Microstructure and Mechanical Properties of Mechanically Alloyed and HIP-Consolidated Fe$_3$Al

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Blended elemental Fe and Al powders were mechanically alloyed in a high energy attritor-type ball milling system. After 100 h milling, a Fe(Al) solid solution with bcc structure was formed in the powders. An ultra-high pressure (1 GPa) hot isostatic pressing (HIP) facility was used in an effort to consolidate the mechanically alloyed powders into fully dense compacts while retaining the nanocrystalline microstructure. Fully dense Fe$_3$Al compacts with a grain size of 180 nm were obtained by HIP treatment at 1073 K, whilst a 973 K HIP treatment produced nearly full density Fe$_3$Al compacts with a grain size of 80 nm. Mechanical properties of the HIPed compacts were evaluated by compression tests from room temperature to 1073 K and compared with those of a coarse-grained cast Fe$_3$Al alloy. The 973 K HIPed compact exhibited a yield strength as high as 2.3 GPa at room temperature, but it suffered from poor ductility. The 1073 K HIPed compact appears promising due to a combination of significantly higher strength, both at room temperature and at elevated temperatures, as well as better room temperature ductility than its cast counterpart.

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I. Introduction

Iron aluminides, such as Fe$_3$Al and FeAl, are attractive materials for high temperature applications because of the combination of low cost, conservation of strategic elements (Cr as an example), low density, and remarkable corrosion resistance. However, commercialization of these intermetallics has been limited due to their poor ductility at ambient temperature$^{10}$. For this reason, considerable work has been devoted to the improvement of the ductility of iron aluminides$^{25-30}$. These studies have shown that the ductility of Fe$_3$Al and FeAl intermetallics can be improved by composition modification and microstructure control.

Mechanical alloying, initially developed by Benjamin$^{6}$ in 1970s for the production of oxide dispersion-strengthened (ODS) superalloys, has been extensively exploited in the synthesis and processing of intermetallic compounds in recent years$^{7,10}$. This technique is unique in that (i) it is an inexpensive method, performed at room temperature using a simple equipment, (ii) it can yield relatively large quantities of powders, and (iii) most importantly, the mechanically alloyed powder generally consists of nanometer-sized grains, which is expected to improve the room temperature brittleness generally exhibited by most intermetallics.

The mechanical alloying process of Fe–Al system powders has been extensively studied by many workers$^{11-17}$. Generally, a nanocrystalline Fe(Al) solution with bcc structure was formed during mechanical milling. Upon heating or sintering at high temperature, the Fe(Al) solution was transformed into Fe$_3$Al or FeAl intermetallic compound with ordered DO$_3$ or B2 structure. However, there are only a few investigations$^{11,17}$ relating to the microstructure and mechanical properties of bulk materials consolidated from the nanocrystalline powders. This is probably due to the following two reasons: (i) laboratory scale milling systems were adopted in most studies and the small amounts of powders processed were not enough for the preparation of bulk materials of appropriate size; (ii) there was a lack of suitable consolidation techniques which could produce fully dense compacts while retaining the nanocrystalline microstructure.

The present study presents an effort to produce fully dense and nanocrystalline Fe$_3$Al intermetallics from elemental Fe and Al powders by mechanical alloying and hot isostatic pressing (HIP). The microstructural characteristics and mechanical properties of the Fe$_3$Al intermetallics produced by this approach were investigated and compared with those of a cast counterpart.

