Tensile response of an Fe–40Al–0.7C–0.5B alloy

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Abstract

The uniaxial tensile properties of a B2 Fe–40Al based alloy containing 0.7 at.% carbon and 0.5 at.% boron have been obtained at room and elevated temperatures in air as a function of strain rate and compared to the response of a similar alloy without boron (i.e. Fe–40Al–0.6C). Furthermore, tests were also conducted as a function of strain rate in oxygen at room temperature to elucidate the effect of environment on these properties. Both alloys contain a dispersion of a perovskite carbide within the grains and at grain boundaries. In addition, the quaternary alloy contains a fine dispersion of metastable borides and complex faults within the grains. Yield stress and tensile elongation appear strain rate insensitive (in the regime tested) for the boron-containing alloy at room temperature in oxygen, whereas some loss in elongation is encountered at the slowest rate in the ternary alloy; in all cases, fracture path is intergranular. In air, at room temperature, a strong strain-rate dependence of elongation is recognized in both alloys, their response being identical at the faster rates but diverging at the slow rates. A strong correlation is noted between the increase in percent transgranular cleavage and the drop in tensile elongation. The brittle-to-ductile transition temperature is sensitive to strain rate, shifting to higher temperatures rapidly with increasing strain rate. A variety of fracture paths is encountered depending on test temperature and strain rate. © 2002 Acta Materialia Inc. Published by Elsevier Science Ltd. All rights reserved.

Keywords: Iron aluminides; Embrittlement; Strain rate; Ductility; Fracture

1. Introduction

The physical and mechanical properties of binary FeAl with the B2 structure existing over a range of compositions skewed to the Al-deficient side have been extensively studied and the results have been systematically documented in several excellent recent reviews [1–4]. Whereas stoichiometric and near-stoichiometric Fe-rich FeAl have been shown to be intrinsically brittle at room temperature, it has been clearly demonstrated that compositions around Fe–40 at.%Al demonstrate adequate ductility at room temperature when tested in oxygen but the ductility is significantly reduced when tests are conducted in air [5,6]. This loss in ductility is attributed to environmental embrittlement caused by water vapor in air. A direct manifestation of this effect is the continuous decrease in ductility with test strain rate at room temperature [7,8]. The fracture process in FeAl is complex. Whereas, off-stoichiometric compositions contain-
ing Al levels in excess of 42–43 at.% typically fail substantially intergranularly, compositions with Al level less than 36 at.% Al fail transgranularly; compositions in the intermediate regime show mixed characteristics [3,9–11]. When environmental effects come into play, the energetic balance between transgranular and intergranular failure modes can be affected, the extent being dependent on the amount of hydrogen ingested.

The effects of strain rate on (i) the variation of tensile ductility with test temperature, and (ii) ductility at elevated temperatures have also been examined. The existence of a brittle-to-ductile transition temperature (BDTT) range in Fe–40Al has been reported [10]. Tensile tests conducted by Pocci et al. [12] on an Fe–40Al alloy containing small amounts of Zr, C and B at a temperature as high as 950 °C illustrated a ductile-to-brittle transition at a strain rate between 0.5 and 0.8 s⁻¹; the strain rate dependence was preserved at an even higher temperature of 1100 °C, although the transition occurred between 4.6 and 5.3 s⁻¹. In addition, these authors [12] also demonstrated a shift in the BDTT from 400 to 500 °C by comparing elongation versus test temperature curves generated at strain rates of 3 × 10⁻⁴ and 3 × 10⁻² s⁻¹. In this case, the alloy examined also contained minor levels of Zr, C, B and Ce.

Relatively fewer studies have been undertaken to examine the effect of macroalloying and of the presence of second phase on the mechanical properties of Fe-rich FeAl [13–16]; there have however been several reports on the effects of minor levels of boron addition to FeAl on point defects migration, flow and fracture [11,17–22]. It was shown that boron (i) accelerates the migration of thermal vacancies in FeAl, (ii) segregates to grain boundaries, and (iii) suppresses propensity to intergranular fracture and thereby provides some enhancement in ductility.

Recently, Pang and Kumar [23] reported on the effect of strain rate on the tensile elongation in air at room temperature of a ternary Fe–40Al–0.5C alloy. The presence of carbon in this alloy resulted in the precipitation of perovskite ternary carbidies (Fe₃AlC₀.₅) with a lath morphology at grain boundaries and within the grains. In this multiphase alloy, elongation decreased with decreasing strain rate as anticipated but reached a minimum at a low strain rate before gradually increasing again at even lower rates. Furthermore, at any given strain rate, the ductility of the ternary alloy was higher than that of binary Fe–40Al. Pang and Kumar [23] also noted that the fracture surface displayed a predominantly intergranular failure mode at the faster strain rates but gradually transitioned to a predominantly transgranular mode at the slower strain rates (> 80% transgranular fracture at the ductility minimum). Interestingly, the fracture reverts to a substantially intergranular mode once again at the slowest rates, coincident with the ductility increase. The gradual increase in ductility at the slowest rates was attributed to competition between surface re-oxidation kinetics and amount of hydrogen generated by water vapor decomposition at the surface.

In this paper, the effect of the combined presence of carbon and boron on tensile properties is presented and discussed in relation to the microstructure of this alloy. The carbon level in the alloy examined in this study is comparable to that in the ternary alloy previously examined [23] and therefore where pertinent, results of the ternary alloy are presented for comparison. Further, the boron level in the alloy in the present study is significantly higher than those reported in the literature; this leads to the precipitation of borides and the formation of planar defects. The stability of the borides and the planar defects has been examined in detail and reported elsewhere [24,25].

