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Mechanical alloying of Fe–Al intermetallics in the DO_3 composition range

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Binary Fe–Al intermetallics with compositions ranging between 10 and 30 at% have been produced by mechanical alloying. The elemental powders were milled together resulting in the dissolution of the Al atoms into the Fe lattice. Subsequent heat treatment of the compacted powder resulted in the formation of the intermetallic. However, complete suppression of the DO_3 (Fe_3Al) structure in favour of the B2 (FeAl) structure was observed. The suppression of the DO_3 structure is considered to be due to the presence of the high density of defects resulting from the heavy deformation incurred during milling. At Al compositions below 22 at%, X-ray diffraction revealed a b c c phase with lattice parameters varying between those of α-Fe and the B2 intermetallic. The structure tended towards that of α-Fe with lower Al contents indicating a decreasing number of Al atoms available to occupy B2 lattice sites. A fine grain size and evidence of tearing indicate that mechanically alloyed Fe–Al intermetallics in the DO_3 composition range are ductile at room temperature.

1. Introduction

The attractive mechanical properties of the ordered intermetallics combined with the potential for excellent corrosion and oxidation resistance have led to increased research in recent years [1]. One of the major problems with these materials is that of poor ductility, particularly at room temperature [2, 3].

Of particular interest among the intermetallics are the iron and nickel aluminides, which attracted more attention when it was discovered that rapid solidification techniques can result in improvements in room-temperature ductility [4]. The brittle nature of intermetallics at room temperature is attributed to poor dislocation motion in highly ordered lattices. Rapid solidification is considered to result in the introduction of disorder due to the quenching in of vacancies, improved homogeneity, the production of a very fine grain size and grain-boundary modification, all of which can result in improved ductility [4, 5].

An increase in intermetallic research followed the well-documented discovery by Aoki and Izumi in 1979 that additions of small quantities of boron dramatically improved the ductility of polycrystalline Ni_3Al [6, 7]. More recently, improvements in properties of intermetallics have resulted from investigations into the use of ternary additions combined with the use of modified processing techniques [5, 8, 9].

Since ductility is found to be sensitive to processing, much work is now being conducted on the effects of alternative processing routes, such as mechanical alloying, a technique originally used by Benjamin in the late 1960s to produce oxide dispersion-strengthened alloys [10] but now also employed to produce intermetallics and non-equilibrium phases (e.g. [11, 12]).

An advantage of mechanical alloying over many other techniques is that it is a solid-state technique and consequently problems associated with melting and solidification (such as segregation or large differences in melting point) are by-passed. Since mechanical alloying results in a very fine mix between the initial constituents, heating of the resulting powder enables the formation of fine grained and homogeneous compounds, similar to those produced by rapid solidification techniques, through a process of enhanced solid-state diffusion [13, 14].

The binary intermetallics Fe_3Al and FeAl are of particular interest because, unlike the majority of intermetallics, they are stable over a range of compositions. At equilibrium, Fe_3Al occurs as a DO_3 structure between 18.5 and 36.5 at % Al at room temperature, while FeAl has the B2 structure and exists between 36.5 and 50 at % Al (Fig. 1). Although the ductility of the binary DO_3 structure is greater than that of the B2 structure, it is only stable up to 540 °C. Above 540 °C, the B2 structure is stable. Increases in the transition temperature have been obtained by making ternary additions [15]. However, an alternative approach has been to suppress the formation of the DO_3 structure in favour of an imperfect B2 structure by employing techniques such as rapid solidification. Although the B2 structure is relatively brittle at compositions near 50 at % Al, the ductility increases with a decrease in the aluminium content. [5].

2. Objective

The objective of this work was to produce binary Fe–Al intermetallics of compositions between 10 and 30 at % Al with the aim of investigating the disordering effects of mechanical alloying on the DO_3

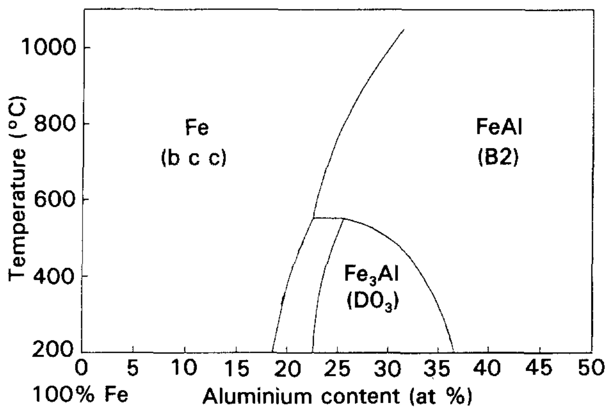

Figure 1 The iron–aluminium phase diagram [20].

structure. As with rapid solidification it is thought that mechanical alloying may enhance ductility by introducing defects (thus suppressing the formation of the $D0_3$ phase), decreasing grain size and improving homogeneity.

