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Ductility improvement in iron aluminides

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Excellent high temperature properties of intermetallic aluminides recommend their use for structural applications in sulphurous atmospheres. Interest was not sustained in them because of their brittleness at ambient temperatures. Fe_3Al based alloys (air induction melted) were taken up to study the effect of deviations from stoichiometry (both sub and super), third and fourth alloy additions, B, Ti (micro as well as macro), on physical and mechanical properties (at ambient temperatures). The columnar grains observed in sub and stoichiometric compositions were found to become equiaxed on additions of alloy. The microstructures became finer on hot forging and rolling. The hot workability of these alloys increased from 65 to 85% at 973 K on B, Ti additions. The ultimate tensile strength (UTS) and per cent, elongation E increased to 80 kg mm^{-2}, 3.0% and 94 kg mm^{-2}, 5%, respectively, for sub and stoichiometric alloys on B and Ti additions. The superstoichiometric alloys displayed dendritic structure and could not be hot worked due to cracking during forging, even after additions of alloys. The stoichiometric Fe_3Al alloy with B and Ti additions exhibited the best properties under the experimental conditions.

1. Introduction

The need to replace strategic metals, such as Cu and Cr, and the need for energy efficiency requiring lighter and stronger parts have led to a revival of interest in the development of ordered alloys for structural applications. Ordered intermetallics have long range ordered crystal structures below the critical ordering temperature, T_c, which give rise to stronger binding and closer packing between atoms. Restricted atom mobility generally leads to slower diffusion dependent processes, giving better creep and fatigue resistance in ordered lattices at high temperatures. Ordered aluminides, such as those of Fe, Ni and Ti, are generally resistant to oxidation and corrosion because they have sufficient aluminium (minimum 15 at %) to form in oxidizing and sulphidizing environments, compact, adherent and thin oxide surface films that protect the base metal from excessive attack [1–6]. Further, these aluminides show the unusual property of increasing yield strength with increase in temperature [7, 8]. Additionally, they display low density, relatively high melting points, good strength – weight ratio, high specific modulus and are inexpensive.

Ordered intermetallics have good hot fabricability, but pose severe embrittlement at room temperature making fabrication difficult. Low fracture toughness affects their use in structural applications. The low ductility in ordered intermetallics, in general, is due to one or more of the following reasons

1. Resistance to the movement of dislocations by an insufficient number of slip systems in polycrystals, as grain boundaries prevent spreading of homogenous deformation.
2. Restricted cross-slip: cross slip of superlattice dislocations is generally very difficult, since work must be done against the anti-phase boundary (APB) tension [9]. The difficulty in cross slip either limits or eliminates completely the nucleation and growth of voids which lead to brittle fracture.
3. Lack of effective dislocation source and difficulty of dislocation multiplication.
4. Grain boundary weakness causing their decohesion before enough number of slip systems can be activated, since some grain boundaries may be less stable than others due to wrong neighbour consideration [6].
5. Defect and impurity segregation at dislocations and grain boundaries [10, 11].
6. Inherent structure of the grain boundary [12].

B2 aluminides, like NiAl, CoAl and FeAl, are promising for applications at temperatures above 1273 K because of their strength and oxidation resistance, but have not yet been ductilized satisfactorily [13–15]. Compounds such as Fe_3Al and Fe_3Si are potentially attractive because of their high strength at elevated temperatures; the temperature at which the strength starts falling depends upon the third alloying element content [16]. Fe_3Al occurs over the composition range 25–30 at % Al. It exists in the ordered DO_3 structure up to 813 K, in the B2 structure between 813 and 1033 K, and in the disordered structure above 1033 K. As the Al content increases beyond 25 at %, the $DO_3 \rightarrow B2$ transition temperature decreases and the B2 ordering temperature increases.

In Fe–Al alloys (0–44 at % Al), the maximum in yield stress is observed near the DO_3 critical temperature and a maximum in isothermal yield stress is observed near the Fe_3Al composition [17]. It supposedly occurs due to a change from single to double dislocations on long range ordering. A sharply

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reduced ductility is reported at 477 K as the Al content approaches 25 at % [2]. Alloys made conventionally by the ingot route, containing 25–50 at % Al, have usually been reported to be brittle at room temperature.

