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Nanophase intermetallic FeAl obtained by sintering after mechanical alloying

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ABSTRACT

The preparation of bulk nanophase materials from nanocrystalline powders has been carried out by the application of sintering at high pressure. Fe–50 at.%Al system has been prepared by mechanical alloying for different milling periods from 1 to 50 h, using vials and balls of stainless steel and a ball-to-powder weight ratio (BPR) of 8:1 in a SPEX 8000 mill. Sintering of the 5 and 50 h milled powders was performed under high uniaxial pressure at 700 °C. The characterization of powders from each interval of milling was performed by X-ray diffraction, Mössbauer spectroscopy, scanning and transmission electron microscopy. After 5 h of milling formation of a nanocrystalline α-Fe(Al) solid solution that remains stable up to 50 h occurs. The grain size decreases to 7 nm after 50 h of milling. The sintering of the milled powders resulted in a nanophase-ordered FeAl alloys with a grain size of 16 nm. Grain growth during sintering was very small due to the effect of the high pressure applied.

1. Introduction

Mechanical alloying is a very convenient and versatile method to produce nanocrystalline powders. Consolidation of milled powders into bulk, full-density compacts preserving nanometric grain size is difficult to attain, although extremely important, to have advantage of the properties offered by a nanophase grain size. The consolidation of powders keeping the nanocrystalline grain size has been tried by a number of different authors [1,2]. One of the most attractive methods has been the application of high pressure and high temperature for short periods of time [2]. Different consolidation processing techniques to obtain bulk nanostructured materials have been carried out, among them high velocity oxy-fuel (HVOF) and spark plasma sintering (SPS) have been studied comparatively [3].

Intermetallic compounds are of particular interest due to their excellent properties to oxidation and corrosion resistance at high temperatures. Among these, FeAl is a very attractive compound for structural applications at elevated temperatures due to its physicochemical properties: low density, high oxidation and corrosion resistance, relatively high strength as well as low cost. However, one of the main drawbacks of the intermetallic compounds is their very low ductility. It has been argued that in nanocrystalline materials ductility is better than in microcrystalline ones, also it has been proposed that ductilisation of brittle materials may result from the nanocrystalline structure [4]. Some authors have shown improved ductility by reducing the grain size from 100 to 1 μm [5]. The influence of different factors on ductility of Fe aluminides has been studied [6].

The objective of the present work is the synthesis and consolidation of nanophase intermetallic FeAl and the study of the phase transformations induced by the sintering process in the mechanically alloyed powders.

2. Experimental

Elemental high purity Fe and Al powders, analytical grade, with an average particle size of 4.5 and 12 μm, respectively, were mixed in a proportion of 50 at.% in a WAV turbule during 1 h and then mechanically alloyed under nitrogen atmosphere, using a SPEX 8000, for different milling periods: 1, 3, 5, 10, 20, 30 and 50 h, using vial and balls of stainless steel, and a ball-to-powder weight ratio (BPR) of 8:1. Sintering of the milled powders was performed at 700 °C and 2 GPa for 30 min in a uniaxial press equipped with a high temperature furnace. The characterization of the milled powders and sintered material was carried out by X-ray diffraction, scanning, transmission electron microscopy and Mössbauer spectroscopy. X-ray diffraction (XRD) was carried out using a Siemens 5005 diffractometer with Cu Kα. Grain size was calculated by the Scherrer equation. Scanning electron microscopy (SEM) analysis was performed in a Phillips XL30 attached with an EDX-DX4. Transmission electron microscopy (TEM) was carried out in a Phillips CM10. Mössbauer spectra were measured at room temperature (RT) using a constant-acceleration transducer arranged in a transmission geometry, with a ^{57}Co source of 50 mCi in Rh matrix. A triangular drive waveform was used and the data were collected in 512 channels, with ~10^6 counts per channel, and were folded to remove any base-line curvature. The velocity scale for Mössbauer spectra was calibrated related to α-Fe at RT. Microhardness

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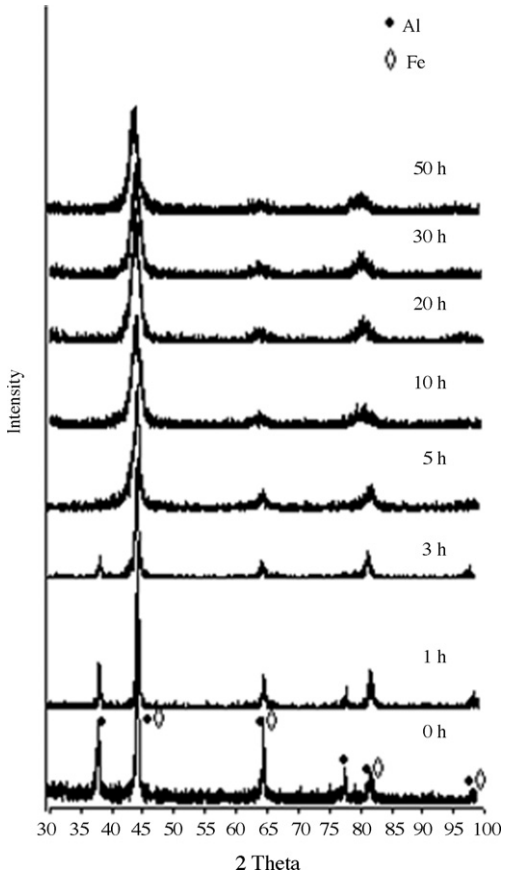

Fig. 1. XRD patterns of Fe–50 at.%Al powders milled.

tests were performed to the sintered samples in a microhardness MTH-4 equipment.