2. Experimental procedure

The alloy used in the present investigation was made by induction melting 99.99% pure aluminum, an Al–Fe master alloy and an Al–B master alloy in appropriate proportions in a zirconia crucible and by tilt-pouring the melt into a graphite mold. The resulting ingot was homogenized to eliminate coring, canned in a mild steel container and extruded into a cylindrical rod at 1000 °C using an extrusion ratio of 16:1. Chemical analysis of the extruded specimens yielded a boron content of 0.53 at.% in the Fe–40Al–0.5B alloy, but in
addition, the presence of 0.7 at. % C was confirmed. The source of carbon in the alloy was either the graphite mold into which the molten metal was poured, or the Fe–Al master alloy.

Flat ‘dog-bone’ tensile specimens with a gage section of 25.4 × 3 × 1.6 mm were electro-discharge machined from the extrusions with the tensile axis lying along the extrusion direction. Tensile specimens of FeAl-based alloys in the as-extruded condition are usually provided a low-temperature anneal for an extended time followed by furnace cooling to room temperature. The purpose of this anneal is to eliminate excess retained vacancies in the as-extruded material [1] and the temperature/time schedule was first determined for maximizing ductility at room temperature for this alloy. Such annealed specimens were ground and mechanically polished prior to testing. The microstructure in this annealed condition, as well as following a variety of heat treatments have been previously described in detail elsewhere [24,25] and only briefly summarized in the next section.

The yield strength, fracture strength and elongation to failure in uniaxial tension of the annealed specimens were obtained (i) as a function of strain rate at room temperature in oxygen and in air, and (ii) as a function of test temperature at different strain rates in air. Additionally, a few specimens that had been heat treated to selectively eliminate second phase(s) were tested in air at room temperature in an attempt to elucidate the contribution of the phase(s) to tensile elongation and fracture. Lastly, the effect of increasing the mobile dislocation density (by prestraining the tensile specimens) on the warm-temperature (300–500 °C) tensile elongation at a nominal strain rate of 10⁻² s⁻¹ was explored. Fracture surfaces in most instances were examined in the scanning electron microscope (SEM) and the relative amounts of intergranular fracture and transgranular cleavage were quantified. Where necessary, transmission electron microscopy (TEM) techniques were used to characterize the microstructure. Thin foil specimen preparation details have been provided in previous papers [23–25].

The specific procedure adopted for the room-temperature tests conducted in oxygen is relevant to appreciate the results obtained. A test chamber with a diffusion pump attachment was used. The chamber was first mechanically pumped down and subsequently diffusion pumped to vacuum levels better than 5 × 10⁻⁶ mm of Hg. Then the diffusion pump had to be shut down and the pump oil allowed to cool before oxygen could be purged in (attempts to do it otherwise resulted in sparking and oil ignition due to a possible minute leak in the diffusion pump gate valve at positive pressure). During the oil cool down (~ 20 min), the mechanical pump continued to operate on the chamber although the vacuum level deteriorated to 10⁻⁴ mm of Hg. The chamber was then repeatedly flushed with oxygen and evacuated to reduce the partial pressure of air (typically 6–8 flushings) before finally being back-filled to atmospheric pressure with oxygen. A static oxygen atmosphere was then maintained during the test.

3. Results

3.1. Microstructure

The extruded and annealed (600 °C/24 h) microstructure consisted of equiaxed grains of the B2 FeAl with a dispersion of other phases at grain boundaries and in the grain interior. X-ray diffraction confirmed them to be perovskite carbides (Fe₃AlC₀.₅), Fe₂B and free carbon in the form of graphite. These phases could be distinguished using the optical microscope by virtue of their shape, size and etching response. The Fe₂B phase often occurred at grain boundaries and the amount of free carbon was minimal. A representative optical micrograph obtained from the transverse section of the extrusion is shown in Fig. 1(a). In addition to these phases, TEM examination confirmed the presence of a metastable, fine, rod-shaped boride phase (termed the ν phase) dispersed in the matrix. These precipitates have their long axis lying along the <001> directions of the B2 FeAl and exhibit a square cross section where the sides of the square are coincident with the <110> directions of the matrix [Fig. 1(b,c)]. Thus, there are three variants of this phase. These rods have aspect ratios that are typically in excess of 10 and their fine-structure and thermal stability have been
Fig. 1. Microstructure of the FeAlBC alloy in the extruded + low-temperature (600 °C) annealed condition: (a) optical micrograph confirming a recrystallized microstructure consisting of at least two types of precipitates identified by x-ray diffraction as Fe₃AlC₀.₅ and Fe₃B; (b–d) TEM images showing the square cross-section of the metastable ν phase bearing a crystallographic orientation relationship with the matrix (b), the rod morphology of the ν phase (c), and the presence of complex planar faults in the matrix (d).

discussed in detail elsewhere [24,26,27]. Furthermore, complex planar faults lying on the cube planes and having their displacement vector in the fault plane in the ⟨001⟩ directions (six fault variants are therefore possible) have also been observed in this alloy [28]. An example of these faults is shown in the bright field image in Fig. 1(d). The fault structure, stability and morphological manifestations have been characterized in detail [25,28]. Often, the ν phase precipitates at the ends of these faults [Fig. 1(b)].

3.2. Tensile response at room temperature

The tensile response in air at room temperature of the as-extruded Fe–40Al–0.7C–0.5B alloy (hereafter referred to as the FeAlBC alloy) was first obtained. Next, the as-extruded material was subjected to several low-temperature annealing schedules to reduce the excess retained vacancy concentration and thereby maximize tensile ductility. For binary Fe–40Al, a 400 °C/5 days schedule has been reported to be adequate [1]; in the case of the ternary Fe–40Al–0.6C alloy, it was previously shown that a two-stage anneal of 400 °C/72 h + 500 °C/24 h produced the best ductility [29]. However, for the FeAlBC alloy, it was found that the two-stage heat treatment used for the ternary alloy was inadequate (Table 1). A heat treatment of 600 °C/24 h yielded the best combination of strength and ductility and thus, this heat treatment was used as a standard anneal for all subsequent test specimens.

