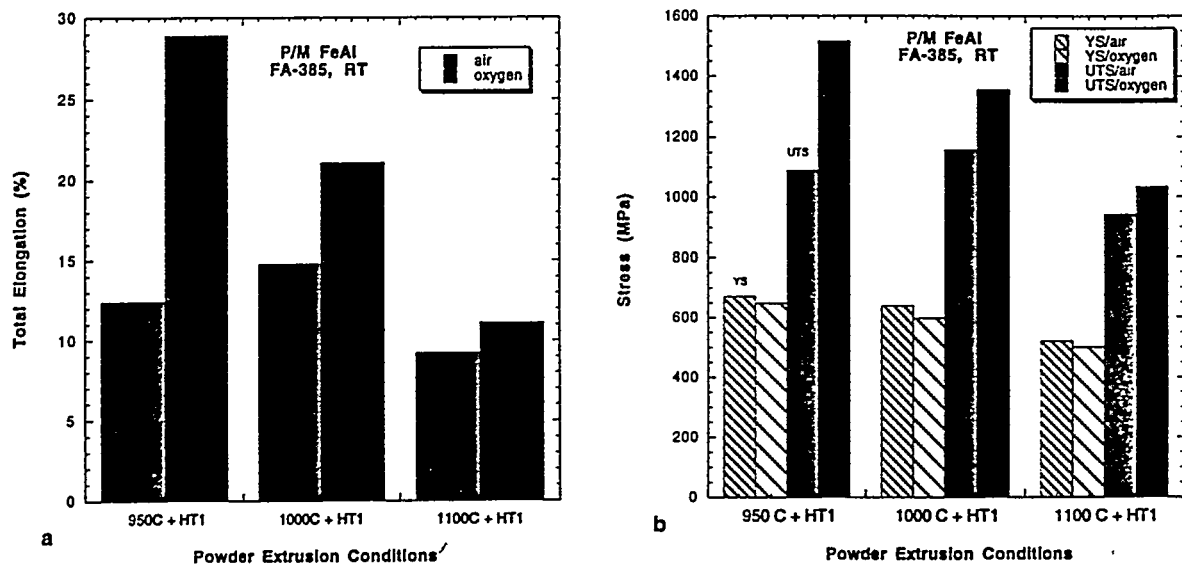


Fig. 14. Tensile properties at room-temperature of P/M FeAl (FA-385) alloys consolidated by direct hot-extrusion at 950, 1000 and 1100°C, and tested in both air and oxygen. (a) total elongation, and (b) YS and UTS.

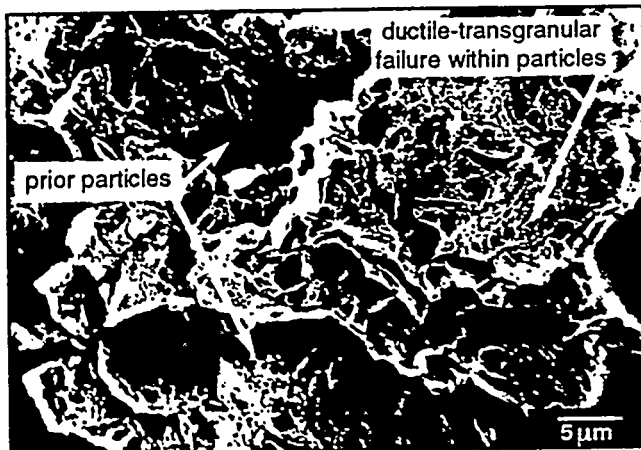


Fig. 15. SEM of fracture surface of P/M FeAl (FA-385) hot-extruded at 950°C and tensile tested in oxygen at room temperature. It shows two modes of fracture, separation along prior powder particle boundaries, and transgranular ductile-dimple fracture within such particles.

instead of “envelopes” (Figs. 16b and 17b). P/M FeAl/1100°C still has good mechanical properties compared to I/M FeAl in other processing conditions, but not as good as the P/M FeAl extruded at lower temperatures. This material has less ductility in air (9%) and strength ($YS = 500$ Mpa) (Fig. 14), and fractures along grain boundaries or by transgranular cleavage with

no secondary cracking along prior particle boundaries [36]. Consistently, reheat-treating the P/M FeAl/950°C material for 1 h at 1100°C produces a properties/microstructure change that is somewhat comparable to that found after direct extrusion at 1100°C [36]. Although complicated and not completely understood, these data definitely establish that high

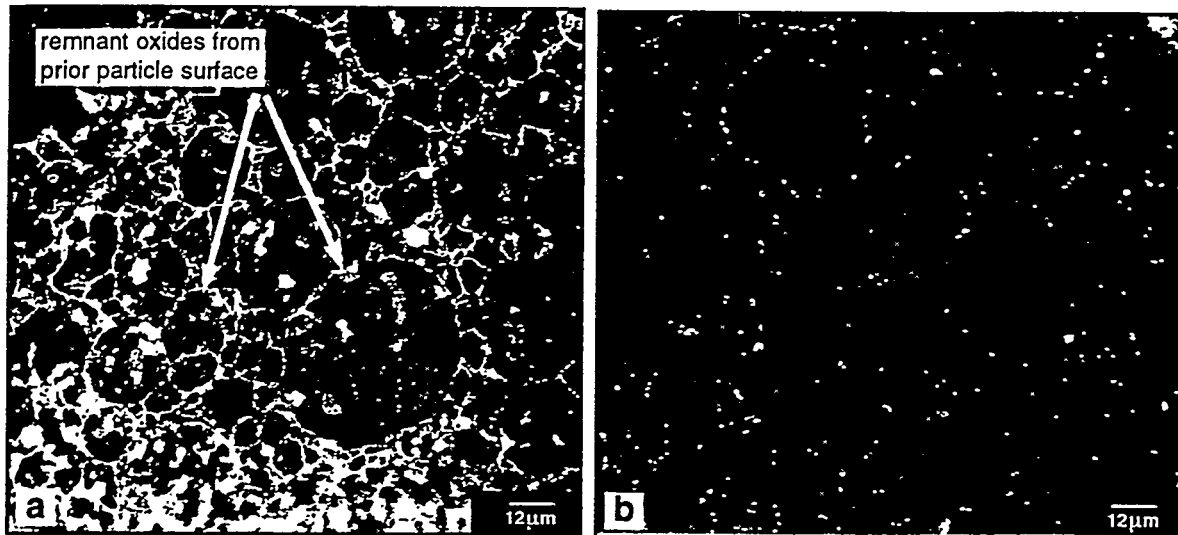


Fig. 16. SEM of electro-polished surfaces of TEM specimens of P/M FeAl alloys, cut transverse to the extrusion direction, for material hot-extruded at (a) 950°C, and (b) 1100°C. Bright, circular features are the remnant oxides from the surfaces of prior powder particles.

