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EFFECTS OF INTERSTITIAL BORON AND ALLOY STOICHIOMETRY ON ENVIRONMENTAL EFFECTS IN FeAl

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ABSTRACT

Room-temperature tensile tests were conducted on B-doped (300 wppm) and B-free polycrystalline FeAl alloys containing 37, 40, 45 and 48 at. % aluminum in pure hydrogen gas at pressures in the range of 10^{-8} to 10^{3} Pa. The ductilities of both B-free and B-doped FeAl decreased with increasing Al content. However, at a given Al level, the ductility of B-doped FeAl was higher than that of its B-free counterpart. Fracture mode was *independent* of environment and dependent mainly on stoichiometry. Ductility was found to be very sensitive to environment, particularly in the lower Al alloys. Alloys that exhibited >10 % ductility in UHV showed a decrease in elongation to fracture with increasing hydrogen pressure. Tests conducted in dry hydrogen gas result in greater ductilities than those conducted in, indicating that water vapor is more detrimental than H_2 to the ductility of FeAl alloys.

DISCLAIMER

INTRODUCTION

Iron aluminides (FeAl) have long been known to be brittle in air at ambient temperature. In alloys with an aluminum concentration greater than about 40 at.%, brittle failure occurred via weak grain boundaries [1-3]. Those with less than ~ 40 at.% Al failed in a transgranular fashion [1-3]. Both types of failure have generally been considered intrinsic properties. However, more recent data suggests that this is not the case. Moisture-induced environmental embrittlement [4-7], an *extrinsic* factor, has been shown to be the major cause of room-temperature brittleness, at least on the transition metal rich side of stoichiometry (< 40 at. % Al). In the absence of moisture (i.e., when testing is performed under vacuum or cover gases such as O_2), FeAl can be ductile with elongations to fracture as high as ~18 % [7]. Atomic hydrogen, generated from the reaction of water vapor in air with the Al atoms in FeAl, is generally considered the culprit behind such embrittlement, as first suggested by Liu *et al* [4]. Recent chemisorption and thermal desorption studies on the surfaces of single crystal [8] and polycrystalline [9] Al based intermetallics confirm that aluminum is the active species responsible for the dissociation of water into atomic hydrogen.

The exact mechanism by which atomic hydrogen causes premature failure remains under debate and is generally considered to be a complex problem [e.g., 10]. However, since there is no evidence of brittle hydride formation in FeAl, two other mechanisms are suggested as possible explanations. According to the first, there exists a localized region of hydrogen-enhanced plasticity in front of the crack tip [11-14]. The second suggests a decohesion or lowering of the bond strength by the presence of atomic hydrogen [e.g. 4]. Consistent with the decohesion mechanism, first principles calculations for FeAl show that absorbed atomic hydrogen can reduce cleavage energy by as much as 70 %, depending on the hydrogen concentrations [15].

Small alloying additions of boron, on the order of a few hundred wppm, are beneficial in increasing room temperature ductility of some ordered intermetallics [e.g., 16-20]. In the case of Ni_3Al, the main role of boron in increasing room-temperature ductility has been established - after much debate - to be related to suppression of H_2O-induced embrittlement [21,22]. In FeAl, boron additions have little ability to suppress H_2O-induced embrittlement [7] and its benefits appear to be limited to just enhancing grain boundary cohesion [2,3,7]. This enhancement is particularly evident at lower aluminum levels (i.e., < 45 at.%). Naturally, the question that arises is what effects other hydrogen containing environments, namely H_2, have on ductility and what role boron might play in such environments (particularly because of the recent discovery [23] that B additions to another intermetallic (Ni_3Al) *promote* embrittlement in low-pressure hydrogen gas i.e., < 10^4 Pa). Furthermore, it is of interest to determine the "true" intrinsic ductility of FeAl alloys as a function of stoichiometry, which can only be measured when all extrinsic factors have been scrupulously eliminated (i.e., when tests are performed in UHV).

In the present study, a comprehensive investigation is made of alloy stoichiometry and H_2 pressure on the room temperature ductility of FeAl. In an ongoing effort to better understand environmental embrittlement, iron aluminides with and without boron additions and ranging in composition from 37 to 48 at.% Al are tested in ultra high vacuum (UHV) and in H_2 with pressures ranging from ~10^{-8} to 10^3 Pa.