Next, the tensile elongation of the FeAlC alloy and the FeAlBC alloy in the appropriate extruded + annealed conditions (i.e. 400 °C/72 h + 500 °C/24 h for the ternary alloy and 600 °C/24 h for the quaternary alloy) are compared as a function of strain rate at room temperature in dry oxygen in Fig. 2. Except at the slowest strain rate examined, for both alloys, elongation is insensitive to strain rate; furthermore, the ternary alloy exhibits higher elongation compared to the boron-containing alloy. The lower elongation for the ternary alloy at the slowest rate examined could be a consequence of small amounts of residual water vapor in the chamber. The sensitivity to embrittlement of FeAl to a few ppm of hydrogen [30] combined with the slow test rate likely produced the decrease in elongation. Such a loss is however not evidenced in the FeAlBC alloy and is an indication of its reduced sensitivity to embrittlement. The overall lower elongation values for the FeAlBC alloy

Table 1

<table>
<thead>
<tr>
<th>Heat treatment schedule</th>
<th>Yield strength (MPa)</th>
<th>Elongation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-extruded</td>
<td>498</td>
<td>2</td>
</tr>
<tr>
<td>As-extruded + annealed at 400 °C/72 h</td>
<td>374</td>
<td>2.8</td>
</tr>
<tr>
<td>+500 °C/24 h</td>
<td></td>
<td></td>
</tr>
<tr>
<td>As-extruded + annealed at 600 °C/24 h</td>
<td>345</td>
<td>5.0</td>
</tr>
<tr>
<td>As-extruded + annealed at 800 °C/24 h</td>
<td>267</td>
<td>4.5</td>
</tr>
</tbody>
</table>

*a All specimens were furnace-cooled to room temperature; tensile tests were conducted at room temperature in air at a nominal strain rate of 4.2 × 10⁻⁴ s⁻¹.
Fig. 2. Variation in tensile elongation with test strain rate at room temperature in oxygen for the extruded and low-temperature annealed FeAlC and FeAlBC alloys.

(~10%) relative to the ternary FeAlC alloy (~15–16%, Fig. 2) is believed to be a consequence of a higher amount of coarse precipitates (Fe2B) at the grain boundaries and within the grains. The fracture surfaces of several of the specimens tested in oxygen were examined in the SEM. Representative micrographs from the ternary FeAlC specimens confirm substantially intergranular failure for all the strain rates examined [Fig. 3(a,b)].

Similar tensile tests were also conducted in air at room temperature over a wide range of strain rates (~4 × 10^{-2} to 4 × 10^{-8} s^{-1}). The effects of strain rate on yield strength and fracture strength for the FeAlC and FeAlBC alloys are shown in Fig. 4(a) and the change in elongation with strain rate is presented in Fig. 4(b). The effect of strain rate on tensile elongation of binary Fe–40Al at room temperature in air taken from the published literature [7] is included in Fig. 4(b) for purposes of comparison.

Several interesting observations can be made. The yield strength is essentially independent of strain rate for the ternary and quaternary alloy. The yield strength of the ternary alloy is ~300 MPa whereas that for the FeAlBC alloy is ~350 MPa. The fracture strength of the ternary alloy however decreases with decreasing strain rate, reaches a minimum and then increases again with further decrease in strain rate. For the quaternary alloy, a similar response is noted except that a distinct minimum is not observed in strength but a fairly strain rate insensitive regime exists at the slower rates (from ~4 × 10^{-5} to 4 × 10^{-8} s^{-1}, see Fig. 4(a). The elongations for the ternary and quaternary alloys are essentially identical over the strain rate regime of ~4 × 10^{-2} to 4 × 10^{-5} s^{-1} and both display a decrease in elongation with decreasing strain rate. At the strain rate of ~4 × 10^{-2} s^{-1}, the elongation for the FeAlBC alloy

Fig. 3. SEM images confirm a predominantly intergranular failure mode in the tensile specimens tested in oxygen at room temperature; (a) FeAlC; 4 × 10^{-4} s^{-1} and (b) FeAlC; 4 × 10^{-7} s^{-1}.
is comparable to the values obtained in oxygen (Fig. 2), suggesting minimal loss in ductility (if any) due to environmental embrittlement. In contrast, the elongation to failure measured for the ternary FeAlC alloy even at the faster rate of ~4 × 10^{-7} s^{-1} is lower than the average value measured for this alloy in oxygen (except at the slowest rate in oxygen, Fig. 2), implying that environmental effects are present even at this fast strain rate when tests are conducted in air. A comparison of the elongation values recorded for the ternary and quaternary alloys with those from the literature for binary Fe-40Al [7] clearly demonstrates that at any given strain rate, the ternary and quaternary alloys are superior.

The response of the quaternary alloy diverges from that of the ternary alloy at strain rates below ~4 × 10^{-5} s^{-1} [Fig. 4(b)]. Whereas the elongation value for the ternary alloy continues to decrease with decreasing strain rate until at least a strain rate of ~4 × 10^{-7} s^{-1}, the quaternary alloy displays elongation values (~3.5%) that are insensitive to strain rate in the strain rate regime from ~4 × 10^{-5} to 4 × 10^{-8} s^{-1}. In the case of the ternary FeAlC alloy, at the very slow rates (~4 × 10^{-7} to 4 × 10^{-8} s^{-1}) ductility is regained to an extent for reasons previously discussed [23].