3. Experimental procedure

Mechanical alloying of elemental iron and aluminium powders in the required ratios were conducted using a Pilamec vibratory mill. The initial mean powder size of both elements was 100 μm. Milling was undertaken at room temperature using hardened steel balls, the powder to balls ratio being 1 : 10 by weight. In order to minimize oxidation, milling was conducted in a dried argon atmosphere and 1 wt % octane was added to the system to prevent excessive welding in the chamber. This allowed a high degree of mixing. After 48 h, the octane was removed and milling was continued for a further 48 h.

Analysis was conducted on both the as-milled powders (i.e. the final milled product) and on the as-milled powders compacted, sealed in argon, heated at 1150 °C for 4 h and air cooled. The powders were compacted at room temperature using a 3 mm diameter die and a 6 kN load. The techniques employed for analysis were optical and scanning electron microscopy together with energy dispersive X-ray analysis (EDX) and X-ray diffraction (XRD). Microhardness testing was also employed to assess mechanical properties.

4. Results

Fig. 2 shows XRD traces taken from specimens containing different levels of aluminium after milling and before heat treatment. From the figure it can be seen that iron peaks were present and that aluminium peaks were no longer detected regardless of composition. As shown in Fig. 3 subsequent heat treatment revealed the presence of the B2 FeAl structure at compositions above approximately 22 at % Al. Below this composition, X-ray diffraction of the milled and heat-treated material revealed peaks lying between the α-Fe and the B2 (FeAl) peak positions. The lower the aluminium content, the closer the peaks were to the α-Fe peak positions. The unidentified peaks at compositions above 17 at % Al are thought to be as a result of contamination which occurred during heat treatment.

In contrast to the XRD traces in Fig. 2, EDX analysis revealed that aluminium was present in the expected proportions and that the general composition of the overall material remained constant throughout milling and heat treatment.

Fig. 4 shows the effect that mechanical alloying has on the hardness of the heat-treated material over the range of compositions investigated. It is clear from the figure that the hardness increases with aluminium content and that the relationship between the hardness and composition is approximately linear. Also shown on the plot are the hardness values of melt-spun Fe–Al (in the as-spun condition) at these compositions [5].

SEM of the surfaces of a fractured compact showed areas in which ductile tearing took place in a very poorly compacted specimen (Fig. 5a). More detailed examination of pores on the fracture surface showed the very fine grain size resulting from mechanical alloying (Fig. 5b).

5. Discussion

Since only the structure of iron was detected by X-ray diffraction, it is assumed that the aluminium became incorporated into the lattice without significant distortion by the coupling of aluminium atoms with defects, forming a disordered solid solution. Similar behaviour has been observed in other mechanically alloyed intermetallic materials (e.g. Ni_3Al and Ti_3Al) and has also been attributed to such a mechanism of dissolution [16, 17].

The formation of the B2 (FeAl) phase following heat treatment (Fig. 3) clearly shows the effect that mechanical alloying has. The $D0_3$ structure (see Fig. 1) is completely suppressed due to the presence of a high quantity of defects resulting from the extensive deformation and the coupling of the aluminium atoms with the defects. This is very similar to the effect that rapid solidification has on the $D0_3$ structure at these compositions where the defects are quenched in vacancies rather than dislocations produced by mechanical damage [18].

At compositions below 22 at % Al, a gradual change occurred in the heat-treated material from the B2 structure to the α-Fe structure with decreasing content (Fig. 3). This is very interesting, because in the as-milled condition, XRD revealed peaks coinciding exactly with the peak positions for α-Fe for all compositions tested (Fig. 2). Consideration of the peak positions reveals that following heat treatment, the 2θ values decreased, denoting an increase in the lattice spacing. In order to maintain stability it is clear that such an increase in lattice spacing must coincide with a corresponding increase in the degree of order. Evidence for this is seen in the reduction in peak widths following heat treatment.