For stoichiometric Fe_3Al, both single crystals and polycrystals show high yield stress, low work hardening and wavy slip in compression tests [18]. Transmission electron microscope (TEM) studies revealed that ordinary dislocations with APB, rather than super dislocations, control the flow behaviour. The reason for this is the rather low APB energies for Fe_3Al alloys [19].

The predominant slip system in Fe_3Al is $\{1\,1\,0\}$ $\langle 1\,1\,1 \rangle$, which provides more than five independent slip systems required for extensive polycrystalline ductility. From these characteristics one should also expect good tensile ductility for Fe_3Al alloys. The reported extreme brittleness in these alloys may be due to any of the following reasons

1. Weak grain boundaries leading to intergranular fractures or because of the disorder associated with the grain boundaries [12].
2. Detrimental grain boundary segregation [20].
3. Environmental effects: McKamey and coworkers [21–23] have established that environmental embrittlement is the major cause of low ductility. Water vapour reacts with Al at the crack tip to form high fugacity atomic hydrogen, which drives into the metal causing embrittlement. Hydrogen is shown [24] to enhance dislocation mobility at crack tips and reduce grain boundary cohesive strength. Iron aluminides exhibit [25] a peak in the susceptibility to hydrogen embrittlement curve at ambient temperature, which diminishes to negligible levels at temperatures above or below ambient [26]. A three-fold increase in per cent E has been reported in Fe_3Al (28 at % Al) in vacuum or oxygen atmosphere [24].

Improvement in ductility of intermetallics has been attempted through

1. Increasing the number of easy slip systems by alloying with solid solution elements.
2. Modification of crystalline structure through macro alloying (several per cent) and process, yielding a more ductile structure [6]. Fe_3Al (DO_3) FeAl (B2) have body centred cubic (b.c.c.) related structures, and are inherently less desirable than face centred cubic (f.c.c.) related structures like LI_2. The LI_2 type ordered structure is stabilized through control of electron concentration (e/a, the average number of electrons per atom outside the inert gas shell) and alloy composition resulting in excellent ductility and fabricability [27, 28].
3. Strengthening of grain boundaries by micro alloying (p.p.m. range) to control grain boundary composition and strength: dopants could be either reactive elements that bind harmful impurities like S in innocuous forms through precipitation, or elements like boron that act as electron donors and thereby strengthen atomic bonding and increase the cohesive strength of grain boundaries [29]. Boron has a strong tendency to segregate to grain boundaries, but not to free surfaces [29], thus enhancing grain boundary cohesion and suppressing intergranular fracture [6].
4. Grain size refinement: the tendency for brittle fracture of both transcrystalline and intercrystalline types in highly alloyed ferritic alloys is dependant on grain size. Therefore, control of grain size throughout casting, hot working and heat treatment phases is essential [30]. NiAl exhibits a critical grain size (20 μm), below which polycrystalline aggregates are ductile in tension. This critical grain size is expected to decrease with decreasing temperature, increasing strain rate and with deviations from stoichiometry [31]. Ductilization is possible by refining grain size by rapid solidification [3, 5, 13]. It also minimizes macrosegregation and grain boundary segregation and causes a reduction in the homogenization time. Rapid solidification is known to cause a reduction in the degree of order as an interim step during processing.
5. Innovative processing techniques.
6. Precise control of the defect and impurity content [6].
7. Formation of thin surface films which reduce the flow stress, e.g. in NiAl, because the interface between substrate and layer acts as a source of mobile dislocations [32].
8. Embedding the brittle phase into a more ductile phase in order to obtain a favourable combination of strength and toughness. Such composite structure may be produced by directional solidification, e.g. in superalloys [33].
9. Single crystal approach: directional solidification leads to production of components formed from single crystals or aligned crystals. The elongated grain morphology developed on directional solidification may impose resistance to environmental effects because the relatively small number of grain boundaries intersecting the surfaces decreases access of the atmosphere to the interior of the materials [34].
10. Control of environment, particularly precluding exposure to moisture.