Fracture surfaces were examined, and at the fastest strain rates, fracture was predominantly intergranular in both alloys. With decreasing strain rate, increasing amounts of transgranular cleavage was mixed with intergranular failure. The relative amounts of the two fracture modes were quantified and the results are shown in Fig. 5. Coincident with the elongation response in Fig. 4(b), the ternary and quaternary alloys show similar trends in the strain rate regime ~4 × 10^{-2} to 4 × 10^{-6} s^{-1} with the fraction of transgranular cleavage being somewhat higher in the ternary alloy. The percentage transgranular cleavage essentially remains flat at slower strain rates in the FeAlBC alloy whereas it increases steeply to >80% for the ternary alloy tested at ~4 × 10^{-7} s^{-1}. At even slower rates, the fraction transgranular cleavage drops down once again for the ternary alloy to values comparable to typical iron microstructures.
to the FeAlBC alloy. The fracture behavior of the FeAlBC alloy is somewhat unexpected as other authors [4,18] have claimed that boron segregates to grain boundaries in FeAl alloys, enhances boundary strength and encourages transgranular cleavage.

A previous effort [24] had shown that if the as-extruded FeAlBC material is annealed at 800 °C for 24 h and subsequently furnace cooled to room temperature, the complex faults as well as the ν phase precipitates that are present in the matrix in the as-extruded + 600 °C/24 h annealed specimens can be completely eliminated (recall that both these microstructural features are metastable). The boron resulting from the elimination of these features leads to the precipitation of some relatively coarse Fe₂B precipitates at grain boundaries and in the grain interior. Except for these Fe₂B precipitates, the microstructure appears remarkably similar to the ternary FeAlC alloy. Examining the tensile response of such heat treated specimens at slow strain rates in air at room temperature and comparing the results to those for the as-extruded + 600 °C/24 h annealed specimens could help understand the role of these complex faults and the ν phase precipitates in affecting the resistance to hydrogen embrittlement [recall from Fig. 4(b) that the elongation in air at a strain rate below \(4 \times 10^{-5} \text{s}^{-1}\) is essentially strain rate insensitive for the FeAlBC alloy in contrast to the FeAlC alloy].

The variation in elongation in air in the slow strain rate regime is compared in Fig. 6 for the FeAlBC alloy subjected to the standard 600 °C/24 h heat treatment with that for specimens subjected to the 800 °C/24 h heat treatment. The data points for the 600 °C/24 h are the same as those in Fig. 4(b). Also included in Fig. 6 are the data for the ternary FeAlC alloy that show a minimum in elongation at a nominal strain rate of \(4 \times 10^{-7} \text{s}^{-1}\). When the ν phase and the planar faults in the FeAlBC alloy are eliminated (confirmed by electron microscopy, see Fig. 7(a,b)), the elongation response over this strain rate regime resembles the response of the ternary FeAlC alloy that a minimum in ductility is noted. This difference in response of the FeAlBC alloy following the 800 °C/24 h treatment relative to the response obtained for the specimens subjected to the 600 °C/24 h treatment is attributed to the faults and long rod-shaped ν phase precipitates (with their large surface to volume ratio) acting as possible hydrogen traps within the grains and retarding embrittlement kinetics. This line of argument is consistent with the reduced level of transgranular cleavage in the FeAlBC alloy containing these microstructural features at the slow strain rates as compared to the ternary FeAlC alloy (Fig. 5). Lastly, a comparison of the percent transgranular cleavage as a function of test strain rate in the two sets of FeAlBC specimens (600 °C/24 h heat treatment and 800 °C/24 h heat treatment) is presented in Fig. 8. The elimination of the ν phase and the planar faults from within the grains appears to promote transgranular cleavage to some degree at the strain rates of \(4 \times 10^{-6}\) and \(4 \times 10^{-4} \text{s}^{-1}\). The fraction transgranular cleavage at each of these two strain rates is more in line with observations for the ternary FeAlC alloy (Fig. 5). At the two slowest rates, the percent transgranular cleavage is comparable in the two sets of specimens. Once again, we note a correlation between the percent transgranular cleavage and tensile elongation when influenced by the environment.
Fig. 7. Bright field TEM images of the υ phase precipitates following an anneal at 600 °C for 24 h (a), and their absence following an anneal at 800 °C for 24 h followed by step-cooling to room temperature (b).

3.3. Tensile response as a function of temperature

The tensile response of the annealed (600 °C/24 h) quaternary FeAlBC alloy was obtained as a function of temperature in the temperature range 25–700 °C at nominal strain rates of 10⁻⁵, 10⁻² and 1 s⁻¹. These tests were conducted in air, and whereas the highest elongation is obtained at the fastest strain rate at room temperature [Fig. 9(a)], the trend reverses at higher temperatures (~200 °C); if the onset of the ‘upward swing’ in elongation is used as a marker for the brittle-to-ductile transition (BDT), then it is clear that the BDT shifts to higher temperatures with increasing test strain rate. At the fastest strain rate (1 s⁻¹), the ductility remains essentially the same at 700 °C as at room temperature and no ‘upward swing’ is recognized. It is pertinent to reiterate at this juncture that interpretation of results from tests conducted at temperatures higher than 700 °C will be complicated by issues related to microstructural instability associated with precipitate dissolution.

The corresponding variation in yield strength with test temperature and strain rate is shown in Fig. 9(b). At the slowest strain rate, the yield stress is virtually unchanged over the temperature regime (100–500 °C) and is ~300 MPa. A comparable value of yield stress was obtained at the faster strain rate of 10⁻² s⁻¹ at 200 °C although the yield stress increases rapidly to ~400 MPa at 400 °C but then levels off to about the same value at 500 °C. At the fastest strain rate of 1 s⁻¹, the yield stress rises sharply from ~390 MPa at 500 °C to ~540 MPa at 700 °C. In contrast, at this strain rate (1 s⁻¹), over the 500–700 °C temperature regime, fracture stress continually decreases from ~660 to ~520 MPa [Fig. 9(c)] while ductility increase, if any, is modest [Fig. 9(a)]. Note that at 700 °C, the yield stress and fracture stress are similar [compare Fig. 9(b) and (c)], implying that thermally activated processes compete effectively with work hardening. At the two slower strain rates, fracture stress first increases with temperature but then rap-
idly drops off with further increases in temperature [Fig. 9(c)]. The temperature at which peak fracture stress occurs appears to depend on the strain rate. At the slowest strain rate, specimens tested at 400 °C and 500 °C exhibit an ultimate tensile strength, implying local necking and overload failure. The onset of local necking occurs at a lower strain at 500 °C than at 400 °C. The early occurrence of necking explains why at 500 °C, the specimen tested at the slowest strain rate exhibits tensile elongation that is marginally lower than the corresponding value at 400 °C.

