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SUMMARY

Small additions of boron have been shown to improve the room temperature ductility of the intermetallic compound Ni$_3$Al in recent studies. The boron is believed to segregate to the grain boundaries and strengthen them, allowing the inherent ductility of the grains to be achieved.

The present study was undertaken to see if boron has a similar effect on the low temperature tensile properties of the equiatomic intermetallic compound FeAl. A binary alloy without boron is compared with an alloy containing 0.78 at% B (0.2 wt %) B, by tensile testing over the temperature range of 300 K to 640 K. Both alloys were processed by powder metallurgy.

Results showed that 0.78 at% B addition to FeAl does indeed change the room temperature fracture mode from intergranular to transgranular, suggesting a strengthening of grain boundaries. The alloy containing boron is, however, still brittle at room temperature. A slight decrease in the ductile to brittle transition temperature is, nevertheless, observed. In addition a significant increase in strength of the alloy is observed with boron addition.

1. INTRODUCTION

Iron aluminide, FeAl, is an ordered intermetallic compound having the B2 (CsCl) crystal structure. This aluminide has recently been the object of considerable interest as a potential structural material for moderate to high temperature applications.

A major problem with this aluminide is that although single crystal FeAl displays considerable ductility at room temperature [1-4], polycrystalline materials are brittle [5]. One possible reason which has been suggested for this brittleness is the lack of sufficient operative slip systems at low temperatures resulting in a failure to satisfy Von Mises criterion. Some of the studies on single crystals and polycrystals [1,3,4,6], however, have determined that at room temperature, $\{110\}<111>$ slip occurs, which would result in sufficient independant slip systems (five) to satisfy the Von Mises criterion. Cross-slip would, however, be restricted if slip were confined to the $\{110\}$ planes. It has been suggested [7,8] that in ordered crystal structures, inability to cross slip may prevent spreading of slip across grain boundaries resulting in brittle fracture even if five independant slip systems are available.

Another possible reason for room temperature brittleness in spite of the
presence of five independent slip systems may be grain boundary decohesion before a sufficient number of slip systems can be activated. This may be particularly applicable to ordered alloys since certain grain boundary structures may be less stable than others as a result of wrong neighbor considerations [9].

Recently, considerable effort has been devoted to the study of polycrystalline Ni<sub>3</sub>Al [6], an ordered intermetallic compound with the L<sub>1</sub><sub>2</sub> crystal structure [10,11]. This is a FCC ordered crystal structure and has enough independent slip systems for polycrystalline deformation. However, polycrystalline alloys were found to be brittle and fractured in an intergranular manner. Additions of small amounts of boron, changed the fracture mode from intergranular to transgranular and dramatically made these alloys ductile at room temperature. The amount of ductility was found to be strongly related to the amount of boron. In addition, the mode of failure was found to change from intergranular to transgranular with the addition of boron. It has been suggested [12] that boron acts to modify the intrinsic nature of the atomic bonds at grain boundaries resulting in greater grain boundary strength and hence the inherent ductility of the individual grains of Ni<sub>3</sub>Al can be obtained without causing the grain boundaries to separate.

A possibility, therefore, exists that polycrystalline FeAl which appears to demonstrate {110} <111> slip with adequate number of independent slip systems, may also be failing in a brittle manner at room temperature due to weak grain boundaries. By analogy with Ni<sub>3</sub>Al, addition of boron may be expected to result in a change in the fracture mode as well as an improved ductility at room temperature. A test of this hypothesis forms the focus of this paper.

Another interesting observation in the previous studies [1,3,4,6] on FeAl is a change in slip system to {110} <100> at approximately 0.40 Tm. The exact temperature of the transition was found to be a function of the orientation of the single crystals and the alloy stoichiometry. Polycrystals of FeAl exhibit a ductile to brittle transition at about this same temperature. Although no clear correlation between the change in dominant slip system and the onset of ductility has been established, it is possible that the ductility may be a result of the observation that the critical resolved shear stress in the <100> direction decreases more rapidly than in the <111> direction.

The purpose of the current investigation, hence, was a preliminary study of the effect of boron on the low temperature mechanical behavior of polycrystalline FeAl. Samples containing 0.78 at% B have been studied at temperatures ranging from 300 to 680 K and compared with binary FeAl.

2. MATERIALS AND PROCEDURE

Materials for this study were made from prealloyed FeAl powder mixed with boron powder in a Vee blender. The powders were canned in 2-inch diameter steel containers, evacuated, sealed and extruded at a 16:1 area reduction ratio at 1250 K. The extruded bars were then cut into 2-inch sections and the can removed by grinding. A homogenization treatment was then
performed at 1373 K for 48 hours in an argon atmosphere, followed by furnace cooling. A few of the specimens received a longer treatment for 100 hours at 1423 K. Tensile bars were then machined by centerless grinding. To avoid any residual effects from the machining, the sample bars were electropolished in a 10% perchloric acid - 90% methanol solution, maintained at a temperature of 253 K and at a voltage of 10 V.