EXPERIMENTAL

Alloy Compositions and Processing:

Four off-stoichiometric FeAl alloys, containing 37, 40, 45 and 48 at.% Al, were arc melted, mixed and drop cast into copper chill molds. Boron doped alloys, produced in the same fashion and containing 300 wppm boron (0.12 at.%), were also made with 40, 45 and 48 at.% Al. The cast ingots, having dimensions of ~127 mm (height) and ~25 mm (diameter), were canned in mild steel billets and hot extruded at a 9 : 1 ratio. Extrusion temperatures ranged from 1073 to 1223 K. A detailed summary of the extrusion temperatures and annealing temperatures and times are presented elsewhere [24].

Tensile Tests

Two different gage sections were utilized for the tests. Preliminary specimens with gage sections $3.2 \times 1.6 \times 0.5$ (mm) were electro-discharge machined (EDM) from the extruded ingots of Fe-40Al + 300 wppm boron and Fe-45Al with their long axes parallel to the extrusion direction. Each specimen had ~0.08 mm removed from all EDM surfaces by grinding. These specimens were then heat treated in vacuum (~7 x 10^{-4} Pa) for 1 h at 1158 K for the B-doped Fe-40Al and 1203 K for the Fe-45Al alloy, followed by 5 days at 673 K for both alloys. The first temperature established the grain size while the long term anneal was used to reduce the vacancy concentration to near its equilibrium value. After annealing, all the specimens were hand polished, finishing with 4000 grit, to reduce surface defects. Subsequent specimens, for all compositions, were EDM with a gage section having dimensions of $7.4 \times 1.7 \times 0.5$ (mm). Each of these specimens were hand polished to remove ~0.07 mm from all EDM surfaces, finishing with 4000 grit, again to diminish surface defects. Since small changes in grain size (based on our preliminary data) did not appear to change ductility, all the latter specimens were given a standard anneal of 1073 K for 1 h followed by 673 K for 5 d for convenience.

Tensile testing was performed in an UHV chamber equipped with two liquid nitrogen cooled sorption pumps and an ion pump. A hydrogen bottle (99.9999%, research purity) was connected by copper tubing to a Varian leak valve attached to the UHV chamber. Details of chamber preparation are described elsewhere [22].

The dimensions of all the specimens were measured using a microscope equipped with a translation stage. Scribe marks were placed at the edges of the radii on all the specimens tested. Ductility was taken as the change in length between the scribe marks divided by the initial gage length. Due to the two different gage lengths used, the strain rate varies between $\sim 5 \times 10^{-4}$ - 1.2×10^{-3} s^{-1}. For iron aluminides, this small deviation in strain rate has been shown not to affect elongation to fracture [25,26].

Fracture Surface Analysis

Fracture surfaces of tested specimens were examined in a scanning electron microscope (SEM) to determine fracture mode as a function of stoichiometry and environment.

RESULTS AND DISCUSSION

Figure 1 shows the room-temperature ductility, obtained in UHV, of the B-free and B-doped FeAl alloys tested as a function of aluminum concentration. In the B-free alloys, ductility drops off precipitously with increasing aluminum concentration until there is no observable plasticity when the aluminum concentration reaches 48 at. %. Similar to the B-free alloys, elongation to fracture of the B-doped FeAl alloys also decreases with increasing Al content. However, the ductile-brittle transition is shifted to higher Al concentration. If one considers UHV tests to be a measure of true "intrinsic" properties, then it is clear that as the composition of the B-free FeAl approaches the stoichiometric composition the intrinsic ductility approaches zero. Furthermore, even though B increases ductility over the entire range of stoichiometry, it is unable to totally eliminate brittle failure at 48 at.% Al where ductility is less than 6%. In agreement with earlier studies [e.g., 6], the most beneficial effect of boron on ductility, even when extrinsic factors have been scrupulously eliminated, occurs when the Al level is less than ~45 at.%.