Low magnification SEM images of the fracture surfaces of the tensile specimens that exhibited a maximum in tensile strength prior to failure are shown in Fig. 10(a)-c. In each of these images, the entire specimen cross-section (that was rectangular prior to testing) at the fracture surface is seen. Clearly, necking is much more severe at 500 °C than at 400°C [compare Fig. 10(a) and (b)] for specimens tested at a nominal strain rate of $10^{-5}$ s$^{-1}$. Thus, although tensile elongation values are similar for the two specimens, the local reduction in area is much more for the 500 °C specimen. Additionally, while the approximate rectangular cross section is maintained in the 400 °C specimen, this is not the case in the 500 °C specimen where the perimeter of the neck cross section develops significant curvatures. A large number of coarse voids are present on the fracture surface of the 500 °C specimen whereas this is not evident at 400 °C. The fracture surface of the specimen tested at 600 °C but at the strain rate of $10^{-2}$ s$^{-1}$ [Fig. 10(c)] bears a striking resemblance in cross-section geometry to the fracture surface seen in Fig. 10(b). A comparison of the microstructural details of the fracture surfaces in Fig. 10(b) and (c) confirms significantly less cavitation in the 600 °C specimen tested at the faster strain rate. It is pertinent to recall that the specimen tested at the faster strain rate at 600°C fractured with a tensile elongation that was double that of the 500 °C specimen tested at the slower strain rate. Necking was not evident in the 700 °C specimen tested at 1 s$^{-1}$.

Fracture surfaces of the tensile specimens were examined at higher magnifications and representative micrographs are shown in Fig. 11(a–f). Fig. 11(a–c) show the fracture surface characteristics as a
function of test temperature (300, 400 and 500 °C) for tests conducted at the slowest strain rate of $10^{-5}$ s$^{-1}$, whereas Fig. 11(e,f) compares the features following tests at 500 °C and 700 °C at a strain rate of 1 s$^{-1}$ respectively. A comparison of Fig. 11(c,d,e) provides an appreciation for the effect of strain rate ($10^{-5}$, $10^{-2}$, and 1 s$^{-1}$ respectively) at a constant temperature of 500 °C. For tests conducted at the slowest rate, at 300 °C, the fracture path is substantially intergranular even though a tensile elongation of ~15% is realized. Secondary intergranular cracks are evident on the fracture surface [Fig. 11(a)]. For the specimen failed at 400 °C at the slowest strain rate [Fig. 11(b)], where an elongation of over 30% was realized, the fracture surface illustrates grains that appear contorted and significant secondary cracking along what are thought to be original grain boundaries but an overall fracture mode that can perhaps be best described as transgranular cleavage. It is relevant to recall that although the overall measured elongation is a little over 30%, the specimen experiences significant necking [see Fig. 9(c) and 10(a)] so that the local strain in the vicinity of the fracture surface is significantly higher and the associated stress state at the necked region prior to fracture is complex. Thus, lateral tensile stresses can potentially be the source of these secondary cracks along grain boundaries, particularly if the grains in the necked region are significantly elongated in the tensile direction; however, creep cavi-
tation and voids are not observed. A high magnification image of the fracture surface shown in Fig. 10(b) (500 °C, 10^{-5} \text{s}^{-1}) is presented in Fig. 11(c). The frequent presence of large cavities (~10 \mu m in diameter) and the general appearance of the fracture surface confirm the dominance of creep mechanisms during deformation and failure. In contrast, when a specimen is tested at 500 °C at a faster strain rate of 10^{-2} \text{s}^{-1}, fracture mode appears to be a mixture of intergranular failure and transgranular cleavage [Fig. 11(d)]. Secondary intergranular cracks are again evident although not as extensively as in Fig. 11(b); in addition, in several locations elongated grains are recognized (for example two such features are marked in Fig. 11(d) by A and B) that appeared to have eventually failed by a combination of intergranular separation (planes parallel to the tensile axis) and transgranular cleavage modes (plane normal to the tensile axis). At the fastest strain rate of 1 \text{s}^{-1} at 500 °C, fracture mode is dominated by transgranular cleavage [Fig. 11(e)] and this failure mode persists at 700 °C [Fig. 11(f)]. Interestingly, this failure mode is not observed in specimens tested at the slower strain rates of 10^{-2} and 10^{-5} \text{s}^{-1} at lower temperatures (200 and 300 °C) where intergranular failure is the dominant mode.