The chemical analysis of the two alloys shows the binary alloy (referred to as the Fe-Al alloy) to contain 51.5 at% Fe and 48.5 at% Al and the ternary alloy (referred to as the Fe-Al-B alloy) to contain 53.1 at% Fe, 46.1 at% Al and 0.78 at% B. The slight difference in stoichiometry of the two alloys is not considered to be very critical in this study.

The tensile specimens were tested in vacuum at a strain rate of -3x10^-3 sec^-1 at temperatures ranging from 300 K to 680 K. Fracture surfaces were examined using scanning electron microscopy. The substructure of untested samples were examined using a Phillips 400T transmission electron microscope operated at 120 kV. TEM specimens were prepared by electropolishing in a twin jet polisher using a 33% HNO3 - Methanol solution. The solution was maintained at a temperature of 253 K and at a voltage of 30 V.

3. RESULTS

The microstructures of both the alloys after the homogenization treatment reveal that the grains are equiaxed, indicating that recrystallization has occurred. Prior particle boundaries decorated by oxide particles and some residual porosity are also evident, as illustrated in Figure 1. No difference is found between the optical microstructures of the Fe-Al specimens given slightly different heat treatments. The Fe-Al-B specimen displays a structure which appears to contain a significant number of second phase particles which are suspected to be borides. A detailed evaluation of these particles forms part of an ongoing study.

A transition from brittle behavior at low temperatures to ductile behavior at high temperatures is clearly evident in both the alloys. Engineering stress- strain curves for the two alloys at different temperatures are compared in Figure 2 and the tensile properties measured are reported in Table I. The Fe-Al tensile specimens fractured in a totally brittle manner up to 600 K, whereas at 640 K, a significant amount of ductility is observed. One specimen exhibited ~ 8% elongation to fracture and another specimen (the one which was heat treated longer) tested at the same temperature of 640 K resulted in ~ 55% elongation to fracture after extensive necking elongation. The alloy containing boron, Fe-Al-B, remains brittle at 300 K, but begins to show some ductility at 560 K, suggesting a modest decrease in the ductile to brittle transition temperature with boron addition.

The addition of boron substantially increases the strength of Fe-Al, in addition to slightly lowering the ductile to brittle transition temperature. This is observed not only in the low temperature brittle fracture regime, but also in the higher temperature ductile fracture regime where both the yield stress and ultimate tensile strength are higher for the samples containing boron.
Of particular importance, however, is the observation that boron addition results in a change in the fracture mode from intergranular to transgranular. Figure 3 shows typical fracture surfaces from specimens tested below and just above the ductile to brittle transition temperature for both alloys (i.e at 300 K and 640 K). The Fe-Al specimens display totally intergranular fracture at 300 K whereas the Fe-Al-B specimens display totally transgranular cleavage fracture at 300 K. At temperatures just above the ductile to brittle transition temperature (640 K), the intergranular mode for Fe-Al and the transgranular mode for Fe-Al-B are still maintained even though some ductility is observed. The specimen of Fe-Al which showed the large ductility at 640 K demonstrated typical dimple rupture features as illustrated in Figure 4.

Typical dislocation substructures observed with TEM are shown in Figure 5. The structure of the Fe-Al consists of dislocations with definite orientations. The dislocations display sharp jogs and no evidence of faulting is observed. The addition of boron, however, has a dramatic effect on the dislocation substructure. The dislocations display smoother jogs and less of a tendency to lie in specific orientations. In addition, a considerable number of features were observed which are interpreted to be stacking faults.

4. DISCUSSION

The most significant finding from this study is that the addition of boron has resulted in strengthening of the FeAl grain boundaries relative to the grains as evidenced by the change in fracture mode from intergranular to transgranular fracture. This result is similar to the effect of boron in Ni$_3$Al. It is possible to only speculate that this effect is due to boron segregating at the grain boundaries, until it can be verified by Auger Spectroscopy. However, no measurable ductility at room temperature is observed, inspite of this fracture mode change. One possibility is that the boron addition and/or stoichiometry of the alloy are not at the optimum values. The sensitivity of this kind of fracture transition to slight changes in composition has been very clearly established in Ni$_3$Al. The level of boron in this study (0.78 at%) is believed to be much higher than that needed for grain boundary cohesion alone. Another possibility, as discussed in the introduction, is the difficulty of cross-slip contributing to early crack formation before plastic deformation spreads across grain boundaries.

An attempt is made here to correlate the increase in strength and slight decrease in the ductile to brittle transition temperature with addition of boron. The stress required to initiate plastic deformation in any alloy typically decreases with temperature as illustrated in Figure 6. Included on the figure are near horizontal lines representing intergranular fracture stress for the case of weak grain boundaries and transgranular cleavage stress for the case of strong grain boundaries. The stress for intergranular brittle fracture will be lower than the stress for transgranular brittle fracture. The competition between the stress required to initiate plastic deformation and the stresses needed for either intergranular or transgranular brittle fracture determines the fracture mode at any temperature. If the stress required to initiate intergranular or transgranular brittle fracture is less than that required to cause plastic flow, the specimen will break in
a brittle manner. Whereas, if the stress required to initiate plastic flow is lower than the brittle fracture stress, as it is at higher temperatures, then the specimen exhibits ductility. Hence, Figure 6 schematically illustrates how the ductile to brittle transition temperature is obtained at the intersections between these curves.