In FeAl alloys, containing more than 15 at % Al, the protection is by the production and the growth of a continuous layer of Al_2O_3 during oxidation. Al_2O_3 scales have a tendency to crack and spall, and it is therefore necessary to add small amounts of other elements, like Ti, Zr, Nb, V, to improve scale adherence. A reactive solute, like Ti, and rare earths suppress intergranular cracking, increase the $DO_3 \rightarrow B2$ transition temperature and also improve solid solution strengthening [3, 35]. Boron improves the elevated temperature strength and the ductility by increasing grain boundary cohesion, especially in Ni_3Al [29, 36–38]. TiB_2 dispersion in Fe_3Al reduces and stabilizes the grain size (1–2 μm) of atomized compacted powders and increases ductility of recrystallized material [3, 39]. Cr improves ductility by enhancement of cleavage strength [21, 22]. Mo improves creep properties by m odifying the transition temperature for $DO_3 \rightarrow B2$ transition [35, 40, 41]. Nb [35, 42], Ta, Zr [35], Hf [35, 43] substantially improve tensile strength at room and elevated temperatures because of precipitation hardening. Mn, Ni, Si additions also raise T_c [44].

Many studies have been undertaken for material development, which are targeted mainly at practical applications. The results are reported only partially in the open literature. Keeping in mind the fact that the alloy approach is more economical than RSP (which may involve costly processing techniques), conventional castings with alloy additions is undertaken in the present study. It is planned to study the effect on structural and mechanical properties (at ambient temperature) of

1. Fe_3Al alloys, deviation from stoichiometry (sub and super);
2. third alloy addition (B); and
3. fourth alloy addition (B and Ti), singly and/or jointly in micro as well as macro compositions.

The main objective of the authors' work is to improve the ambient temperature ductility and workability of these alloys.

2. Experimental procedure

2.1. Casting and mechanical working

The alloys (4 kg heat) were melted in a basic lined air induction furnace and cast in metal moulds. These alloys are categorized, Table I, as stoichiometric S (26–28 at % Al), substoichiometric SB (21–23 at % Al) and superstoichiometric SP (33–35 at % Al). The alloys were made without any addition (WA), with micro addition of B and Ti (μA), macroaddition of B and Ti (MA) and only boron addition (BA). The ingots were homogenized at 1373 K for 31 h and furnace cooled. The ingots were forged and rolled at 1173 K (multi pass with intermittent soaking) to a final thickness of less than 1.0 mm.

2.2. Structural characterization and mechanical testing

Optical metallography, microhardness, X-ray diffractograms (XRD) of homogenized, forged, rolled samples were taken. Flat subsize tensile specimens of length 100 mm were prepared from the rolled sheets and tested on an Instron tensometer for UTS and per cent *E*. The fractured surfaces were examined by scanning electron microscope (SEM).

3. Results

Optical microstructures of homogenized samples of SB–WA show mostly columnar grains, precipitates and macrocracks Fig. 1a. Microaddition of B and Ti produces some coarse equiaxed grains in these alloys, Fig. 1b. Alloys S–WA also display columnar grains; microadditions of B and Ti give rise to coarse equiaxed grains, Fig. 1c; and macroadditions of B and Ti reveal fine equiaxed grains, some dendritic structure and plenty of fine precipitate (supposedly of TiB_2) on grain boundaries, Fig. 1d. Alloy SP–BA and SP–MA show generally dendritic structure, sub grain boundaries and inclusions inside some grains, Fig. 1d, e, respectively. SP–MA shows finer grains compared to SP–BA. The forged samples show uniform equiaxed grains in SB and S series of alloys and fine dendritic structure continues to exist in SP alloys. Grain growth and equiaxed grains were noticed in forged and rolled samples in SB–WA and SB–μA alloys, Fig. 2a, b, respectively. Alloy S–μA in forged and rolled conditions shows very fine grains without any grain growth, Fig. 2c. The finest grains once again were observed in S–MA along with elongated precipitates, Fig. 2d.

XRD studies establish the presence of B2 order in general in homogenized and rolled samples and DO_3 order in forged samples, Fig. 3. The average microhardness of these alloys corresponds to that of Fe_3Al (350 VHN), being less in homogenized samples and increasing in forged and rolled samples.