The microstructure in several of the tensile specimens on a plane orthogonal to the fracture surface but adjacent to it was examined and representative micrographs are shown in Fig. 12(b–d). A comparison of the microstructures of the grip and gage sections following the 700 °C/1 \text{s}^{-1} test illustrates a fully recrystallized structure with similar grain size [Fig. 12(a,b)] implying the absence of any dynamic recrystallization or subsequent static recrystallization during cooling arising as a consequence of the tensile test. The recrystallized microstructure observed in these two micrographs is a consequence of hot extrusion. It is relevant to note that in this alloy, for all temperatures investigated in this study, the carbides at grain boundaries and the carbides and borides in the grain interior remain intact and make grain growth difficult. Thus any recrystallization that occurs during a 700°C tensile test or during subsequent cooling to room temperature will likely reflect a refinement in grain size. The edge of the fracture surface can be seen in the top portion of Fig. 12(b) and the dominance of transgranular cracking is evident and in agreement with Fig. 11(f). Microstructures from the gage section of the 500 °C/1 \text{s}^{-1}, 500 °C/10^{-2} \text{s}^{-1}, 400 °C/10^{-2} \text{s}^{-1}, and 300 °C/10^{-5} \text{s}^{-1} were all similar to that observed in Fig. 12(b) and confirm that recrystallization likely did not occur during or after the tensile test. In all these cases, tensile elongation recorded was less than twenty percent. In contrast, the specimen deformed at 400 °C/10^{-5} \text{s}^{-1}, demonstrated ~30% elongation [Fig. 9(a)], a distinct ultimate tensile strength [Fig. 9(c)] and local necking [Fig. 10(a)]. The microstructure in the immediate vicinity of the fracture surface of this specimen [Fig. 12(c)] consists of elongated unrecrystallized grains. However, regions in the gage section away from the necked section exhibit
equiaxed grains much like those observed in Fig. 12(b). Thus, there is no evidence of recrystallization during or after the test even in a specimen exhibiting \( \geq 30\% \) elongation at 400 °C. Finally, the microstructure in the region adjacent to the fracture surface of the specimen that was tested at 600 °C/\( 10^{-2} \) s\(^{-1} \) is shown in Fig. 12(d). This specimen exhibited tensile elongation of \( \sim 60\% \) prior to failure. Once again there is no obvious evidence of recrystallization; the original grains are elongated in the direction of the tensile axis and moreover, regions away from the fracture surface also showed grains with a substantial aspect ratio. Thus, recrystallization does not appear to be a prerequisite for obtaining large values of tensile elongation in this alloy.

The substructure of the deformed specimens (700 °C/1 s\(^{-1} \) and 600 °C/\( 10^{-2} \) s\(^{-1} \)) was examined in the TEM. Representative bright field images from the section adjacent to the fracture surface of the 700 °C/1 s\(^{-1} \) specimen [Fig. 13(a,b)] confirm the presence of a high density of dislocation tangles but no clear evidence for cell walls or subgrains. However, parallel regions of bright and dark contrast seen in these images (particularly in the low magnification image in Fig. 13(a)) are indicative of the beginnings of subgrain evolution and perhaps, the presence of texture. In contrast, the 600 °C/\( 10^{-2} \) s\(^{-1} \) specimen illustrates a well-developed subgrain structure but not recrystallization. The necked region immediately adjacent to the fracture surface is shown in Fig. 13(c) whereas, the image in Fig. 13(d) corresponds to a region about 5 mm away from the fracture surface. There appears to be no significant differences in the subgrain structure and subgrain size. The microstructure observed in Fig. 13(c,d) could either be a consequence of dynamic recovery, or a static recovery process that occurred subsequent to the test during cooling down to room temperature, although the latter is thought to be unlikely as the specimen was extracted from the furnace immediately after fracture and air-cooled to room temperature.

Prior efforts [31,32] on the companion B2 compound NiAl have demonstrated that a small amount of prestrain can be beneficial to the room-temperature ductility and fracture toughness of that compound. However, it was not clear from those studies [31,32] whether the beneficial effects are a direct consequence of the increased mobile dislocation density, or an indirect effect due to interstitial solute gettering by the prestrain dislocations. In this effort, tensile specimens were provided 0.5% plastic prestrain at room temperature and then tested at the moderately high strain rate of \( 10^{-2} \) s\(^{-1} \) at 300, 400 and 500 °C to examine potential beneficial effects. It is argued that if strain rate dependency of the BDT is associated with difficulty in dislocation nucleation, then the presence of the prestrain dislocations would assist plastic deformation. If however, the problem is related to dislocation mobility, then it is unlikely that any direct benefit can be realized from the prestrain. Since environmental embrittlement is an issue at room temperature, the specimens were provided a prestrain at a nominal strain rate of \( 10^{-3} \) s\(^{-1} \), and then fine polished to eliminate surface markings resulting from the prestrain. The tensile elongation

![Fig. 13. Bright field images from TEM specimens prepared from the gage sections of fractured tensile specimens. (a,b) 700 °C/1 s\(^{-1} \) specimen—region immediately adjacent to the fracture surface; (c,d) 600 °C/\( 10^{-2} \) s\(^{-1} \) specimen—(c) region immediately adjacent to the fracture surface, and (d) region located about 5 mm away from fracture surface.](image-url)
and yield strength values at the three test temperatures simply overlapped the corresponding values for specimens that had not been prestrained. Thus, there appears to be no beneficial effect of prestraining the specimens.

A fracture map in strain rate-temperature space is presented in Fig. 14 and was generated using experimental data from the ternary FeAlC and quaternary FeAlBC alloys. The hatched area in the vicinity of room temperature, spanning roughly the 10^{-3} to 10^{-7} s^{-1} strain rate regime, is a consequence of environmental embrittlement and would not be present in a similar map generated using data from tests conducted in a dry oxygen environment. A specimen such as the one tested at 400 °C/10^{-5} s^{-1} showed features on the fracture surface that appeared ‘smeared’ and is categorized as ductile transgranular cleavage while the extensive secondary cracking in the necked region indicates intergranular failure and so the specimen is included in the field that is labeled as ‘ductile TC + IG’ in Fig. 14. In contrast, the specimen fractured at 700 °C/1 s^{-1} shows mostly brittle transgranular cleavage. Lastly, the rapid increase in yield stress with temperature at the higher strain rates and the concomitant loss in elongation suggests a loss in toughness at higher temperatures, a phenomenon reflected in Charpy impact tests [33]. The effect of test temperature on fracture mode in Charpy test specimens are also included in Fig. 14.