II. Experimental Procedure

Commercial metal powders of iron and aluminum (100 mesh, 99.9% purity) were blended in an atomic ratio of 75:25 to produce Fe$_3$Al intermetallic compound in the present study. Mechanical alloying was carried out in a high energy attritor-type ball milling system (Mitui Mike Attritor, Model MA5D-X) at an agitation speed of 150 min$^{-1}$ for up to 360 ks. The weight of powders was 3 kg, and the ball-to-powder weight ratio was 10:1. No process control agent was used in the milling. To prevent powders from oxidation during mill process, the milling was conducted under a pure argon atmosphere with an oxygen concentration below 100 ppm. Furthermore, the container was cooled with water to keep its temperature at about 293 K.
After the mechanical alloying, the milled powders were sieved (180 mesh), degassed at 773 K for 3.6 ks, and sealed into steel cans in vacuum. Then the cans were HIP-treated using an ultra-high pressure (up to 1 GPa) HIP facility. Under the high hydrostatic pressure, the cans were heated to the prescribed consolidation temperatures with a graphite tubular heater. Consolidation was performed at 973 K, 950 MPa or at 1073 K, 980 MPa, both for a hold time of 10.8 ks.

Mechanical properties of the HIP-consolidated compacts were evaluated by Vickers hardness and compression test. The Vickers hardness was measured under a load of 98 N. For compression test, specimens with dimensions of 3 mm × 3 mm × 6 mm were sectioned from the consolidated compacts by electro-discharge machining. These specimens were then tested in air at temperatures ranging from room temperature to 1073 K. The cross head speed employed was 8.3 × 10^{-3} mm/s, corresponding to an nominal strain rate of 1.4 × 10^{-3} s^{-1}.

The microstructures of mechanically alloyed powders and HIP consolidated compacts were characterized using X-ray diffraction (XRD), optical microscopy, scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Foils for TEM examination were prepared by electrolytic thinning at 228 K in a solution containing 30% nitric acid and 70% methanol.

### III. Results

1. As-milled powders

The main characteristics of 360 ks milled powder Fe_{75}Al_{25} are summarized in Table 1. The composition of Fe and Al is very close to the nominal one, and the impurities (C, O and N) are present at only several hundreds of atomic ppm. These illustrate that the contamination induced by mechanical alloying is not severe. The XRD patterns of Fe_{75}Al_{25} powder milled for different times are shown in Fig. 1. It can be seen that the blended Fe and Al powders are completely transformed into Fe(Al) solid solution with bcc structure after 360 ks milling. This result is consistent with those reported earlier^{10,11}. The average grain size of the milled powders is estimated to be 12.6 nm by using the Scherrer formula^{10}. This value is almost identical to that reported by Bonetti et al.,^{14} which was thought to be the intrinsic limit of grain size of mechanically alloyed Fe(Al). The particle morphology and cross-sectional microstructure of the milled powders are shown in Figs. 2(a) and (b), respectively. The average particle size of the milled powder is estimated to be about 80 μm. The cross-sectional image (Fig. 2(b)) shows that a homogeneous structure is formed after 360 ks milling.

2. HIPed compacts

To consolidate the 360 ks milled powders into fully dense compacts while retaining the nanocrystalline microstructure, ultra-high pressures of about 1 GPa and relatively low temperatures were used in the present study. Optical micrographs of the consolidated compacts after HIP treatment at 973 and 1073 K are shown in Figs. 3(a) and (b), respectively. Porosity is observed in the 973 K treated compact, and the pores tend to be located at the triple junctions of prior particle boundaries (Fig. 3(a)). However, the 1073 K treated compact exhibits a fully dense microstructure without any distinct porosity (Fig. 3(b)). Moreover, the prior particle boundaries are diminished. Thus, the low temperature limit to produce fully dense Fe_{75}Al_{25} compacts under a ultra-high pressure of 980 MPa is probably 1073 K. These results confirm that temperature is one of the most important controlling parameters for consolidation of mechanically alloyed

![Fig. 1 X-ray diffraction patterns showing structural evolution of Fe_{75}Al_{25} powder during mechanical alloying.](image)

<table>
<thead>
<tr>
<th>Particle size (μm)</th>
<th>Gain size (nm)</th>
<th>Lattice parameter (nm)</th>
<th>Chemical analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td>81</td>
<td>12.6</td>
<td>0.2905</td>
<td>C (ppm)</td>
</tr>
<tr>
<td>210</td>
<td>277</td>
<td>254</td>
<td>24.7</td>
</tr>
</tbody>
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powders by HIP treatment.\textsuperscript{19,20}