The mechanism for such a change may be attributed to a transfer of the aluminium atoms from unstable interstitial sites in the iron lattice to a more stable (and

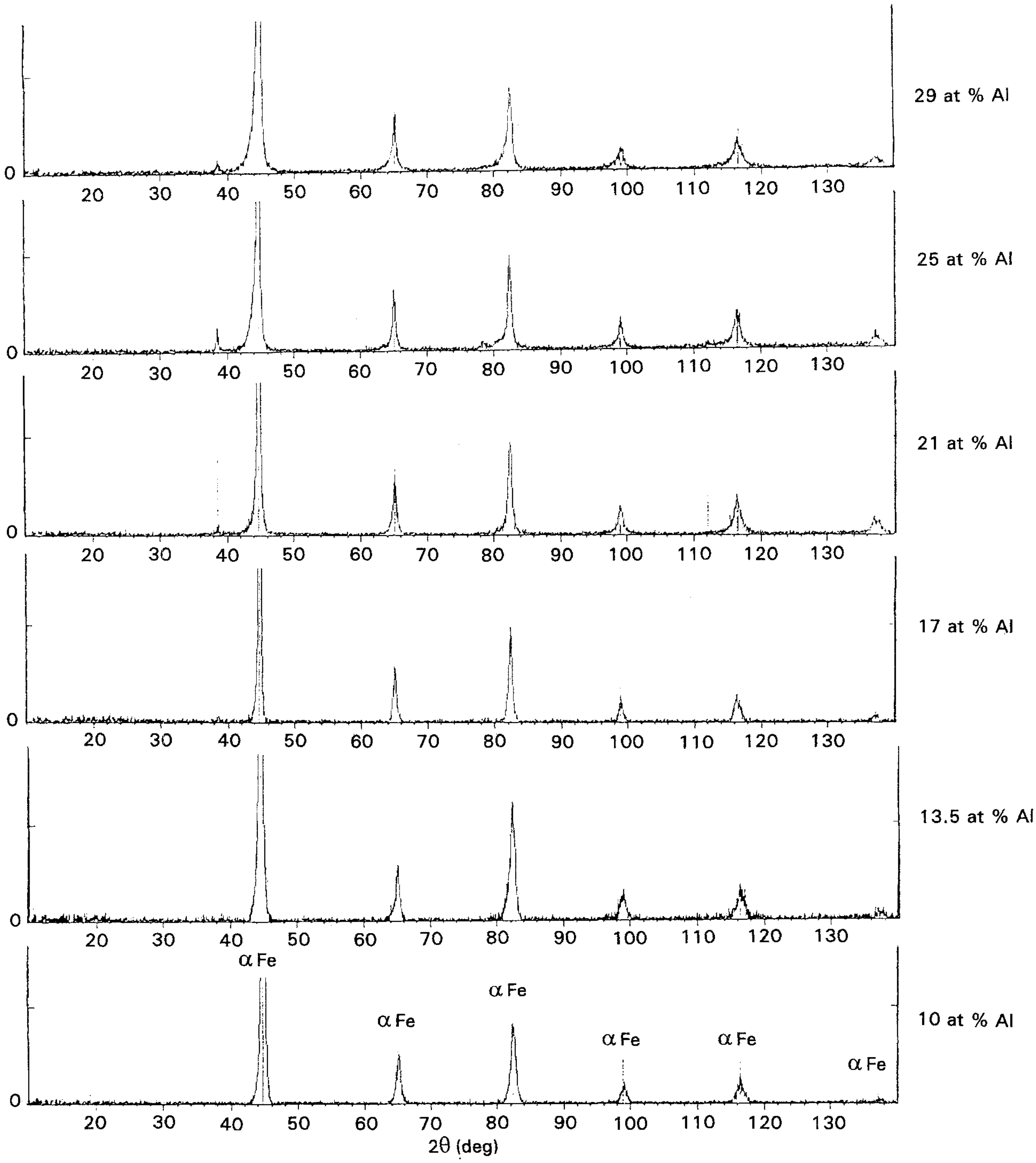

Figure 2 X-ray diffraction traces taken from specimens containing different levels of aluminium after milling.

ordered) B2 structure in which they occupy specific iron lattice sites. This would explain the absence of both the aluminium and the FeAl diffraction peaks prior to heat treatment. If a substitutional solid solution had existed prior to heat treatment then any rearrangement following heat treatment would clearly result in little change in the lattice spacing.

The gradual change from the α-Fe structure to the B2 structure with increasing aluminium content is of interest because there appears to be no definite phase boundary. Such a change is assumed to be an ordering effect because order, and therefore the B2 structure, would become more and more difficult to maintain with the decreasing number of aluminium atoms available to occupy iron lattice sites.