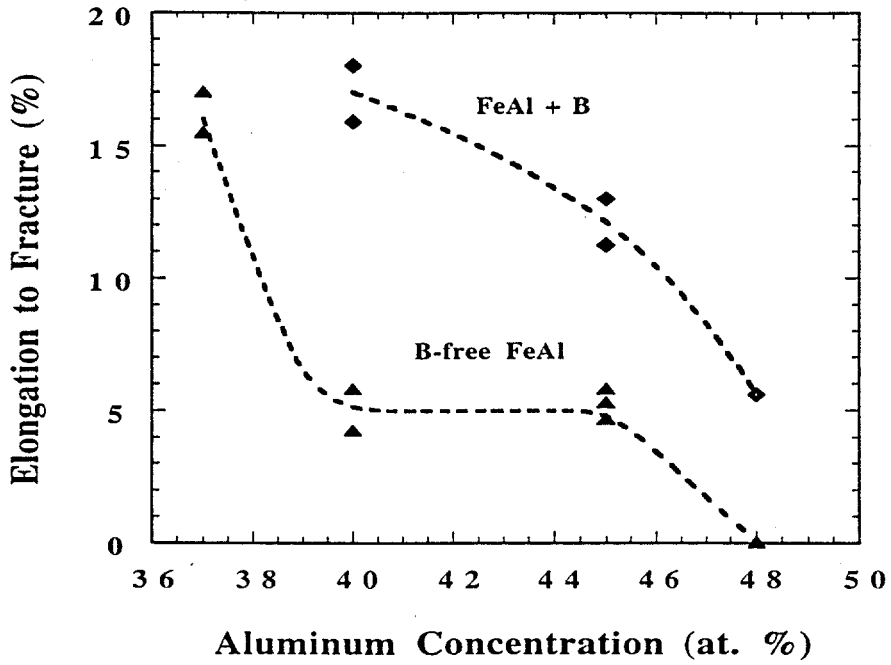


Fig. 1. Elongation to fracture of B-doped and B-free FeAl in UHV as a function of Al content.

Figure 2 shows the elongation to fracture at room temperature of B-free FeAl alloys as a function of hydrogen pressure. The most comprehensive data were obtained for the 37 at.% alloy and shows that as the hydrogen pressure increased the ductility decreased from ~17% at UHV to ~9 % at 1300 Pa. The 40 and 45 at. % Al alloys were similar in that their ductilities were ~ 5.5% at UHV and remain relatively constant as the hydrogen pressure increased. The Fe-48Al showed no plasticity, even in UHV. In the case of B-free Fe-37Al, elongation to fracture decreases by a factor of approximately two over the hydrogen pressures tested (Fig. 2). Since the ion gage (a known source of atomic hydrogen [22]) has no measurable effect on the ductility of B-free FeAl, this implies that dissociation of molecular hydrogen into atomic hydrogen is occurring on fresh

surfaces during deformation, at least to some degree. The driving force for hydrogen diffusion ahead of the crack tips is likely to increase with increasing H_2 pressure. Therefore, the amount of atomic hydrogen diffusing into the material also probably increases. This would increase the concentration of absorbed atomic hydrogen and, hence, lower the ductility as the hydrogen pressure increases. The undoped 40, 45 and 48 at. % Al alloys are intrinsically brittle (Fig. 1) and, therefore, little (if any) additional effect from the hydrogen environment is seen.

