ISSN 2816-573X

ORIGINAL ARTICLE

Microstructural and Thermal Properties of the Mechanically Alloyed Fe_3Al Powders Doped with Boron

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Mhadhbi M, Driss M, Safi B, et al. Microstructural and Thermal Properties of the Mechanically Alloyed Fe_3Al Powders Doped with Boron. Int J Adv Nano Comput Anal. 2024;3(1):01-05.

Abstract

A nanocrystalline Fe_3Al powders free and doped with boron was prepared by mechanical alloying (MA) of Fe and Al elemental powders using a high-energy planetary ball mill. The evolution of microstructure and the thermal behaviour of the MA Fe_3Al powders have been studied using X-ray diffraction (XRD), scanning electron microscopy (SEM) with energy dispersive X-ray analysis (EDX) and differential scanning calorimetry (DSC). The obtained results showed that the crystallite size was refined and that the lattice parameter was increased by addition of boron. The DSC analysis revealed that the addition of boron results a decrease of microstructural parameters and recrystallization take place.

Key Words: *Nanostructure; Thermal properties; Mechanical alloying; Boron; FeAl*

Introduction

Fe-Al intermetallics have been widely studied among this field because of their low cost, low density, good wear resistance, ease of fabrication, and resistance to oxidation and corrosion. However, there are some inferior properties to be conquered for commercial applications, such as low ductility exhibited at low temperature and limited workability [1-3]. There are many methods to improve the material properties, such as composites, addition of elements, heat treatment, control of grain size, and so on [4]. Especially, the control of grain size to nanometer scale and the addition of elements will effectively improve the poor properties of Fe_3Al intermetallics, because the fine grain size can increase the yield strength and improve the ductility [5], and the addition of elements can strengthen the matrix phase and grain boundaries, and may suppress the grain growth.

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Received: June 02, 2024, Accepted: June 27, 2024, Published: July 02, 2024

Conventional methods of processing Fe_3Al intermetallic, including melting and casting, and traditional powder metallurgy, have been investigated. In recent years, some efficient methods were reported to fabricate the fine-grain-size metals and alloys. These included mechanical alloying and spark plasma sintering (SPS) [6,7]. The mechanical alloying process involves repeated cold-working, fracturing, and welding results in microstructure refinement and alloy formation. Microstructural refinement may easily result in fine grains of micrometer size particles. The shortcoming of this process is the ease of formation of microdefects that may decrease the properties of the final products and import pollution [8]. The SPS is a rapid solidification processing method; and uniform, dense, and fine-grain materials can be obtained by applying pressures and passing electric pulse current to the compact. The atom migration activity and diffusion rate were enhanced because of the spark plasma between the particles [9, l0]. The addition of elements was another effective method to improve the mechanical properties of Fe_3Al intermetallics, and nickel, chromium, boron, and carbon etc. have been used [11-13] for this purpose. In another hand [14], the Fe_3Al-based intermetallics were prepared by mechanical alloying and spark plasma sintering, and the influence of milling time on the properties of materials was investigated.

Additionally, Bormio-Nunes et al. [15] investigated the magnetostriction of the polycrystalline $Fe_{80}Al_{20}$ alloy doped with boron. The XRD results showed an increase in the volume fraction of α-FeAl phase and a decrease of the volume fraction of Fe_3Al phase as the boron content increases. Gedsun et al. [16] studied the new Fe-Al-Nb(B) alloys for structural applications at high temperatures. They revealed that no significant effect of boron on the microstructure and the mechanical properties was observed. In addition, the yield stress of Fe-Al-Nb alloys was in the same order of magnitude as for Fe-Al-Ta alloy of comparable microstructure and composition. Furthermore, the concept of strengthening via incoherent precipitates borides has been developed to enhance the mechanical properties of Fe_3Al based iron aluminides [17].

The purpose of the present work was to study the effect of born addition on the microstructural and thermal evolution of Fe_3Al powder prepared by mechanical alloying process.