One problem of mechanical alloying is the potential for excessive oxidation or elemental loss. However, this appears to have been overcome in this work as shown by the XRD traces in Fig. 2, although it is inevitable that pre-existing oxide films are folded into the alloyed structure.

As shown in Fig. 4, the increase in hardness with increasing aluminium content of the mechanically alloyed materials is very similar to that observed in the melt-spun material. The almost identical slope of the curves indicates that the same properties (i.e. the increasing order) govern the hardness increase for both conditions. This is a consequence of the greater ability of ordered structures to retard dislocation motion. The greater hardness of the mechanically

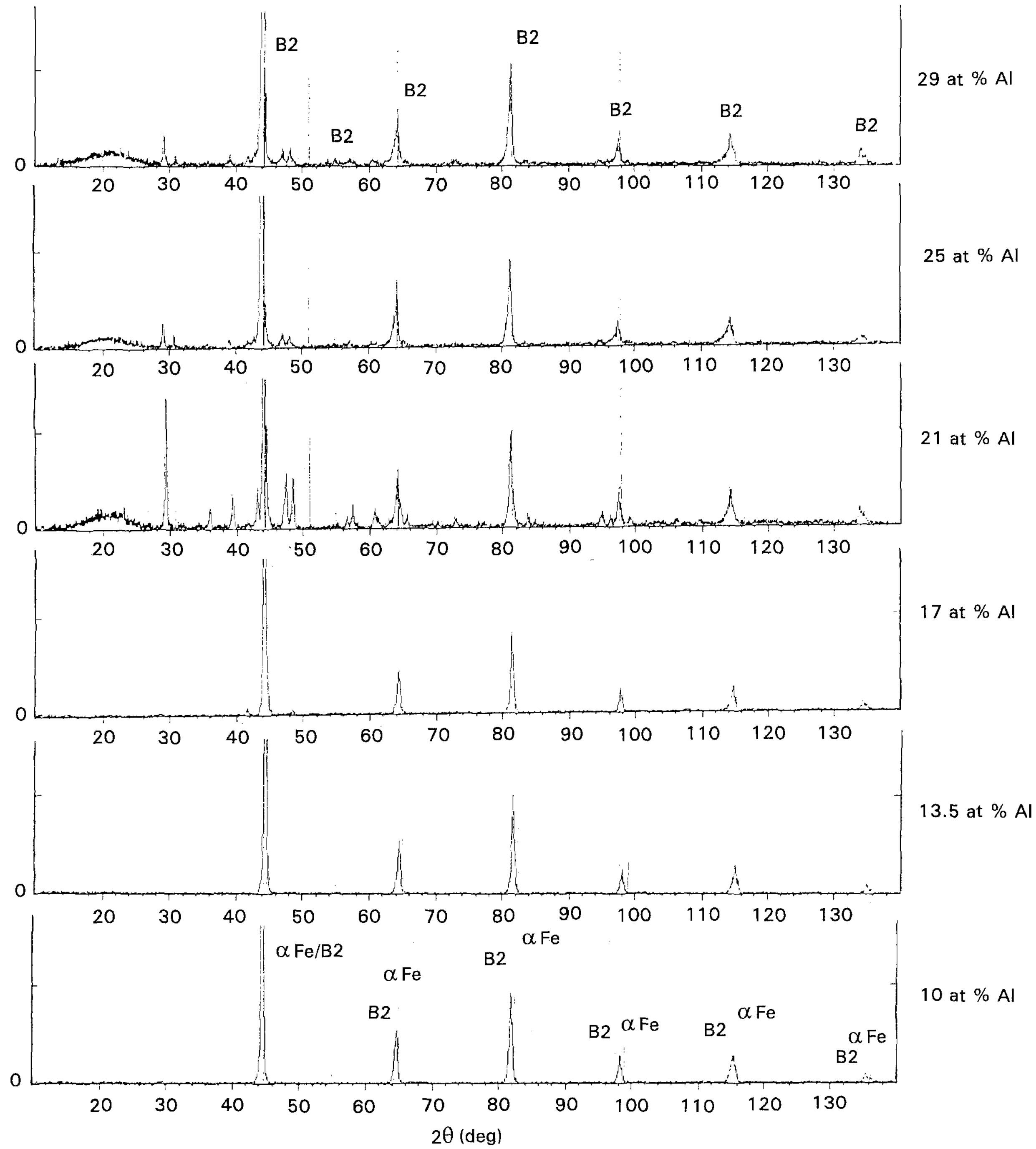

Figure 3 X-ray diffraction traces taken from specimens containing different levels of aluminium after heat treatment.

alloyed intermetallic is clearly a result of the heavy deformation and work hardening experienced during milling coupled with the dispersion content.