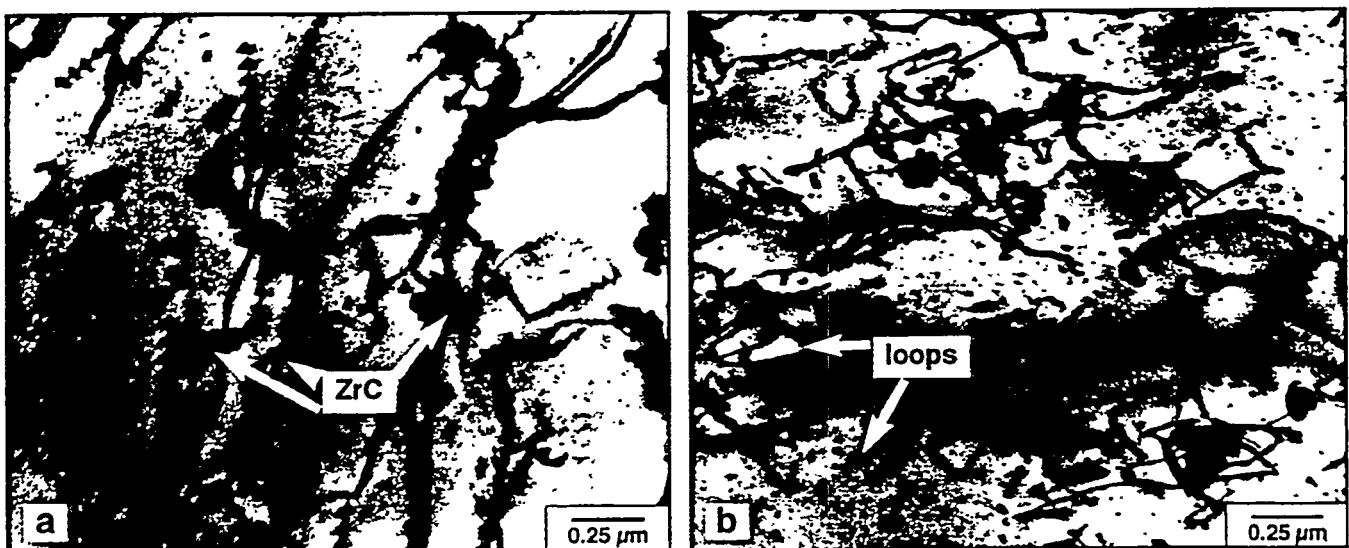


Fig. 17. TEM of the intragranular dislocation and precipitate (ZrC) microstructures found in P/M FeAl alloys hot-extruded at (a) 950°C, and (b) 1100°C.

ductility and strength are possible for FeAl alloys, and demonstrate that special processing-induced microstructures are the key to such properties.

Impact Toughness at Room Temperature

There are very few studies of impact toughness of FeAl alloys, because generally they have poor toughness. Charpy impact tests at room-temperature of a boron-modified Fe-36 Al alloy (FA-350) with 2 to 4% ductility in air ($> 100 \mu\text{m}$ grain size) showed only 3 to 5 J absorbed energy [38]. Impact toughness depends on, among other things, grain size and fracture mode. To date, Pocci et al. [21] reported the highest impact toughness values of 53 to 55 J/cm² for Fe-40 Al alloys isothermally forged to create ultrafine grained ($1 \mu\text{m}$) necklaces around an otherwise coarser-grained (240 to $280 \mu\text{m}$) microstructure. The same material with uniform, equiaxed grain-sizes of 74 and $162 \mu\text{m}$ drops to toughness values of 39 and 6 J/cm², respectively, so that toughness is extremely sensitive to processing-induced microstructure.

Charpy impact tests on hot-extruded I/M FeAl alloys developed at ORNL showed 25 J (31 J/cm²) at room-temperature for the FA-385 base alloy, and 63 J (78 J/cm²) for the alloy doped with 210 appm boron (FA-385M2) (Fig. 18). The big difference in impact toughness for these alloys appears related to the effects of boron on the fracture behavior. The boron-free alloy fractures intergranularly whereas the boron-doped alloy fractures by transgranular quasi-cleavage with more localized ductile tearing than was evident for tensile fracture [36]. Impact testing of the P/M FeAl alloys revealed even higher toughness values of 85-107 J (107 to 132 J/cm²) for the ultrafine grained materials extruded at

950 and 1000°C (Fig. 18). The rugged, more ductile appearance of the two fractured specimens of P/M FeAl processed at lower temperatures clearly shows the tortuous crack-deflection (shear along the prior particle oxide "envelopes") that makes them so tough (Fig. 19). It amplifies the importance of controlling the processing-induced microstructure of FeAl. The P/M FeAl extruded at 1100°C has an impact toughness of 25 J, similar to that of the hot-extruded I/M FeAl (FA-385) alloy, and still a good level of toughness for these FeAl materials.

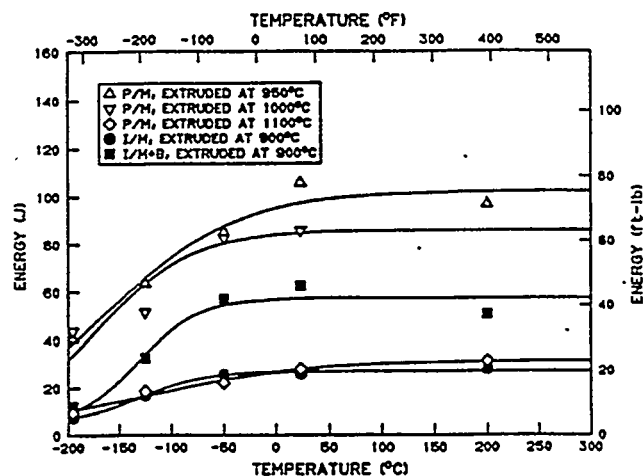


Fig. 18. Absorbed energy versus test temperature for Charpy impact tests of hot-extruded I/M and P/M FeAl alloys.

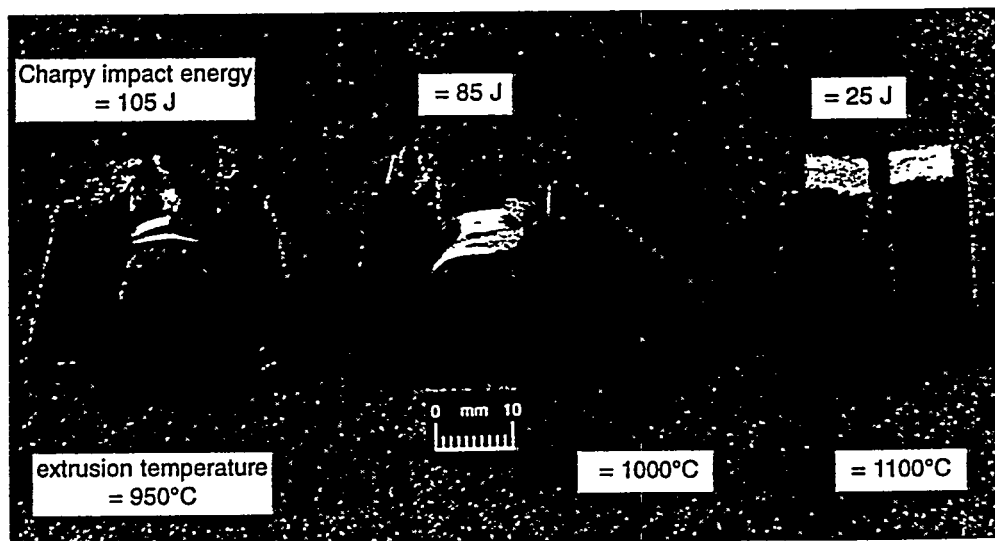


Fig. 19. Fractured Charpy impact specimens of P/M FeAl (FA-385), hot-extruded at the temperatures indicated and tested at room-temperature. Note the jagged, more ductile appearance of the two specimens with lower extrusion temperatures.