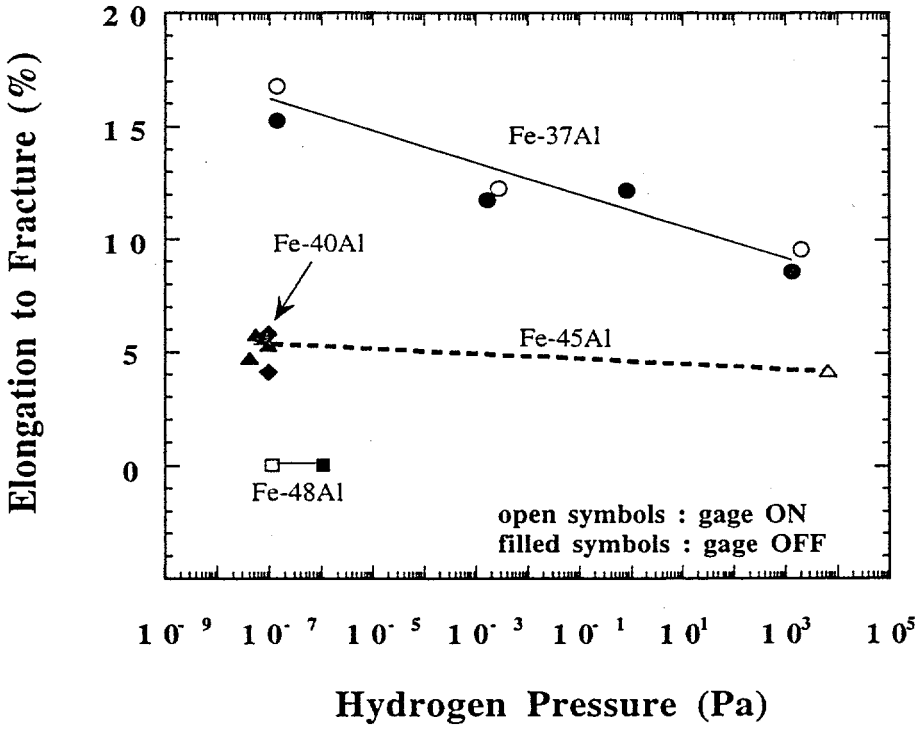


Fig. 2. Elongation to fracture of B-free FeAl as a function of H_2 pressure.

Figure 3 shows the ductility of B-doped FeAl as a function of hydrogen pressure. Like the B-free material, elongation to fracture decreases with increasing hydrogen pressure for compositions that exhibit sufficient intrinsic ductility (> 10%). As in the case of B-free FeAl, surface dissociation of molecular hydrogen into atomic hydrogen is probably also occurring on B-doped FeAl. Comparing the earlier reported ductilities [6,24,27] of B-free and B-doped alloys (in alloys that experience environmental embrittlement, i.e., < 45 at. % Al), it is observed that molecular hydrogen at equivalent pressures to that of water vapor in air - assuming 50 % relative humidity (i.e., ~1300 Pa) - is not as harmful as moisture in air.

Figures 4 & 5 represent typical micrographs of the B-free and B-doped FeAl alloys (respectively) which show that as the aluminum concentration increases the fracture mode changes from transgranular to intergranular. This fracture behavior was the same in all environments tested. Note that the addition of boron (again, regardless of environment) increases the Al level at which intergranular fracture becomes dominant. The interpretation of this, since B-doped FeAl alloys (particularly those with less than 45 at.% Al) still suffer from environmental embrittlement, is that the main role of boron is to increase grain boundary cohesion. This is in

contrast to the results of another intermetallic (Ni_3Al) [21,22], where the main role of boron is to suppress environmental embrittlement.

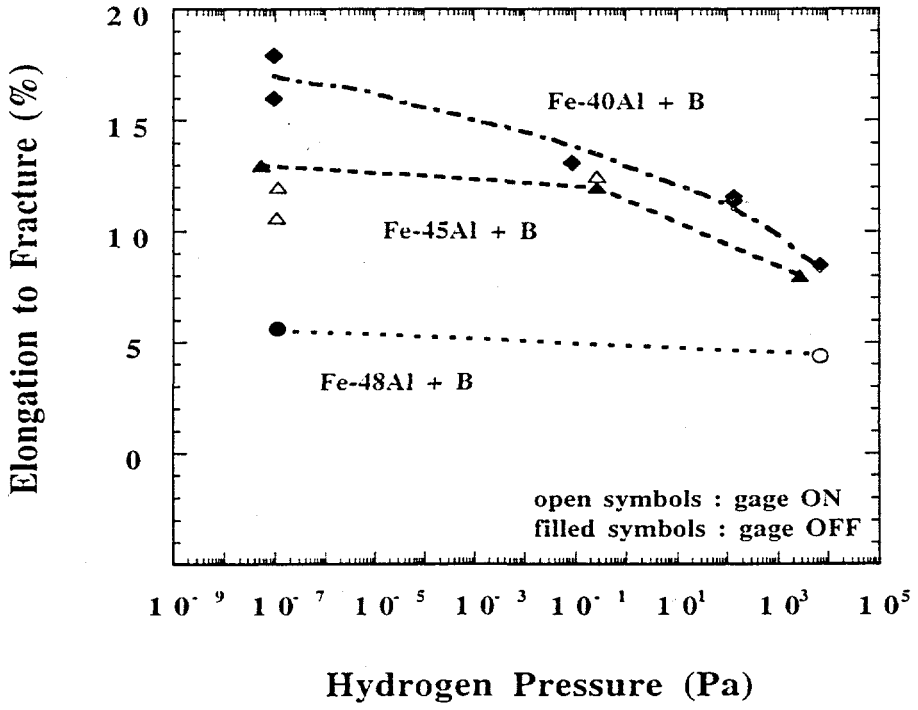


Fig. 3. Ductility of B-doped FeAl as a function of hydrogen pressure.