The hot workability (at 973 K) of SB and S alloys is found to be excellent. The SP alloys could not be hot worked due to cracking during forging itself. Table II lists the tensile test data of SB and S alloys. It shows that the highest values of UTS and $\%E$ are obtained in S–μA as well as S–MA alloys. The SEM microstructure studies of fractured samples revealed brittle fracture in general, e.g. Fig. 4a, b for SB–WA and SB–μA alloys. Some dimples in limited areas were noticed in samples possessing somewhat higher ductility, Fig. 4c for S–μA alloy.

TABLE I Chemical composition and nomenclature of alloys

Alloy	Composition (wt %)				(at %)
	Al	B	Ti	Fe	Al
SB–WA	11.5	–	–	Balance	21.2
SB–μA	12.9	0.005	0.32	Balance	23.4
S–WA	16.1	–	–	Balance	28.4
S–μA	15.1	0.004	0.24	Balance	26.9
S–MA	15.5	0.630	1.10	Balance	26.9
SP–BA	20.5	0.220	–	Balance	34.5
SP–MA	20.1	0.700	1.50	Balance	33.4

4. Discussion

There are two main factors which are at play in this alloy development work: stoichiometry and alloy additions. Fe_3Al passes through two ordered structures, DO_3 and B2, before becoming disordered above 1033 K. The deviations from stoichiometry are accommodated either by the incorporation of vacancies in the lattice or by location of antisite atoms in one or the other of the sublattices. Many of the aluminides exist over a range of composition, but the degree of order decreases as the deviation from stoichiometry increases. Fe_3Al occurs over the range 25–30 at % Al. As the concentration of Al increases above 25%, the $DO_3 \rightarrow B2$ transition temperature decreases and the B2 ordering temperature increases. Additional atoms may also be incorporated in the structure without losing the ordered structure. Therefore, in many cases, the so-called intermetallic compounds may be used as the basis for alloy development to improve or optimize properties for specific applications.

It has been established earlier [2] that up to 20 at % Al alloys fracture in a ductile manner by void nuclea-