Tensile Properties at High-Temperatures

While room-temperature mechanical behavior is important in enabling FeAl alloy components to survive fabrication, handling and assembly prior to service, it does not qualify them for service as corrosion- and heat-resistant materials. High-temperature tensile and creep strength determines at what temperatures FeAl alloys can be used, and how long they might last in-service. Previous work had shown that wrought (hot-rolled sheet) FeAl alloys had YS of about 350 MPa at 600°C, became much weaker at higher temperatures, and were often weaker than comparable Fe₃Al or lower-aluminum iron-aluminides [11,39]. The strongest of these first-generation developmental FeAl alloys was the FA-362 alloy, as shown in the comparison of YS at 600°C for various FeAl alloys in Fig. 20. Clearly, it was stronger than the corresponding binary Fe-36Al alloy (FA-324) and the more weldable FA-385 alloy, which contained carbon instead of boron. However, boron microalloying additions to the FA-385 alloy, or chromium additions, improved the YS to nearly 500 MPa, clearly stronger than the FA-362 alloy and stronger than the other modified FA-385 alloys (Fig. 20) [12]. The boron doped FA-385 alloys were chosen for scale-up and for more detailed processing/properties studies.

CAST MATERIAL - The tensile properties of the FA-385 base alloy and the boron-doped alloys are plotted as functions of temperature in Fig. 21 (no stress-relief or additional heat treatments). The properties of type 310 austenitic stainless steel and cast HU alloy (Fe-39Ni-18Cr

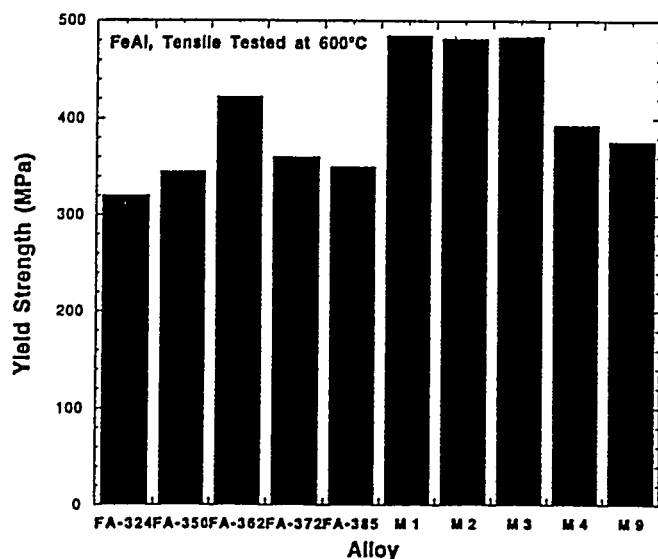


Fig. 20. Yield strength of various FeAl developmental alloys (compositions in Table I), for specimens punched from sheet hot-rolled at 850 to 900°C, heat-treated for 1 h at 700 to 800°C, and tensile tested at 600°C.

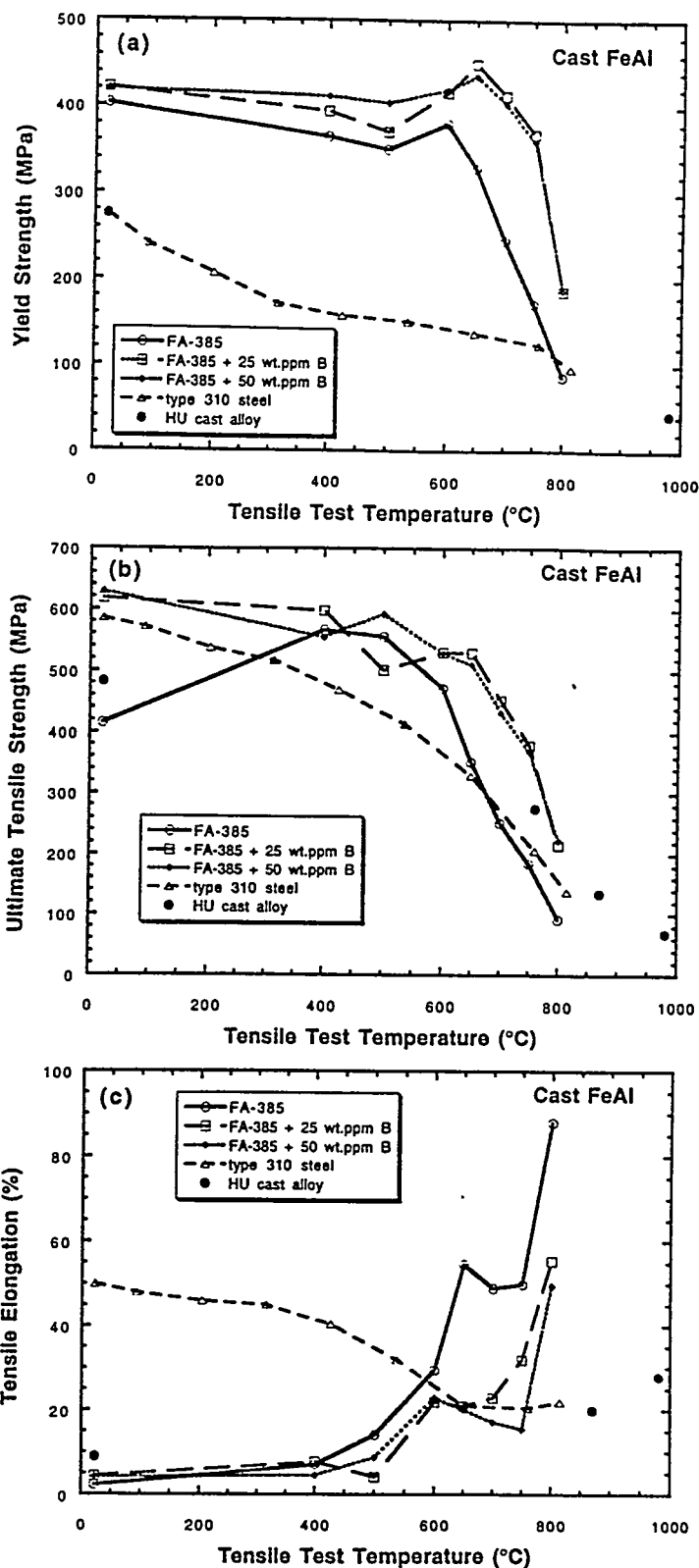


Fig. 21. Plots of (a) yield-strength (YS), (b) ultimate tensile strength (UTS), and (c) total elongation (TE), for as-cast FeAl (FA-385) alloys with and without boron doping. Specimens were stress-relieved 1 h at 750°C after machining and tensile tested in air. Data for type 310 stainless steel and HU alloy are included for comparison [40].