Experimental

In first route, elemental powders of Al (99.93% purity) and Fe (99.99% purity) were mixed at Fe-28at.% Al and milled at room temperature under argon atmosphere. In second route, the mixture powder was doped with boron (Fe-28at.% Al + 0.2 at.% B) The purity of boron powder was 99.99%. MA process was carried out in a high-energy planetary ball mill Fritsch Pulverisette 7 (Germany). A steel vial and balls were used. The time of milling was shared to avoid the elevation of temperature inner the vial. The thermal behaviour of the samples was examined by differential scanning calorimetry using a DSC822e calorimeter at the range temperature of 35ºC to 700ºC in purified argon atmosphere and at a constant heating rate of 20°C/min. The obtained powders were characterized by using X-ray diffraction with CuK_α radiation and the characteristic peaks of the XRD being collected within a diffraction angle ranging from 20 to 90 degree. The morphology of the samples was studied using a FEI Quanta 200 environmental scanning electron microscopy (SEM) coupled with energy dispersive X-ray analysis (EDX).

Results and Discussion

Figure 1 shows the XRD patterns of the Fe-28 at.%Al powder without and with born after different milling times. As can be seen, a single

DO_3 structure appears for the sample without boron. For the un-milled powder, the pattern shows only the peaks of principal elements of Fe and Al Figure 1a. After 4 h of milling, the starting powder (3Fe+Al) reacted producing highly disordered phase (Fe_3Al), shown in Figure 1b. After 20 h of milling Figure 1c, the peaks became more broadened and more intense due to the decrease of crystallite size caused by plastic deformation introduced by MA. When boron is added Figure 1d, no superlattice reflection was observed. Thus, the milled powder exhibits a fully disordered structure. In addition, the profiles of all Bragg peaks of MA powder are significantly broadened. This is due to the reduction of mean crystallite size and to the increasing of lattice strain. This decrease can be associated with a segregation of B atoms in the grain boundaries [18], which avoids the growth of crystallites of the DO_3 phase.

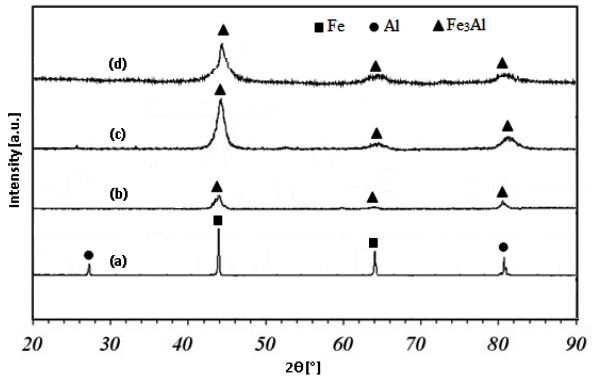

Figure 1) *XRD of Fe_3Al powder: (a) un-milled, (b) MA for 4 h, (c) MA for 20 h and (d) of Fe_3AlB powder MA for 20 h.*

The Table 1 shows the microstructural parameters of milled powders calculated from XRD data using Rietveld refinement program. For the case of FeAl without boron, the mean crystallite size decreases from 45 nm after 4 h to 9 nm after 20 h of milling. However, after addition of boron, the crystallite size decreases to 7 nm due to the ordering of alloy. The mean microstrain increases from 0.52 for 4 h to 1.71 % after 20 h of milling. This increasing is related to the plastic deformation introduced during MA process. After 20 h of milling, we noticed that the lattice parameter is larger than one milled for 4 h. In the case of doped powder, the lattice parameter increases slowly to reach a value of 0.58862 nm. This suggests a solutioning of boron in the concentration range [19].

TABLE 1
Microstructural parameters of the milled powders before and after doping.

Sample	<D> (nm)	$<e^2>^{1/2}$ (%)	a (nm)
Fe28Al MA for 4 h	45	0.52	0.57835
Fe28Al MA for 20 h	9	1.71	0.58272
Fe28Al + 0.2% B MA for 20 h	7	1.08	0.58862

The DSC thermograms of samples are shown in Figure 2. For the un-milled powder (Fe28 at.%Al + 0.2 at.% B), we can be seen the presence of an endothermic peak attributed to the fusion of small quantity of aluminium. After milling for 4 h, three exothermic peaks are observed at about 379°C, 488°C, and 627°C, respectively. The low temperature peak (T = 379°C) corresponds to the ordering of disordered Fe_3Al alloy by mechanisms of atomic-scale interchange and the movement of defects. The peak at about 488°C relates to the formation of FeAl phase. The peak at higher temperature (627°C) is related to the loss of various defects and grain growth in the FeAl intermetallics. In the case of powders milled for 10 h, three exothermic peaks are observed at 217°C, 484°C, and 538°C. After 20 h of milling, four exothermic peaks are observed at about 227°C, 418°C, 534°C, and 585 °C. The first peak is attributed to the short-range ordering of the alloy. The second peak (at 418°C) is probably due to the long-range ordering. The third peak (at 534°C) is related to annealing out of dislocations. The peak at higher temperature (585°C) is attributed

to the recristallization process related to the interaction of boron atoms with point defects. This results are similar to the one obtained by Baro et al. [20].