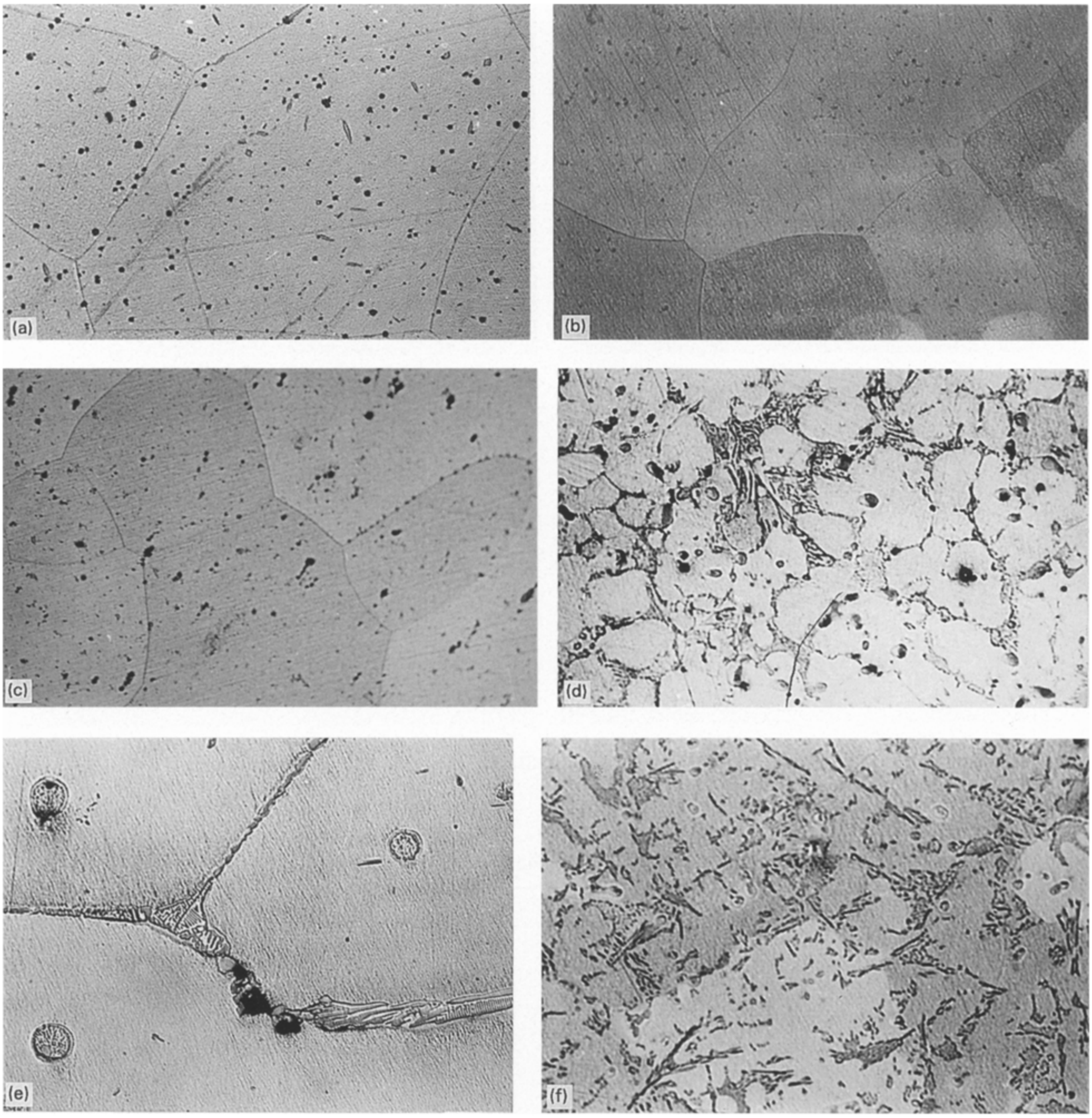


Figure 1 Optical microstructures of cast and homogenized alloys ($\times 150$): (a) SB–WA, (b) SB–μA, (c) S–μA, (d) S–MA, (e) SP–BA, and (f) SP–MA. (83% reduction)

tion and coalescence. Alloys containing 20–25 at % Al fail in a brittle manner by transgranular mode. Higher Al alloys fracture in intergranular mode. Changes in stoichiometry apart from influencing the yield strength also influence the dislocation substructure and fracture behaviour, e.g. plastic deformation is supposedly caused by unit dislocation in hypo-stoichiometric Fe_3Al and by paired dislocations in $Al > 26$ at % alloys [17]. Also, hypostoichiometric Fe_3Al are found to be more ductile than hyper-stoichiometric compositions. In the authors' work, SB alloys are also found to be better than SP alloys as regards ductility. The subject of nature and effects of lattice defects as a function of stoichiometry needs further exploration.

At room temperature, workability and ductility of iron aluminides is poor, which could be due to the presence of B2 ordering. It is therefore surmised that conversion of $B2 \rightarrow DO_3$ at room temperature may improve the ambient temperature workability and ductility. During heating, conversion of $DO_3 \rightarrow B_2$ is easy, but during cooling the reverse transformation is not easy. Prolonged holdings of tensile samples at 773 K for 100 h, followed by slow cooling to produce DO_3 order is therefore recommended to increase the amount of DO_3 order in the alloys. Since that procedure was not followed in the present work, one is likely to be working with whatever little DO_3 may have formed during cooling after homogenizing. The DO_3 order was not detected by XRD in as-cast and homogenized samples, presumably due to the coarse grain size. The superlattice lines manifest themselves in forged samples which have finer grains. There may also be an increase in the degree of order on forging due to enhanced diffusion. The larger deleterious effect of the