alloy with superior resistance to high-temperature oxidation and sulfidation [40]) are included for comparison. All three alloys maintain nearly constant YS from room-temperature to about 600°C, with some evidence of the YS anomaly characteristic of FeAl intermetallic alloys at 600 to 650°C (Fig. 21a) [3,41,42]. Yield strength of the FA-385 alloy drops off rapidly above 650°C, while the boron-doped alloys still have a YS near 400 MPa at about 750°C, and then weaken rapidly at higher temperatures. At temperatures of 750°C and below, the boron-doped alloys are more than twice as strong as type 310 stainless steel. Heat-treatments for 1 h at 900°C or at 1200°C did not appreciably affect the YS of these Fe-36Al (FA-385) alloys.

The UTS of the boron-doped FeAl alloys remains at 500 to 600 MPa up to about 700°C and then declines with increasing temperature (Fig. 21b). These boron-doped alloys have higher UTS than type 310 stainless steel or HU alloy up to about 800°C. Total elongation of all these cast FeAl alloys is 5 to 10% from room-temperature to about 400°C, and then increases to > 20% at 600°C and above (Fig. 21c). There is a drop in the ductility with increasing temperatures at 600 to 650°C that nearly coincides with the YS anomaly (Figs. 21a and 21c). These alloys then become very ductile at 800°C when their fracture mode makes a transition from transgranular cleavage with little necking to ductile-dimple fracture with a high degree of necking (Fig. 22). At 700 to 750°C and above, these FeAl alloys are much more ductile than type 310 steel or HU alloy.

The tensile properties of some of the cast Fe-30Al alloys were also measured. Generally, they were considerably weaker than the Fe-36Al alloys at 650°C and above, with 30 to 40% ductility at 650 to 800°C.

HOT-EXTRUDED I/M AND P/M MATERIAL - The tensile properties of P/M FeAl extruded at 1100°C (and stress-relieved for 1 h at 750°C) were tested over the entire temperature range of room-temperature to 1000°C, and data are plotted in Fig. 23. Other I/M or P/M alloys were tested only at 600°C or at 600 to 800°C.

All of the hot-extruded alloys were stronger than cast alloys at 100 to 600°C with YS of 400 to 550 MPa. The P/M FeAl alloys extruded at various temperatures had similar YS values, and were slightly stronger than the I/M alloys (Fig. 23a). UTS values for all the I/M and P/M alloys declined almost continuously from the high values of 800 to 1200 MPa found at room-temperature to about 200 MPa at 800°C (Fig. 23b). The ductility behavior with temperature was more complicated for these fine and ultrafine grained hot-extruded FeAl alloys, which also had more complex microstructures (particularly the P/M FeAl alloys) than the cast FeAl alloys (Fig. 23c). While ductility is higher in all these alloys (> 10%) compared to cast material up to about 500°C, it then shows a maximum at about 600°C followed by a new minimum at

700 to 800°C. Ductility for the P/M FeAl alloys varies from 10 to 30%, and is slightly higher for the I/M FeAl alloys at 600°C.

Creep-Rupture Properties at High-Temperatures

Previous studies of high-temperature creep of Fe₃Al alloys have established several facts that are also applicable to

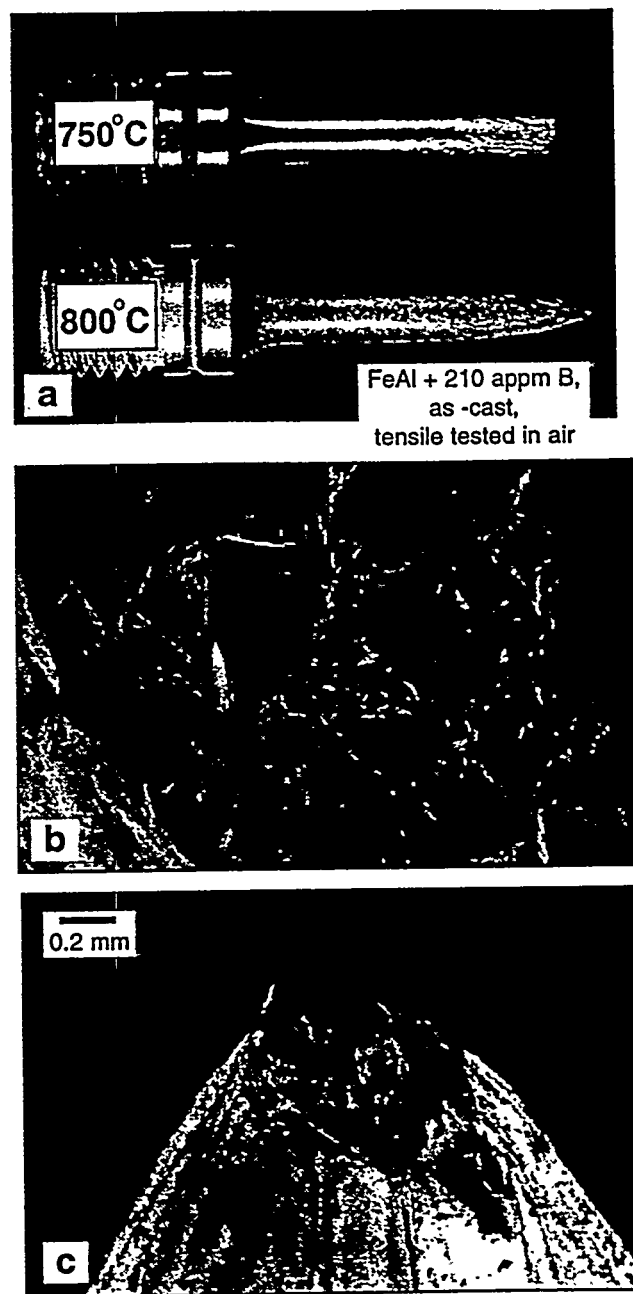


Fig. 22. Comparison of fractured cast FeAl specimens tensile tested at 750 and 800°C to show the transition to a ductile fracture mode. (a) optical photo, and (b) and (c) SEM fractography.