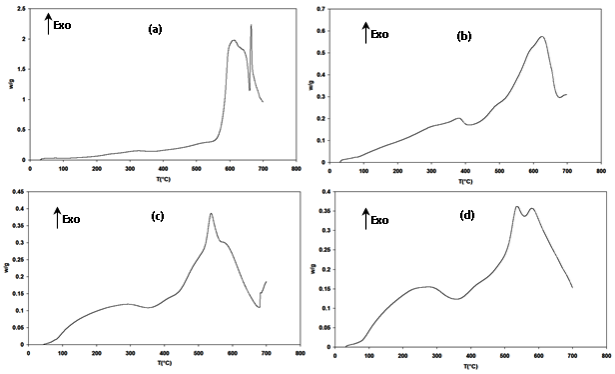

Figure 2) *DSC thermograms of the Fe3Al powders doped with born (0.2 at% B) samples (a) un-milled and milled for (b) 4 h, (c) 10 h, and (d) 20 h.*

Figure 3 shows SEM image of Fe_3Al powders milled for 20 h. We can be seen that Fe and Al were uniformly distributed and the particles of milled powder are refined and the mean grain size obtained is about 50 μm. Furthermore, during milling process, iron and aluminum powders were wrapped with each other by server plastic deformation resulting in grain refinement.

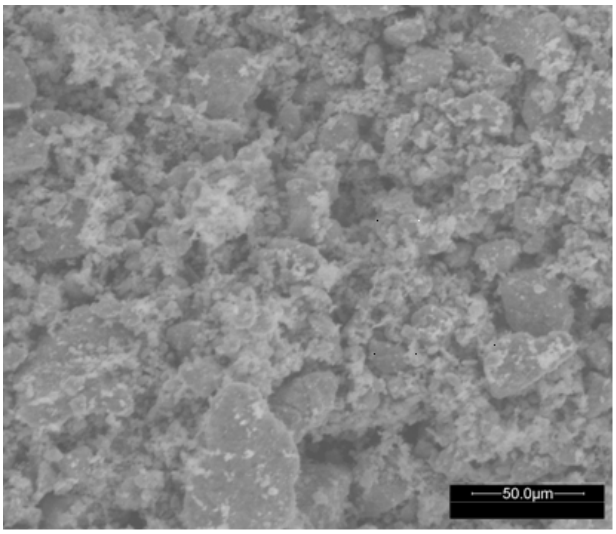

Figure 3) *SEM image of Fe3Al powder milled for 20 h.*

Figure 4 shows typical example of energy dispersive X-ray analysis (EDX) spectrum of the Fe_3Al powder milled for 20 h. As can be seen in figure, the EDX examination shows the presence of main elements (Fe and Al) and carbon. Indeed, a small amount of oxygen with iron and aluminium, which is explained by contamination of the powder upon manipulating the vials.

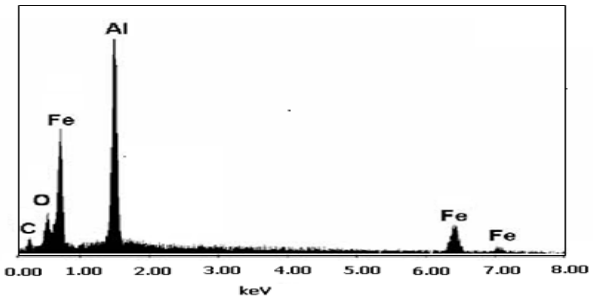

Figure 4) *EDX spectrum of Fe3Al powder MA for 20 h.*

Conclusion

- The effect of 0.2 at.% B on the microstructure and thermal properties at room temperature of the Fe-28at.% Al powders was investigated.
- Nanocrystalline FeAl-B powders were successfully obtained by MA process. The XRD results revealed that the crystallite size was refined whereas the lattice parameter was increased by addition of boron.
- The DSC analysis showed that the addition of boron results a decrease of microstructural parameters (mean crystallite size and mean microstrain) and the recrystallization of structure.
- The SEM results showed the agglomeration of powder particles for longer milling time.

Acknowledgement

The authors would like to express sincere thank to Mr. Allison Tancredi, the Managing Editor, for his efforts.

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