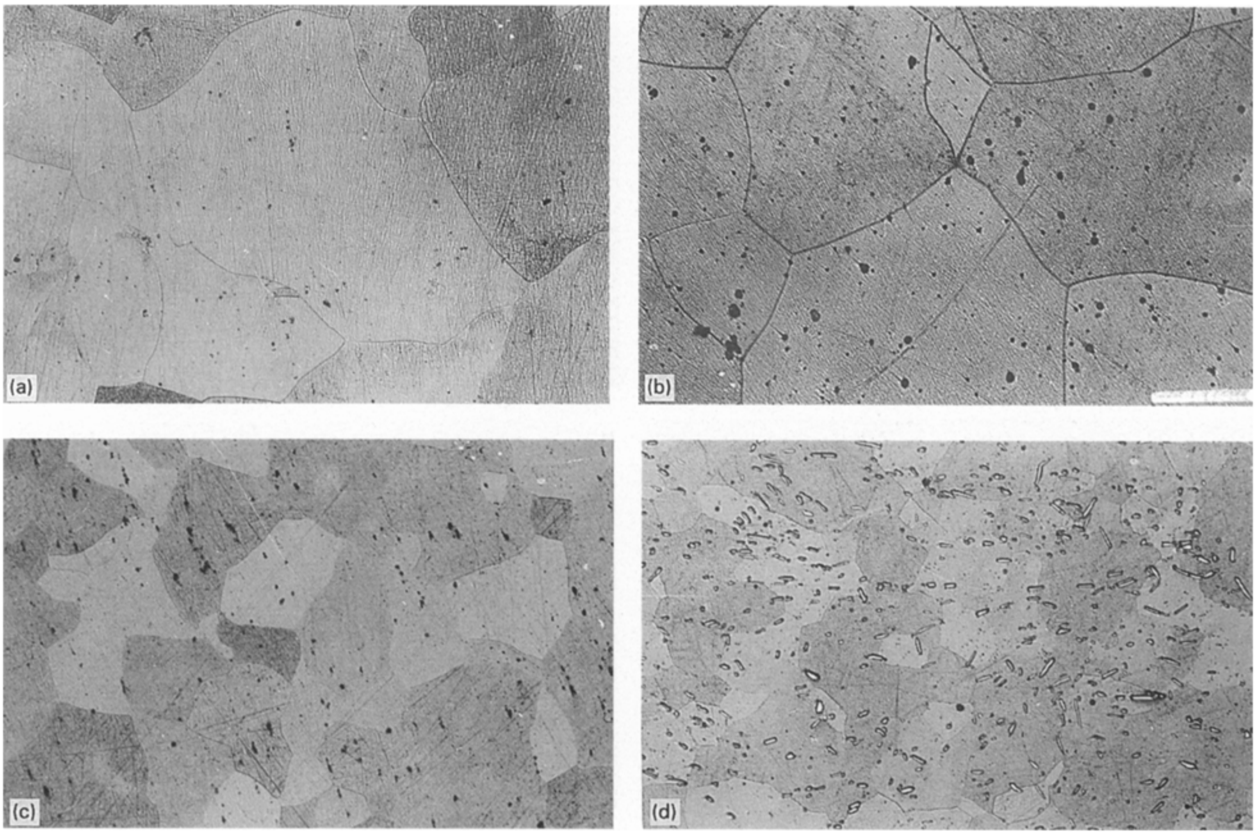


Figure 2 Optical microstructures of forged and rolled alloys (× 150): (a) SB–WA rolled, (b) SB–μA rolled, (c) S–μA rolled and (d) S–MA rolled. (83% reduction)