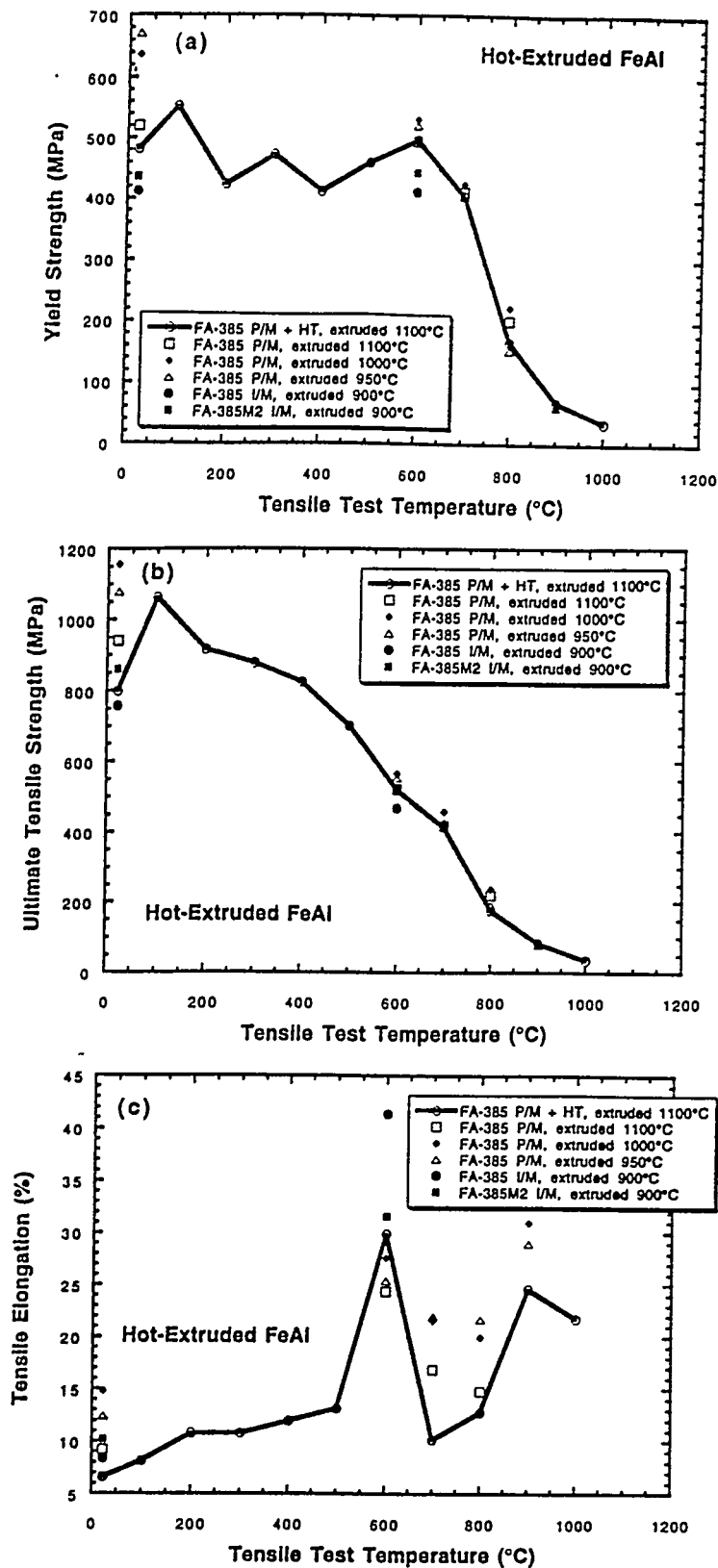


Fig. 23. Plots of (a) yield-strength (YS), (b) ultimate tensile strength (UTS), and (c) total elongation (TE), for hot-extruded I/M FeAl (FA-385) alloys with (FA-385M2, 210 appm) and without boron doping, and P/M FeAl (FA-385) hot-extruded at 950, 1000, and 1100°C. Specimens were stress-relieved 1 h at 750°C after machining and tensile tested in air.

FeAl alloys [25,32,38,43-45]. These include: (a) much more creep than one would expect for an ordered intermetallic alloy or an alloy with comparable tensile strength, (b) creep-rupture behavior that is very sensitive to prior processing/heat-treatment, and (c) very effective precipitation-strengthening from fine MC carbides for creep-resistance.

The boron microalloyed Fe-36Al (FA-385) alloys were found to have by far the best creep-rupture resistance at 600°C/207 MPa, by comparing hot-rolled sheet specimens of the M1 through M11 modified FA-385 series of alloys (Fig. 24). The same is also true for cast

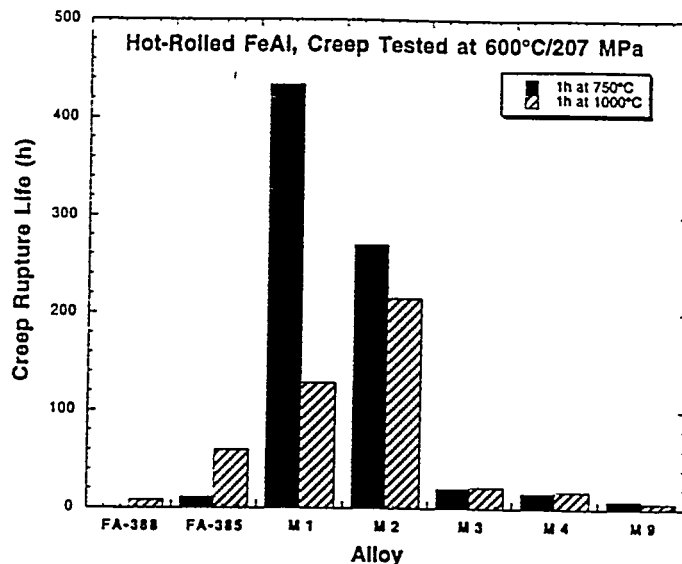


Fig. 24. Creep rupture lifetime of various FeAl developmental alloys (compositions in Table I), for specimens punched from sheet hot-rolled at 850 to 900°C, heat-treated for 1 h at 750°C or 1 h at 1000°C, and creep-tested at 600°C and 207 MPa.

FeAl (FA-385) alloys similarly creep tested (Fig. 25). The as-cast boron-doped alloys have rupture lives of about 500 to 700 h compared to only about 12 h for the boron-free alloy, and their creep resistance is consistent with the more abundant dispersions of fine ZrC they contain (Fig. 11). These Fe-36Al alloys are also clearly more creep-resistant than the Fe-30Al alloys.