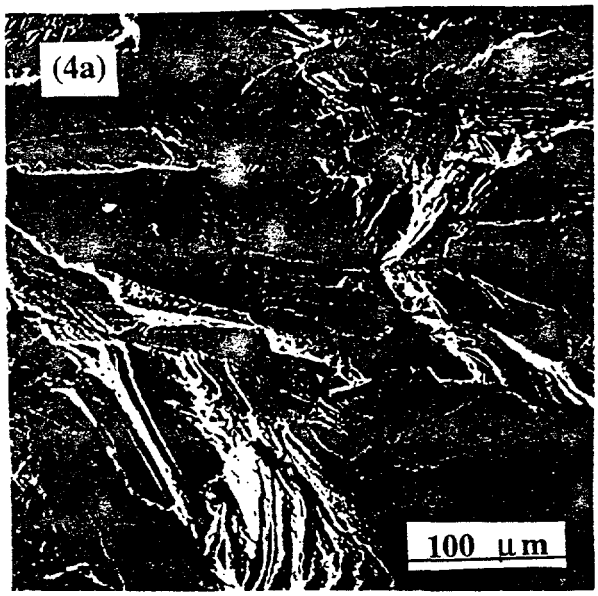
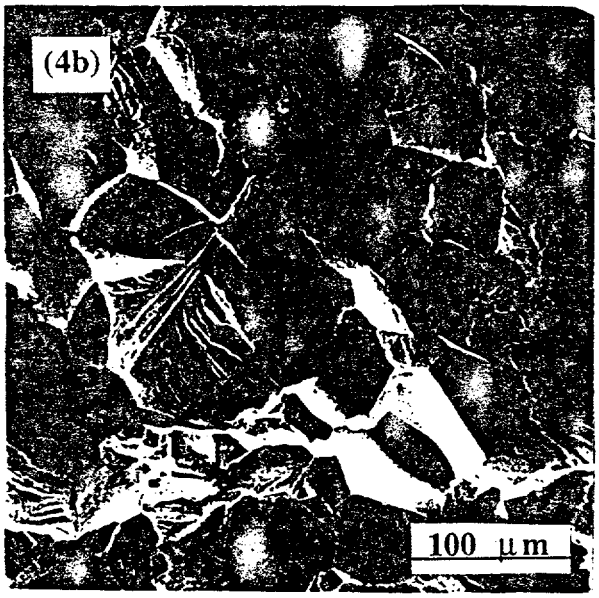
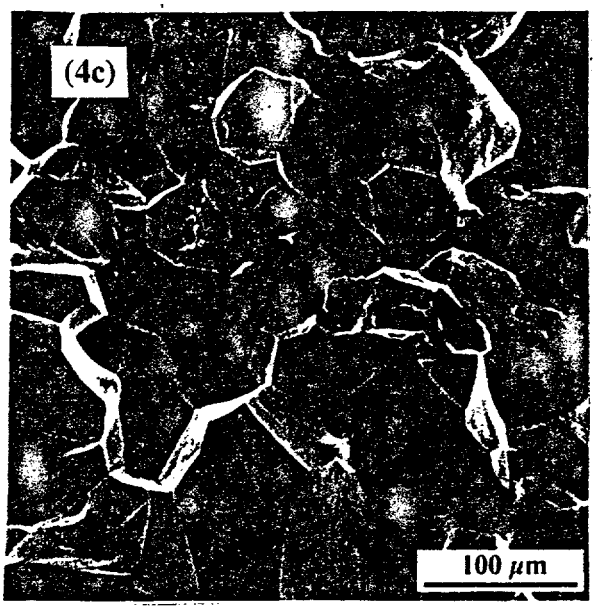


Fig. 4. Room-temperature fracture surfaces of B-free FeAl alloys with: (4 a) 37 at.% Al, (4 b) 45at.% Al, and (4 c) 48 at.% Al. Note the increase in intergranular fracture with increasing Al concentration.