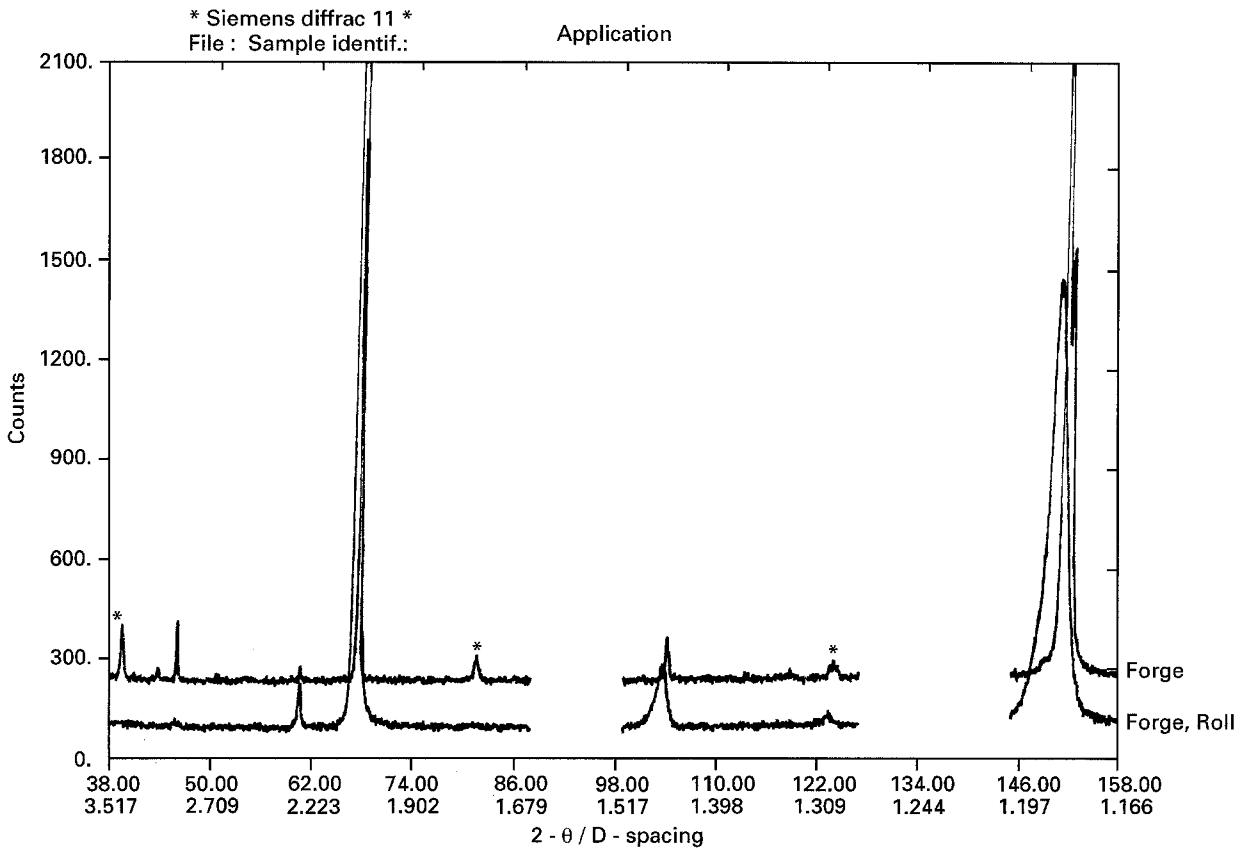


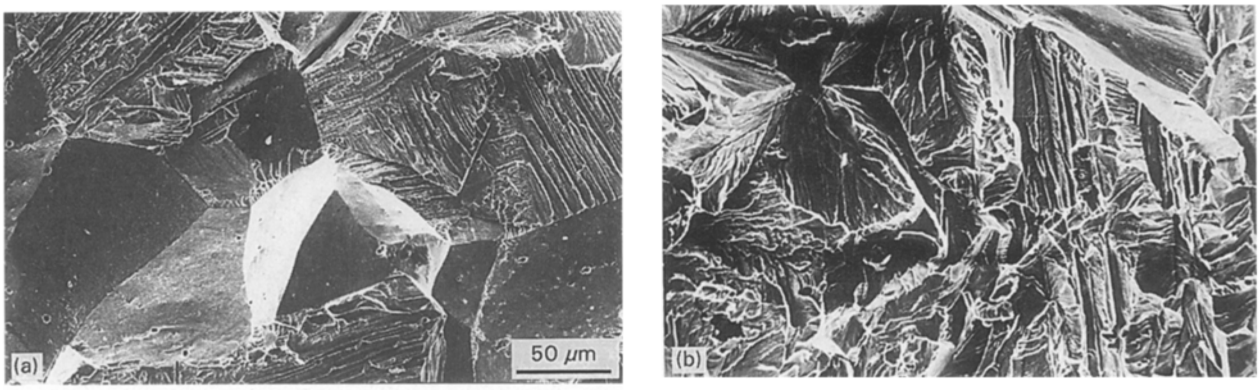
Figure 3 X-ray diffractogram of S–μA alloy in forged, and forged and rolled conditions. Forged alloy shows DO_3 order peaks (marked*).

environment may have masked the improvement obtained in ambient temperature ductility due to alloying additions.