The XRD patterns of HIP-consolidated compacts at 973 and 1073 K are shown in Fig. 4. It is evident that Fe\textsubscript{3}Al intermetallic compounds with ordered B2 or D\textsubscript{0\textsubscript{3}} structure were formed in these compacts. By comparing Figs. 1 and 4, one can see that the diffraction peaks become sharper after consolidation, which indicates the occurrence of grain growth and/or internal strain release. To determine the grain size of consolidated compacts, TEM analysis was performed on the consolidated compacts and the typical micrographs are shown in Fig. 5. The average grain size was determined to be about 80 nm for the 973 K treated compacts and about 180 nm for the 1073 K treated compacts, indicating the occurrence of grain growth during HIP process.

3. Mechanical properties

The mechanical properties of the HIP consolidated compacts were investigated by means of hardness and compression tests. For comparison, a cast alloy with the same nominal composition was also tested. This cast alloy was prepared by vacuum induction melting with a grain size of about 1 mm. The hardness results are shown in Table 2. It can be seen that both of the HIP-consolidated compacts exhibit much higher hardness values than the cast counterpart. Moreover, the hardness of the 973 K HIPed compact is higher than the 1073 K HIPed one.

Figure 6 shows the room temperature stress-strain curves of the HIP consolidated compacts and the cast counterpart. Evidently, the HIP treated compacts exhibit significantly higher flow stress than the cast counterpart, in which the flow stress of the 973 K HIPed compact is higher than that of the 1073 K HIPed one. This result is consistent with the hardness measurements. It is noted that a yield strength as high as 2.3 GPa is attained in the compressive stress-strain curve of the 973 K HIPed compact. Such a high strength is considered to result from the nanocrystalline microstructure retained in the 973 K HIPed compact. However, nanometer-sized grains of the 973 K HIPed compact does not result in a better compressive ductility than the 1073 K HIPed compact and even the coarse-grained cast counterpart, which is thought to be associated with the pronounced porosity.
(Fig. 3(a)). On the contrary, the fully densified 1073 K HIPed compact exhibits not only higher flow stress but also better compressive ductility than the cast counterpart. A yield strength of 1.5 GPa is attained in the 1073 K HIPed compact. 

**Figure 7** presents the testing temperature dependence of the yield strength of the HIPed compacts and the cast counterpart. It can be seen that both of the HIPed compacts show superior yield strength to their cast counterpart in the whole temperature range. It is also noted that the 973 K HIPed compact shows higher yield
strength than the 1073 K HIPed one at temperatures below 673 K. Above 673 K, however, the strength of the 973 K HIPed compact decreases rapidly and becomes lower than that of the 1073 K HIPed compact.

IV. Discussion

The present work shows that Fe(Al) solid solution with a grain size of 12.6 nm can be formed by mechanical alloying of Fe and Al powders. The formation of the bcc Fe(Al) solid solution rather than the Fe₃Al intermetallic compound is suggested to be associated with the relatively high diffusivity of Al in α-Fe and low formation enthalpy of Fe₃Al."\(^{(22)}\) This nanocrystalline Fe(Al) solid solution is the precursor for producing nanocrystalline Fe₃Al intermetallic compound. Moreover, the fine particle size and nanometer grain size in mechanically alloyed powders are beneficial to the subsequent full densification due to reduced diffusion paths."\(^{(22)}\)

However, consolidation of the mechanically alloyed powders into fully dense compact while maintaining nanoscale grain size remains a major challenge in this work. Full densification of mechanically alloyed powders requires exposure to high temperature, which leads to grain coarsening and/or diminishing of the nanostructure. As a result, the consolidated compacts are either of near full density (above 90% of theoretical density) with nanometer grain sizes (smaller than 100 nm) or of full density with grain sizes ranging from submicron to several microns. In the present study, by using an ultra-high pressure (1 GPa) HIP facility, nearly full density Fe₃Al compacts with a grain size of 80 nm had been produced at 973 K, whilst fully dense Fe₃Al compact with a grain size of 180 nm had been produced at 1073 K. Compared with the initial grain size of 12.6 nm, it appears that, though grain growth has occurred, the grain growth is not very strong. This is because the atomic jumps across grain boundaries are greatly inhibited by the high pressure applied, as suggested by Hahn et al."\(^{(22)}\)