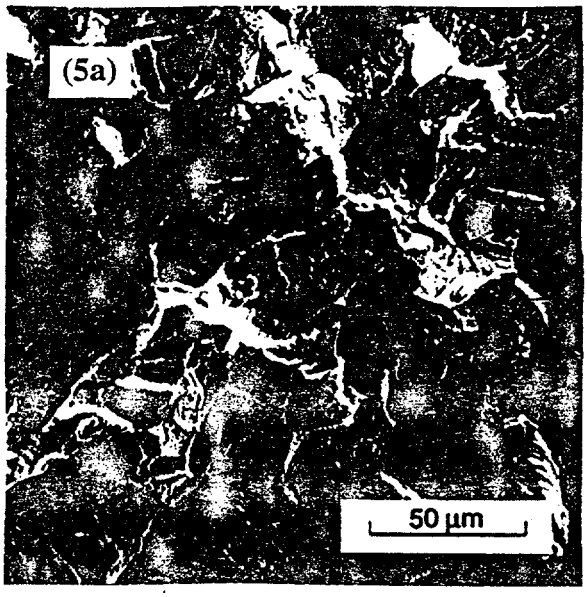
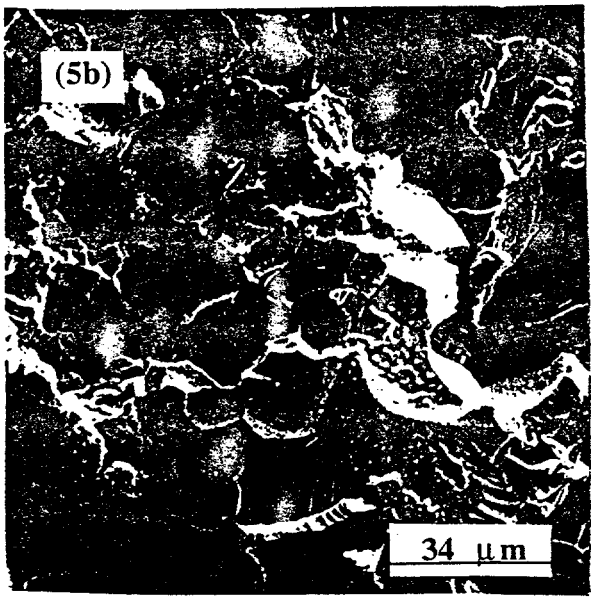
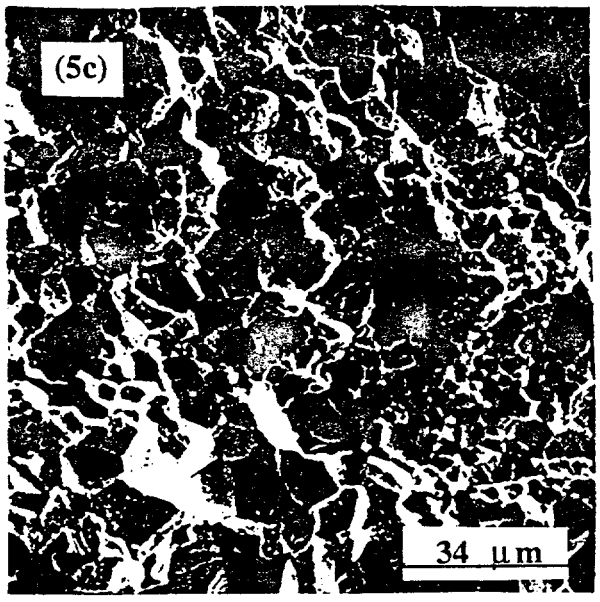


Figure 5. Room-temperature fracture surfaces of B-doped FeAl alloys with: (5 a) 40 at.% Al, (5 b) 45at.% Al, and (5 c) 48 at.% Al.





CONCLUSIONS

Based on tests conducted on B-free Fe-37, 40, 45, 48 Al and B-doped Fe-40, 45, 48 Al in UHV and hydrogen containing environments the following conclusions were made.

1) The intrinsic ductility (measured in UHV) of B-free and B-doped FeAl alloys decreases with increasing aluminum content.

2) The main role of boron is to enhance grain-boundary cohesion in FeAl. Consequently, there is a shift in the ductile to brittle transition to higher Al levels.

3) The effects of environment on the B-free FeAl alloys are most noticeable when Al content is less than ~40 at. %, but are still observable up to the 45 at. % level in the B-doped alloys.

4) Elongation to fracture of intrinsically ductile B-free and B-doped FeAl decreases with increasing hydrogen pressures. This has been interpreted to mean that molecular hydrogen, to some degree, dissociates on FeAl surfaces during deformation.

5) Ambient air (which contains moisture) is more detrimental to ductility than dry hydrogen gas for both B-free and B-doped FeAl alloys.

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