Strengthening of the grain boundaries by addition of boron can result in a significant decrease in the ductile to brittle transition temperature if the stress required to initiate plastic deformation (the curve for Fe-Al) remains unchanged as illustrated by points A and B in Figure 6. Point A represents the transition temperature when the grain boundaries are weak and hence brittle fracture occurs intergranularly. Point B represents the transition temperature when the grain boundaries are strengthened so that brittle fracture occurs by transgranular failure.

However, if the matrix also becomes stronger, the curve for the stress required for plastic deformation will shift upwards (curve for Fe-Al-B) and the fracture transition temperature shifts to a higher value (point C), thereby neutralizing some of the advantage gained by strengthening the grain boundaries.

Using this approach, it is postulated that the higher strength of the Fe-Al-B matrix (as evident from Figure 2) has reduced the effect of grain boundary strengthening on the ductile to brittle transition temperature in the alloys investigated. The grain boundaries, however, remain stronger than the matrix resulting in a transgranular fracture mode and a significantly stronger alloy.

The observation of intergranular fracture in Fe-Al even at temperatures where some ductility starts appearing, is probably a reflection of a delicate balance between the grain boundary strength and the grain matrix strength. Although the grains deform initially without grain boundary decohesion, work hardening can strengthen the grains relative to the grain boundaries, resulting in intergranular failure. Higher temperatures, where work hardening cannot be expected to make the grains stronger than the grain boundaries, will therefore result in typical ductile fracture features. The fact that one specimen tested at 640 K showed extensive ductility with ductile fracture features whereas another specimen tested at the same temperature showed less ductility with intergranular fracture features, is believed to be either a result of changes in the material caused by the slightly different heat treatment or a result of experimental scatter in the properties at temperatures very close to the transition temperature.

The transgranular fracture of Fe-Al-B at 560 K where some ductility is observed is probably a result of a similar balance between the transgranular cleavage stress and the stress required for plastic deformation of the matrix.

Based on the results of the limited TEM information it is only possible to speculate on the reasons for the increase in the strength of the matrix with the addition of boron. As discussed earlier, the addition of boron resulted in a dislocation structure containing stacking faults as opposed to
a non-faulted structure in the material without boron. The presence of these stacking faults suggests a lowering of stacking fault energy (the nature of the stacking faults being unknown at present), making it more difficult for slip to occur and possibly resulting in the observed increase in matrix strength.

5. CONCLUSIONS

The addition of a small amount of boron to FeAl has been shown to result in a change in fracture mode from intergranular to transgranular at low temperatures. The fracture, however, remains brittle at room temperature. The strength of the alloy is increased very significantly and the ductile to brittle transition temperature is lowered slightly with boron addition. These results are rationalized in terms of grain boundary strengthening due to boron segregation at grain boundaries and strengthening of the matrix.

ACKNOWLEDGEMENTS

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REFERENCES


Figure 1. Comparison of Optical Micrographs of the (a) Fe-Al (b) Fe-Al-B Alloys Illustrating Recrystallized Grains and Prior Particles Boundaries in Both Alloys.
Figure 2. Comparison of Engineering Stress-Strain Curves for (a) Fe-Al and (b) Fe-Al-B Alloy.
Figure 3. Comparison of the Fracture Surfaces for the Two Alloys: (a) Fe-Al and (b) Fe-Al-B alloy at Two Different Temperatures. (Scanning Electron Micrographs, SEM).
Figure 4. SEM Fracture Surface of Fe-Al Alloys Showing Large Ductility and Typical Ductile Fracture at 640 K.

Figure 5. Transmission Electron Micrographs Illustrating the Major Differences in the Substructure of the Two Alloys (a) Fe-Al and (b) Fe-Al-B.
Figure 6. A Schematic Representation of a Possible Explanation for the Observed Differences in the Behavior of the Two Alloys.
Table I

Results of Tensile tests on Fe-Al and Fe-Al-B as a function of Temperature. Constant strain rate of \(-3 \times 10^{-3}\) sec\(^{-1}\).

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<td>300 K</td>
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<td>560 K</td>
<td>137</td>
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<td>600 K</td>
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<td>130</td>
<td>2.5</td>
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<tr>
<td>640 K(1)</td>
<td>89</td>
<td>92</td>
<td>8</td>
<td>116</td>
<td></td>
<td>129</td>
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<td>640 K(2)</td>
<td>71</td>
<td>85</td>
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Y.S. : 0.2 % offset yield strength for specimens which exhibited plastic yielding.

T.S. : Ultimate tensile strength for specimens which showed maximum load followed by necking elongation.

F.S. : Stress at which brittle fracture occurred before yielding for brittle specimens.

OR Stress at which ductile fracture occurred before necking elongation for ductile specimens.

El% : Percent elongation to failure in a gage length of 3 cm. for ductile specimens.
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