The fine grain size resulting from mechanical alloying (Fig. 5b) is particularly important, because a fine grain size in Fe–Al intermetallics has been found to produce a significant improvement in mechanical properties [19]. Although further mechanical evaluation of HIPed compacts (in which all porosity has been removed) is required, the fine grain size and the tearing on the fracture surface shown in Fig. 5 indicates that mechanically alloyed Fe–Al intermetallics in the $D0_3$ region of the equilibrium phase diagram have significant room-temperature ductility.

6. Conclusions

Mechanical alloying has been used to produce binary Fe–Al intermetallics for a range of aluminium contents varying from 10–30 at %. Initial milling together of the elemental powders resulted in the dissolution of the aluminium into the iron lattice. Heat treatment of compacts of the milled powder resulted in complete suppression of the $D0_3$ (Fe_3Al) structure in favour of the B2 (FeAl) structure. It is thought that the high density of defects resulting from the heavy deformation are responsible. At aluminium contents below 22 at %, heat treatment of the milled material results in a gradual change from the B2 structure to that of α-Fe and this is considered to be due to the decreasing number of aluminium atoms available to occupy the B2 lattice sites. The change to this structure from the α-Fe structure following heat treatment indicates a transfer of the aluminium atoms from interstitial sites in the disordered b c c. Fe lattice to specific lattice sites

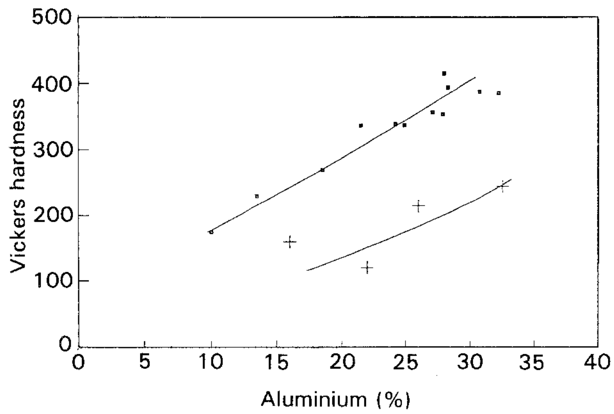

Figure 4 Plot of Vickers hardness versus per cent aluminium for (□) mechanically alloyed samples heat treated at 1150 °C for 4 h and for (+) melt-spun Fe–Al in the as-spun condition.

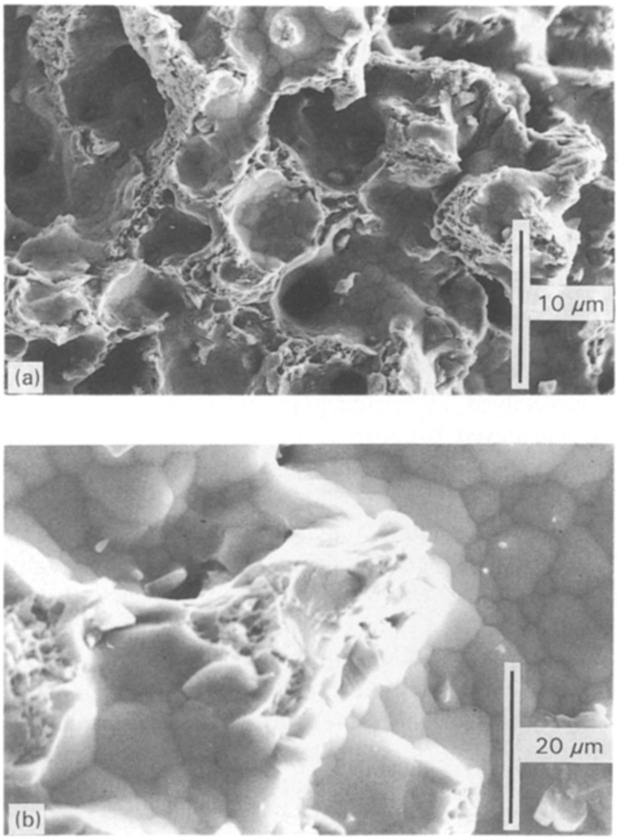

Figure 5 Scanning electron micrographs of fractured compact (heat treated for 4 h at 1150 °C).

in a more ordered B2 structure. A fine grain size and evidence of tearing indicate that mechanically alloyed Fe–Al intermetallics in the $D0_3$ composition range are ductile at room temperature.

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