The fine-grained hot-extruded I/M FeAl alloys (FA-385, -385M1 and -385M2) have much less creep-resistance, but creep-resistance increases after heat-treatment of 1 h at 1200°C (Fig. 26). Heat-treatments of 1 h at 1200 to 1250°C produce creep behavior similar to that found in as-cast material for the boron-doped FA-385 alloys (Fig. 26). Significant heat-treatment/processing effects on the creep behavior of FeAl alloys were first found in an Fe-36Al alloy (FA-328M11, Table I). It was found that heat-treatments of 1200 to 1250°C produced

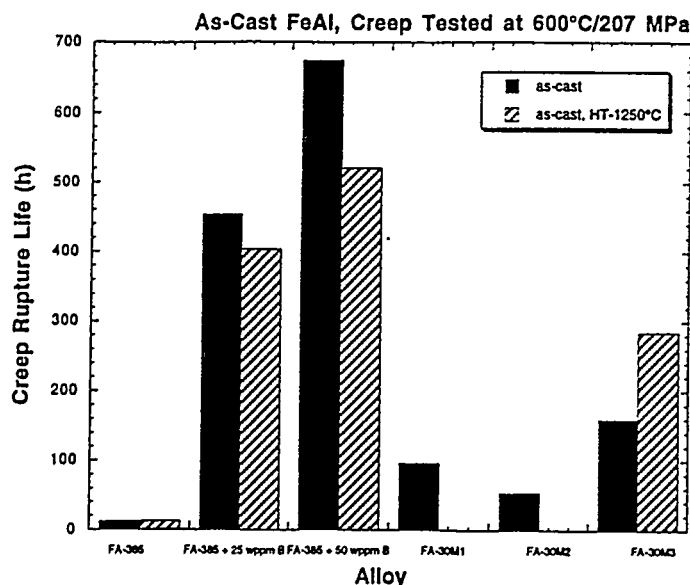


Fig. 25. Creep rupture lifetime of Fe-36Al (FA-385) and Fe-30Al (FA-30) FeAl developmental alloys (compositions in Table I), for specimens machined from as-cast material, and then either stress-relieved for 1 h at 750°C or heat-treated for 1 h at 1250°C, and creep-tested at 600°C and 207 MPa.

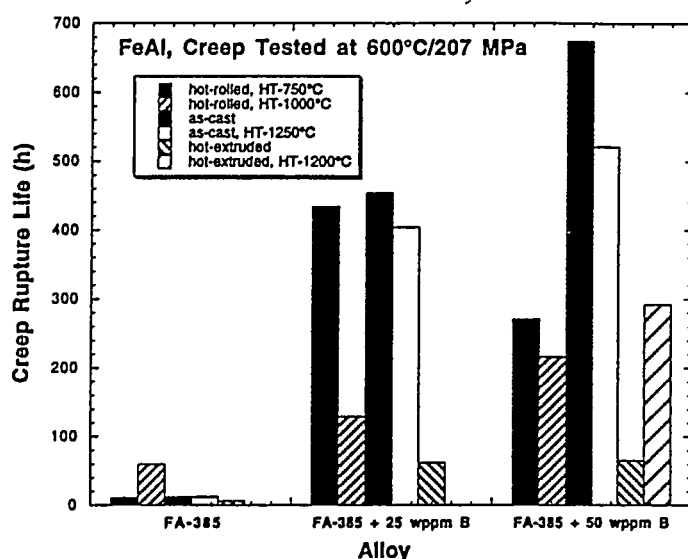


Fig. 26. Creep rupture lifetime of Fe-36Al (FA-385) FeAl alloys with and without boron, for specimens punched from sheet hot-rolled at 900°C, rod hot-extruded at 900°C, or from as-cast material, and then given various heat-treatments and creep-tested at 600°C and 207 MPa.

fine precipitates (FeTiP and Ti-rich MC in this case) which made this alloy much more creep-resistant with than without such precipitates (Fig. 27). The creep behavior of the as-cast boron-doped FeAl (FA-385M2) is

comparable to that of type 304 austenitic stainless steel, and is significantly better than type 403 ferritic stainless steel (Fig. 28). By comparison, even ultrafine-grained P/M FeAl with ZrC precipitates falls between type 403 and type 304 steels.

Welding and Weld-Overlay Technology

One unique aspect of this particular FeAl alloy development effort has been the inclusion of studies of welding and weld-overlays. Although there are some cast applications that may not require welding, welding is usually necessary for many structural applications. The ability to join dissimilar metals or to make weld-overlays of a corrosion-resistant material as a cladding to protect an otherwise adequate conventional structural material greatly enhances new application possibilities. Although welding issues for iron- and nickel-aluminides are covered in more detail elsewhere in this proceedings [46], it is important to indicate the current status of these efforts and how they relate to and benefit from the properties studied to develop monolithic FeAl alloys.

When the FA-385 alloy was first found to be more weldable than other FeAl alloys, attempts were immediately made to produce FeAl weld-overlay cladding on conventional type 304L and 2.25Cr-1Mo steel substrates (Fig. 2) [23]. While multipass FeAl weld deposits were made without any hot-cracking problems, cold-cracking did occur during cooling [23]. Solutions to the cold-cracking problem were found by preheating (350 to 400°C) and by post-weld heat-treating (750 to 800°C). A large number of FeAl weld-consumables with various alloy compositions have been examined (including up to 7 wt % Cr [12]), and the best results are currently being found with chromium-free Stoodly coiled FeAl wires (Al-core, Fe-sheath, 1.6 mm in diam.) developed for automated gas-metal-arc (GMA) welding (Fig. 29). Good, crack-free FeAl weld-overlays have been produced using these wires. The FeAl weld deposit in these cases have alloy compositions similar to that of the monolithic FA-385 alloy. Such weld-overlay deposits have been made on 9Cr-1MoVNb, type 410 and 310 stainless steels, and plain-carbon steels. More fundamental neutron-scattering and finite-element modeling studies of such FeAl weld-overlay deposits on 2.25Cr-1Mo steel show that welding produces high residual tensile stress in the FeAl clad, and that the post-weld heat-treatments help to relax such stresses [47,48]. It is also clear that an FeAl alloy composition that is more ductile and stronger in air at room-temperature should be more resistant to cold-cracking. Work is in progress to better control the composition of the FeAl weld-deposit, and to find ways to reduce the preheat and postweld heat-treatment requirements.