Having five independent slip systems is necessary, but not sufficient, for ductility. All slip systems should be operative simultaneously for dislocations to pass

TABLE II Mechanical properties of alloys

Alloy	% Deformation (Forge)	% Deformation (Forge + roll)	UTS ($kg\,mm^{-2}$)	$\%E$	Remarks
SB–WA	83	98	57	2.5	Brittle fracture very coarse grains
SB–μA	78	98	80	3.0	Brittle fracture Finer grains
S–μA	85	97	94	5.0	Limited dimples
S–MA					Finer grains
SP	Cracked	–	–	–	Could not roll



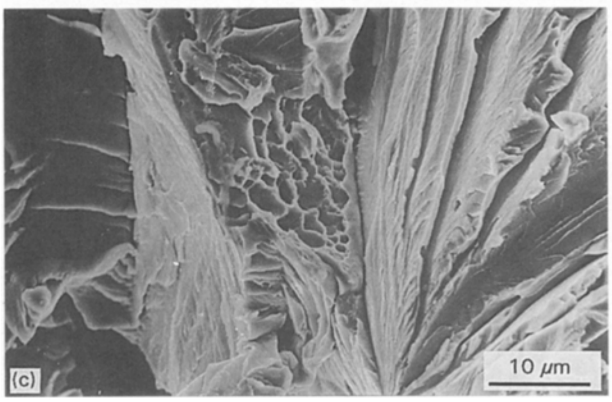


Figure 4 SEM photomicrographs of fractured tensile samples: (a) SB–WA, (b) SB–μA, and (c) S–μA.

through; otherwise dislocations move in a grain and then pile on the grain boundary, building up pressure on both sides.

Boron in steels or in FeAl alloys reacts with the available nitrogen to form boron nitride and renders B unavailable for any grain refining work. Titanium is added primarily to take care of nitrogen, by forming titanium nitride and leave B free for grain refining work. B and Ti together are seen to refine grain size of Fe-Al alloys even in microadditions. However, when added as macroadditions, they give rise to fine precipitates (as seen by SEM) which restrict grain growth and produce finer equiaxed grains in as-cast as well as forged samples. It is also seen that on B and Ti addition (micro as well as macro), the hot workability improves, there is a greater increase in UTS and less improvement in $\%E$ in SB and S alloys. The stoichiometric alloys display better mechanical properties than off-stoichiometric alloys. It is known that B addition strengthens both the matrix as well as the grain boundaries of Fe alloys, thereby neutralizing some of the advantages obtained by grain boundary strengthening.

A small B addition reportedly produces a dramatic improvement in ductility in NiAl alloys. B was found to be more effective in Ni alloys containing 24 at % Al and less effective at higher Al concentration and B segregation at grain boundaries decreasing with increasing Al concentration [29]. It is therefore considered that B may become less effective in improving the ductility of Ni_3Al alloys when less than a critical amount is present at grain boundaries. Such a beneficial effect is not evident in Fe_3Al alloys, which may possibly be due to not striking the optimum amount of B *vis a vis* Al concentration. The sensitivity of the fracture transition to slight changes of composition are well established.

Iron aluminides can be used for high temperature structural components in advanced coal conversion systems, e.g. in thermal power plants to replace ferritic (9 Cr, 1 Mo) steels. The yield strength increases up to 813 K, T_c, in Fe_3Al alloys and then decreases drastically due to the onset of B2 ordering. For a thermal power plant, 813–873 K is a very critical temperature region and a raise in T_c of aluminides makes their components useful up to a higher temperature range. Iron aluminides could also be used in molten salt containment and automobile components subjected to high temperature. All said and done, it can be stated that the brittleness of a material does not preclude its use as a structural material. A limited toughness can be imparted by appropriate design of components.

5. Conclusions

1. Air melting and additions of B and Ti (micro and macro levels) in stoichiometric and substoichiometric alloys changes the microstructure from columnar to

equiaxed; the finest grains are obtained in the case of S–MA.

2. Super stoichiometric alloys, SP–BA and SP–MA, show dendritic structure.

3. Modification of S and SB alloys by B and Ti makes them more amenable to hot working, with 98% reduction leading to a 0.6 mm rolled sheet compared to 65% hot workability in unmodified alloys.

4. In SB alloys, on micro addition, the ductility improves to 3.0% and UTS to 80 kg mm^{-2}, as compared to 2.5%E and 57 kg mm^{-2} in SB–WA.

5. The ambient temperature ductility improves from less than 1 to 5% E and UTS increases to 94 kg mm^{-2} on B and Ti addition (micro and macro levels) in stoichiometric alloys.

6. Super stoichiometric alloys (with additions) are not amenable to hot working.

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