3. Results and discussion

Fig. 1 shows the XRD pattern for the Fe–Al powders for different milling periods; peak broadening and shifting towards lower 2θ angles is observed. After 5 h of milling, the system forms an α-Fe(Al) solid solution that remains stable up to 50 h. This transformation has been reported by different authors [7–13]. The lattice parameter of the Fe(Al) increases from 2.866 to 2.889 Å and the grain size, calculated by the Scherrer equation, reaches 7 nm after 50 h of milling. Fig. 2 shows the morphological changes of the powder particles observed by SEM, during the first milling periods. The characteristic lamellar structure is present from 1 h to 3 h of milling. These results suggest that deformation during milling induces diffusion of one of the elements into the other forming a disordered solid solution.

The formation of a nanocrystalline material is obtained after 5 h of milling. The evolution of the grain size observed by TEM is shown in Fig. 3, after 5 h of milling the grain size is about 20 nm and reaches an average grain size of 7 nm after 50 h of milling. The determination of grain size from the XRD patterns, using the Scherrer equation was 14 nm after 5 h of milling and 7 nm after 50 h in good agreement with the values determined from TEM. The Mössbauer spectra for these samples are shown in Fig. 4. For 5 h of milling a complex spectrum is obtained, the fitting of this spectrum was carried out with four sub-spectra: one formed by the typical sextet with a hyperfine magnetic field of 330 Gauss assigned to metallic iron, and the other three are non-magnetic sub-spectra associated to the different environments of the atoms of iron surrounded by aluminium atoms.

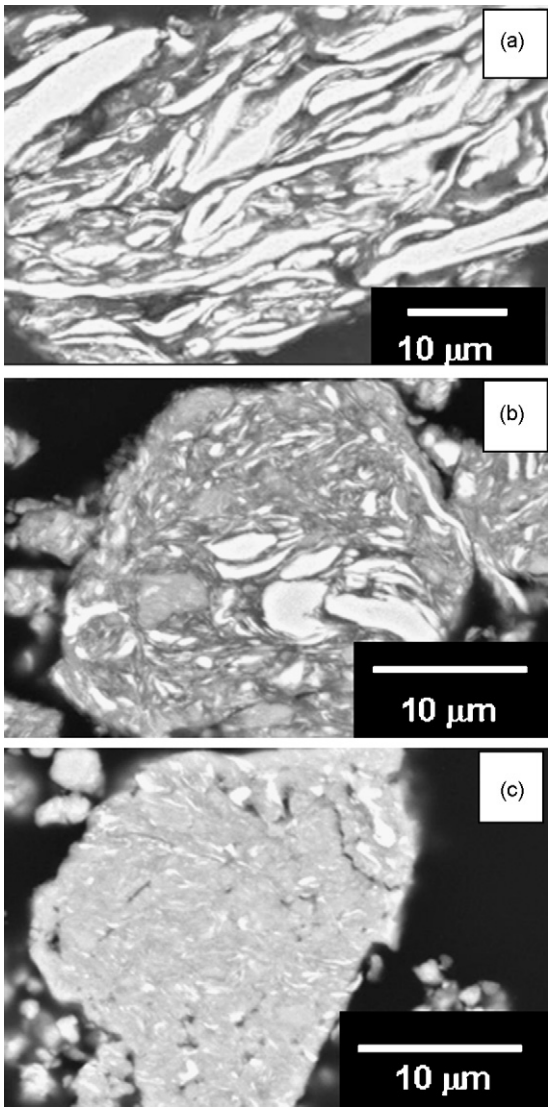

Fig. 2. SEM images of mechanically alloyed powders Fe–50 at.%Al, at (a) 1 h, (b) 3 h and (c) 5 h.

For the sample mechanically alloyed 50 h, a non magnetic spectrum is obtained, formed by a singlet associated to the formation of a paramagnetic Fe(Al) bcc solid solution and a doublet associated to the contribution of a disordered paramagnetic phase. The fitting parameters for the spectra are shown in Table 1. A paramagnetic spectrum in Fe–Al after prolonged milling has been reported by Ealman et al. [13] for samples with Al content higher than 50 at.%, prepared after prolonged milling by mechanical alloying.

Table 1
Fitting parameters of the Mössbauer spectra for Fe–50 at.%Al mechanically alloyed for 5 and 50 h.

Milling time (h)	Sub spectra	Parameters			
		IS (mm/s)	QS (mm/s)	AG (mm/s)	H (kG)
5	1	−0.160	0.000	0.1	1
	2	−0.107	0.000	0.2	330
	3	0.132	0.000	0.2	0.00
	4	0.112	0.533	0.2	0.00
50	1	0.126	0.000	0.2	0.00
	2	0.088	0.399	0.196	0.00