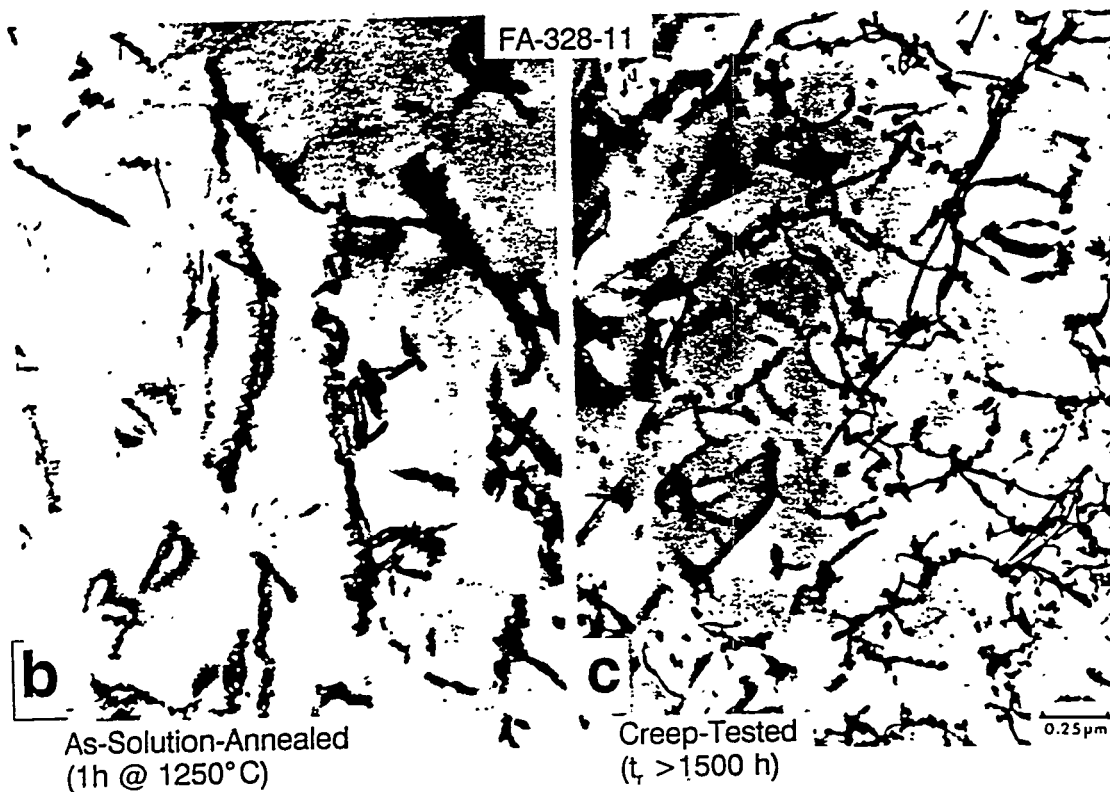
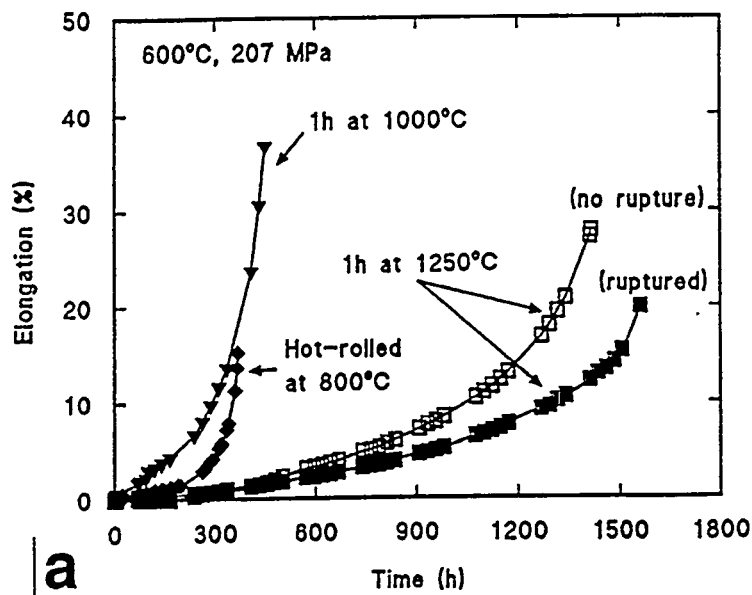


Fig. 27. Effects of processing/heat-treatment on the creep-rupture lifetime (a) for a complex FeAl alloy (Fe-36Al-5Cr, FA-32811, see Table 1) specimens punched from sheet hot-rolled at 800°C and then heat-treated for 1 h at 1000°C or 1 h at 1250°C and creep-tested at 600°C and 207 Mpa. (b) and (c) TEM showing that fine Ti-rich MC carbides form after 1 h at 1250°C, and evolve into a mixture of MC and FeTiP particles during creep (unpublished data of Maziasz and McKamey, ORNL).

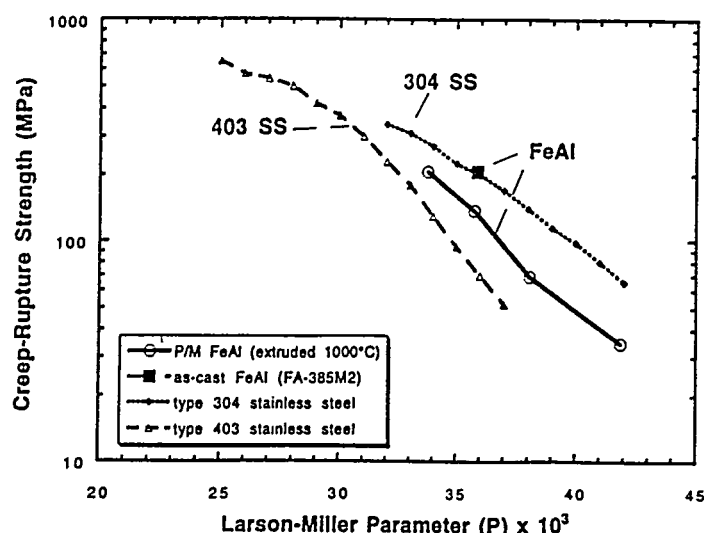


Fig. 28. Larson-Miller plot of creep rupture life versus creep stress for as-cast or P/M FeAl, with data for types 403 and 304 stainless steels included for comparison.

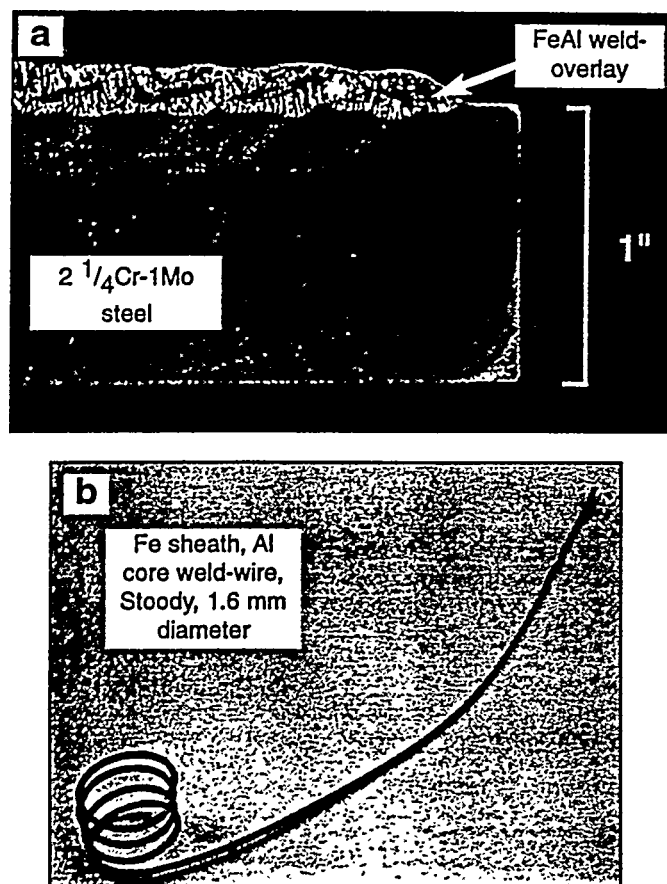


Fig. 29. (a) FeAl weld overlay (single-layer) made on 2.25Cr-1Mo steel substrate using (b) Fe-Al weld wire produced by Stoody for automated GTA welding.