In the present study, the HIPed compacts exhibit superior hardness, room temperature strength and high temperature strength to the cast counterpart. Grain refinement is considered to be the dominant factor which contributes to the high hardness and strength of the HIPed materials. The strengthening due to grain refinement generally follows the well-known Hall-Petch relationship which is based on dislocation pile-ups at grain boundaries. However, as pointed out by Jain and Christman"\(^{(23)}\), the dislocation pile-ups are unlikely to occur in nanophase intermetallic compounds where the grain boundary strength tends to be lower than the yield strength. They suggested that Li’s model"\(^{(20)}\), which is based on dislocation generation from grain boundary ledges, may hold in nanophase intermetallic compounds. According to this model, the generated dislocations produce a Taylor dislocation forest, and the yield strength of the material is determined by the stress required to move the additional dislocations through the forest. The macroscopic shear yield strength is expressed by

\[
\tau = \tau_0 + \alpha \mu b \left( \frac{8m}{\pi d} \right)^{1/2}
\]

where \(\tau\) is the shear strength (taken to be half the axial yield strength), \(\tau_0\) the coarse-grained shear strength, \(\alpha\) a constant ranging from 0.2 to 0.4, \(\mu\) the shear modulus, \(m\) the ledge density, \(b\) the Burger’s vector, and \(d\) the grain size. A rough calculation is made here by using the following parameters: \(mb = 0.2\)"\(^{(20)}\), \(b = 0.25\), \(\tau_0 = 0.3\) (the coarse-grained strength), and \(\mu = 70\) (all length terms in nm and strength terms in GPa). The calculated yield strength is 1.7–2.8 GPa for the 973 K HIPed compact and 1.3–2.1 for the 1073 K HIPed compact. The observed yield strength (2.3 GPa for the 973 K HIPed compact and 1.5 GPa for the 1073 K HIPed one) appears to be within the calculated strength range, respectively. This suggests that the strengthening by grain refinement can be modelled well by the grain boundary strengthening theory based on dislocation generation from grain boundary ledges.

Although the 973 K HIPed compact exhibits very high hardness and room temperature strength, it suffers from low room temperature ductility. Moreover, the strength of the compact decreases very rapidly at temperatures above 673 K. This decrease is considered to result from a fast diffusion process which is associated with the nano-sized grains and porosity. From this point of view, the 1073 K HIPed compact is more promising due to its combination of high strength at both ambient and elevated temperatures, as well as good room temperature ductility. The high strength exhibited by the 1073 K HIPed compact at temperatures up to 1073 K is attributed to the possible dispersoids induced during mechanical alloying and/or HIP consolidation. Further creep tests together with dislocation substructure examinations will help to gain valuable insight into the strengthening mechanism of this fine-grained Fe₃Al material at elevated temperatures.

V. Conclusions

(1) Mechanical alloying of blended Fe and Al powders led to the formation of nanocrystalline Fe(Al) solid solution with bcc structure. The Fe(Al) solid solution was transformed into ordered Fe₃Al intermetallic compound upon HIP consolidation.

(2) By using an ultra-high pressure (980 MPa), fully dense Fe₃Al compacts with a grain size of 180 nm was obtained by HIP treatment at 1073 K, whilst 973 K HIP treatment produced nearly full density Fe₃Al compacts with a grain size of 80 nm.

(3) The HIP-consolidated Fe₃Al compacts exhibited much higher hardness and strength than the cast counterpart due to grain refinement. This strengthening can be modelled well by the grain boundary strengthening theory based on dislocation generation from grain boundary ledges.
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REFERENCES