4. Discussion

In discussing the results presented in the previous section, we focus on the two broad aspects of this study: (i) the effect of microstructure on room temperature tensile response in oxygen and in air, and (ii) the effect of strain rate on the ductile-brittle transition temperature.

The tensile elongation in oxygen for the ternary FeAlC alloy at the faster strain rates is significantly higher than the values reported for binary Fe-40Al in the literature (for example, ~7% at a strain rate of ~1/s according to [7]). Since coarse precipitates at grain boundaries are usually detrimental to ductility, we speculate that elemental carbon segregation to the grain boundaries likely strengthen the boundaries. Additionally, the perovskite carbides at the grain boundaries assume a lath morphology with the laths being inclined to the grain boundary plane [29]. This arrangement forces a crack to repeatedly deflect into the grains in order to propagate along the carbide/matrix interface. The resulting ‘grain boundary facets’ on the fracture surface thus assume a rough fibrous appearance rather than the smooth facets that are typically seen when grain boundary failure occurs in binary FeAl. The loss in elongation for the FeAlC alloy at the slowest strain rate is indicative of hydrogen embrittlement even in pure oxygen; it has been previously shown that it only takes a few ppm of hydrogen to embrittle FeAl [30]. We recall that fracture mode in this condition continues to remain completely intergranular. For the quaternary FeAlBC alloy, elongation values in oxygen are lower than those observed for the FeAlC alloy for comparable test conditions and this is likely a consequence of the coarse Fe2B precipitates additionally present at the boundaries. The elongation values for the quaternary FeAlBC alloy are however still significantly higher than those reported in the literature for the binary Fe–40Al alloy [7] but lower than that reported for an Fe–40Al alloy containing 300 ppm boron tested in.
In the slow strain rate regime (4 × 10−5 s−1), improvement has been previously discussed [23]. The role of grain boundary carbides in causing this appears superior to the binary alloy and the possible predominately intergranular. Both these alloys in this regime, transgranular cleavage fracture plays an important role. We invoke the idea that precipitate-matrix interfaces can act as hydrogen traps [34–36] and that the presence of the fine, numerous rod-shaped ν phase boride precipitates significantly increases the hydrogen trap density thereby effectively decreasing hydrogen arrival at the crack tip and reducing embrittlement through transgranular cleavage. It has previously been shown that hydrogen can lower the cleavage stress in FeAl [37], the extent being dependent on the amount of hydrogen ingested. In contrast, the absence of the boride phase within the ternary FeAlC alloy grains implies fewer traps (carbides are still present within the grains) and continued embrittlement, although less aggressively than in a binary alloy that contains no second phase particles within the grains or at grain boundaries [Fig. 4(b)]. At strain rates below 4 × 10−7 s−1, there is a recovery in ductility for the ternary alloy which is thought to be a consequence of reoxidation kinetics at the surface slip steps effectively competing with the hydrogen generation and transport kinetics [23]. The experimental results in Fig. 6, 7 and 8 are consistent with the arguments presented above.

The effect of strain rate and temperature on the tensile yield stress of B2 FeAl has been previously studied and it has been shown that the yield stress peak shifts to higher temperatures and larger stresses with increase in strain rate [38]. This has been interpreted as being a consequence of the delay in onset of dislocation creep mechanisms [22]. Furthermore, it was concluded that to a first approximation, the test strain rate does not affect the rate of increase in yield strength with temperature in the regime where there exists a positive temperature dependence of yield strength. Such trends are also evident in Fig. 9(b) in this study although the yield strength at a given temperature in the 300–500 °C regime appears to show some dependence on strain rate. The effect of strain rate on the variation of tensile elongation with temperature is less explored. Strain rate has been shown to affect the tensile elongation obtainable at a given elevated temperature [12,29] and the higher ductility at slower strain rates is thought to be a consequence of dynamic recrystallization [1].

In this study, we have shown that dynamic oxygen [4]. Fracture mode in the quaternary FeAlBC alloy remains intergranular, contradicting the observation in literature [4] that boron segregates to grain boundaries and promotes transgranular fracture. Taken together, these observations indicate that boron addition to FeAl may have to be limited to below the saturation level to preclude Fe2B formation to realize substantial improvement.
recrystallization is not a pre-requisite for obtaining large tensile elongation. A specimen with 60% elongation at 600 °C shows a well-defined subgrain structure indicative of dynamic recovery. The 700 °C specimen tested at the fast rate of 1 s⁻¹ however does not show a fully-defined subgrain structure although it demonstrates approximately 10% elongation and yield and fracture strength values that are similar. In contrast, the specimen deformed at 500 °C at the same strain rate shows a lower elongation, a lower yield strength and a higher fracture strength than its 700 °C counterpart. Evidently, the 500 °C specimen work hardens significantly more than does the 700 °C specimen and this is likely a consequence of diffusion-assisted dislocation processes playing a more active role in the 700 °C specimen. A contribution from precipitate/matrix interface cavitation in lowering the fracture stress at the higher temperature is an additional consideration unique to the alloy examined in this investigation. Finally, the deformation process in these materials is complicated by dislocation decomposition and reaction processes, and transitions in slip modes [1,39-43] which are temperature, strain and strain-rate dependent. As the population of <111>, <110> and <001> dislocations was not monitored in this study, we cannot make any arguments related to this aspect on the observed macroscopic deformation and fracture behavior.