Autogenous welding is a demanding requirement for most engineering materials, including type 316 or 310 austenitic stainless steels and other Fe-Cr-Ni alloys. Autogenous welding is very difficult for most intermetallics because they usually have low ductility and/or environmental embrittlement at room-temperature. Preliminary welding studies of as-cast FeAl with only about 2% ductility indicated that it does not hot-crack, but does cold crack after welding with no preheat or post-weld heat-treatment. To test the hypothesis that better ductility and strength of the base FeAl material in ambient air would prevent such cold-cracking, similar welds were made on pieces of P/M FeAl extruded at 1000°C and on I/M FeAl (FA-385M2) extruded at 900°C, which both had much better ductility. Consistently, neither showed any cold-cracking after autogenous GTA welding without pre-heat or post-weld heat-treatment (Fig. 30). The refined microstructures of both of these FeAl substrates also appear to have refined the grain size of the weld metal. While still preliminary, these experimental data represent a major break-through in the welding of iron-aluminide intermetallic alloys that is encouraging for efforts to commercialize these materials.

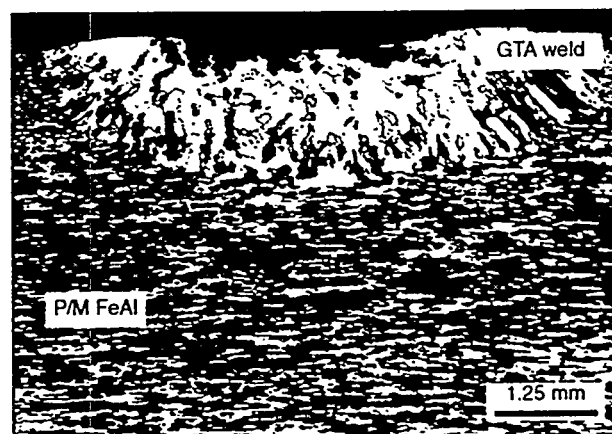


Fig. 30. Crack-free GTA autogenous weld made on P/M FeAl (FA-385) hot-extruded at 1000°C.

Industrial Testing and Potential Applications

Currently, there has been some industry testing of FeAl alloy resistance to various oxidizing molten salts at 650°C and at 900°C [5,7,18,19]. Testing is also underway in neutral molten salts used for heat-treating die-steel blocks. INCO has tested the carburization resistance of FeAl alloys at 1000°C and 1100°C in gaseous environments that simulate steam/methane reformer conditions (found in hydrogen production) or ethylene pyrolysis. A large petrochemical company has expressed



Fig. 31. Centrifugally-cast FeAl (FA-385M2) radiant heating tubes (2.44 m long, 95.25 mm O.D. with wall thicknesses ranging from 6.35 to 19 mm) produced by Alloy Engineering & Casting Company using the Exo-Melt™ process [12].

interest in these data, and has indicated that superior oxidation, sulfidation and corrosion-resistance in steam, air, or combustion gases, particularly sulfidizing environments, would enable FeAl to be compared against 300 series stainless steels (i.e. type 304). A large construction engineering company is also testing FeAl for its sulfidation resistance at 800°C and above. Additional, broader corrosion testing of FeAl by INCO is in progress.

FeAl alloys can be cast in large heats using the recently developed Exo-Melt™ process [2,16]. FeAl radiant heating tubes were centrifugally-cast and U-bends were sand-cast by Alloy Engineering & Casting Company from a 1,320 kg heat of FA-385M2 (Fig. 31). FeAl castings are being made for several large scale, low-stress applications that involve resistance to metal wastage in oxidizing/sulfidizing materials processing applications.

Because it has lower density and much higher electrical resistance than conventional steels and alloys [12], FeAl can also be considered for heating elements in rod, wire or sheet/strip form. The good mechanical properties of P/M FeAl also raise the possibility of making P/M parts for automotive or other applications that require strength/corrosion resistance comparable to that of 300 or 400 series stainless steels or better. FeAl also has good potential as sintered porous gas-metal filters made from powders for coal-gasification and hot-gas clean-up with high sulfur contents.

FeAl, particularly when preoxidized, does show some evidence of resistance to dissolution in molten aluminum [12]. Industrial testing of FeAl in several different molten metal environments (both static and wear/cavitation) is in progress. FeAl alloys also have the potential to complement Ni₃Al at somewhat lower temperatures as processing equipment or furnace furniture. FeAl should be compared with 300 and 400 series stainless steels, HU, or other Fe-Cr-Ni alloys in oxidizing, carburizing or sulfidizing environments in which alumina-formers

perform better than chromia formers in heat/corrosion resistant applications. In some applications where Cr and/or Ni are considered toxic or environmental problems, FeAl alloys have the added advantage of being Cr- and Ni-free.

Summary

FeAl alloys have been developed with 36 to 38 at. % Al and minor additions of Mo, Zr, C, and B that produce the best combination of room-temperature ductility, high-temperature strength and weldability (hot-cracking resistance) for a given processing condition. Processing effects are most pronounced at room-temperature. Refined grain size and/or P/M processing via hot-extrusion can greatly improve ductility, strength and impact-toughness in air. Such alloys also have good resistance to moisture-induced environmental embrittlement and cold-cracking after welding. At room-temperature, boron additions also help improve environmental embrittlement and produce a transgranular cleavage or quasi-cleavage fracture mode. Boron-doped FeAl alloys with refined grain structures are tougher than boron-free alloys.

At higher temperatures, boron microalloying additions are the most effective in increasing YS and creep-rupture resistance. This is caused by boron enhancing the formation of fine ZrC precipitation. Cast material with the combination of coarser grain size and fine ZrC precipitates has the best high-temperature strength.

Finally, cast FeAl alloys with sufficient boron, zirconium and carbon benefit in terms of room-temperature ductility from stress-relief heat-treatments at 750, 900 or 1200°C. Fine-grained FeAl alloys benefit most from a 750°C heat-treatment, but are also ductile at room-temperature without any heat-treatment. For high-temperature properties, solution-annealing heat-treatments at 1200 to 1250°C produced good strength and creep-resistance in cast FeAl. Wrought, fine-grained FeAl alloys tend to be weaker at high-temperatures, but solution annealing at 1200 to 1250°C also improves their high-temperature strength significantly. Heat-treatments at 1000 to 1250°C also preoxidize FeAl.

Acknowledgments

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