The inability to sustain large elongation values at the slowest strain rate at elevated temperatures (for example at 500 °C/10⁻⁵ s⁻¹), is directly attributed to the dominance of creep cavitation which is further accelerated locally by necking. At 600 °C, at the faster strain rate of 10⁻² s⁻¹, the cavitation phenomenon is not as catastrophic and the specimen deforms to large elongation values more uniformly and necking is delayed. Dynamic recovery permits significant plastic deformation prior to failure. At the fastest strain rate, the onset of dynamic recovery is postponed to higher temperatures. Prestrain appears ineffective in substantially improving the tensile elongation at a given temperature. Thus, arguments related to difficulty in nucleating mobile dislocations are likely not valid. In any event, these FeAl alloys demonstrate at least 6–10% elongation in the temperature and strain rate range examined. The effect of test strain rate on dislocation mobility in FeAl alloys has not been studied thus far nor is it known in detail how effective strain rate is in influencing the operating slip system(s). A proposal has been previously made [39] that <111> superdislocation motion is independent of strain rate whereas <001> dislocation motion is strongly strain-rate dependent, although no evidence was presented to support the argument. This proposal is linked to the idea that at elevated temperatures, the dominant deformation mode transitions from the motion of <111> dislocations to <001> dislocations; however as alluded to earlier, whether such a transition occurs or not is still under debate [1,10,39-43].

5. Conclusions

From extensive tensile testing of two multiphase B2 Fe–40Al-based alloys, (i) Fe–40Al–0.6C and (ii) Fe–40Al–0.7C–0.5B, the following conclusions may be drawn.

5.1. Tests in oxygen at room temperature

1. In oxygen at room temperature, the tensile elongation for both alloys is insensitive to test strain rate with the exception of the test conducted at 4 × 10⁻⁷ s⁻¹ for the FeAlC alloy where a decrease in elongation is noted. Furthermore, the tensile elongation values for the FeAlC alloy is higher than those for the FeAlBC alloys in the 4 × 10⁻⁴ to 4 × 10⁻⁶ s⁻¹ regime.

2. The reduction in elongation in the ternary FeAlC alloy at the slowest strain rate of 4 × 10⁻⁷ s⁻¹ is attributed to the high sensitivity of the alloy to trace levels of water vapor, whereas the absence of such a loss in ductility in the FeAlBC alloy in the corresponding situation signifies a decrease in susceptibility to environmental embrittlement.

3. For all tests conducted in oxygen, for both alloys, fracture mode remains principally intergranular failure.
5.2. Tests in air at room temperature

1. The yield strength for each of the two alloys is insensitive to test strain rate over a large strain rate regime \((4 \times 10^{-2} \text{ to } 4 \times 10^{-8} \text{ s}^{-1})\). Furthermore, the yield strength of the FeAlBC alloy is consistently higher (by approximately 50 MPa) than that for the FeAlC alloy for the heat treatment conditions adopted as baseline in this study for each alloy.

2. The tensile elongation for the ternary alloy at the fastest strain rate of \(4 \times 10^{-1} \text{ s}^{-1}\) is lower than that measured in oxygen; this suggests that even at this fast strain rate, environmental effects cannot be completely eliminated. In contrast, the FeAlBC alloy tested at \(4 \times 10^{-2} \text{ s}^{-1}\) exhibits an elongation value comparable to that measured in oxygen.

3. With decrease in test strain rate in the regime \(4 \times 10^{-2} \text{ to } 4 \times 10^{-5} \text{ s}^{-1}\), both alloys exhibit a decrease in elongation, the values being identical for the two alloys.

4. At slower strain rates, the response diverges; whereas the elongation continues to decrease for the FeAlC alloy reaching a minimum at \(4 \times 10^{-7} \text{ s}^{-1}\) before increasing again, the elongation values for the FeAlBC alloy becomes strain rate insensitive in the range \(4 \times 10^{-5} \text{ to } 4 \times 10^{-8} \text{ s}^{-1}\).

5. The fracture stress mirrors the elongation response as a function of strain rate. Interestingly however, the fracture stress values for the FeAlBC alloy are significantly higher than those for the FeAlC (~150 MPa) alloy in the strain rate regime \((4 \times 10^{-2} \text{ to } 4 \times 10^{-5} \text{ s}^{-1})\) where their elongations are almost identical. This implies a higher work hardening rate in the FeAlBC alloy.

6. Fracture modes in the two alloys vary as a function of strain rate. The percent transgranular cleavage increases steadily with decreasing strain rate in both alloys in the regime \(4 \times 10^{-1} \text{ to } 4 \times 10^{-6} \text{ s}^{-1}\), coincident with the elongation decrease. At slower rates, the percent transgranular cleavage remains constant for the FeAlBC alloy (as does elongation) whereas it increase sharply for the ternary alloy to a maximum at \(4 \times 10^{-7} \text{ s}^{-1}\) before dropping down rapidly at slower strain rates. This latter response is consistent with the elongation variation with strain rate in the slow strain rate regime.

5.3. Tests in air as a function of temperature and strain rate

1. The BDTT, defined by the ‘upward swing’ of the elongation versus temperature curve is a strong function of test strain rate. Prestrain of 0.5% provided at room temperature does not influence the warm temperature (300–500 °C) tensile elongation at the test strain rate of \(10^{-2} \text{ s}^{-1}\) and thus, does not shift the BDTT to lower temperatures.

2. Yield stress, work hardening rate, onset of necking and fracture stress are all functions of test strain rate and temperature. Likewise, a rich variety of fracture modes can occur depending on test strain rate and temperature, ranging from intergranular failure through creep cavitation and ductile tearing to almost complete transgranular cleavage. All these observations must be reconciled against a backdrop of the variation of yield stress with temperature for B2 FeAl alloys, which includes a positive temperature dependence of strength, a peak in strength at intermediate temperatures, and a rapid drop off in strength thereafter due to the onset of creep mechanisms. The temperature of occurrence and the magnitude of the strength peak are thus strongly influenced by the test strain rate.

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References

[38] Li X, Baker I. Scripta Mater 1997;36:1387.