



Microstructure and room-temperature mechanical properties of crystalline and amorphous FeAl based melt spun ribbons

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ARTICLE INFO

Keywords:

Fe–Al alloys

Microstructure

Mechanical properties

Melt spinning

ABSTRACT

The microstructure and mechanical properties of crystalline Fe81%–Al19% and amorphous Fe72%–Al14%–B14% ribbons obtained by the melt spinning technique are studied. A different grain size in each surface of the crystalline ribbon has been found. This is caused by the temperature gradient originated by the manufacturing process. The micro-hardness and the Young's modulus were evaluated by instrumented micro-indentation (P-h curves) and conventional tensile test. The results show different Vicker's hardness on each surface for each of the ribbons, being these values greater in the amorphous ribbon than in the crystalline one. The values of the Young's modulus are 133 ± 11 GPa and 210 ± 20 GPa for the crystalline and amorphous ribbon respectively which are in good agreement with the theoretical values obtained by the mix rule. The surface fracture shows a brittle-like aspect with presence of cleavage in the Fe81%–Al19% ribbon and small localized ductile behavior before reaching the fracture. In the other hand, in the Fe72%–Al14%–B14% ribbon, the surface fracture characteristics reveal a fragile fracture and the presence of chevron marks. There is a concordance between the mechanical behavior and the micro-mechanism responsible for the fracture.

1. Introduction

FeAl alloys have attracted a great deal interest for their potential structural applications at elevated temperature in hostile environments. This is due to their excellent oxidation behavior and relatively low density and low cost [1]. Recent studies about the mechanical properties of FeAl alloys [2] have shown that these properties, are dramatically affected by the fabrication process and their ductility in air is low [3]. Various reviews [4,5] have shown that the mechanical properties of FeAl alloys depend on a wide variety of parameters, including the manufacturing process, vacancy concentration, heat treatments, environmental conditions, alloy stoichiometry and grain size.

The use of some non-equilibrium processing techniques such as rapid solidification technologies and powder metallurgy allow a broader range of compositions of Fe–Al alloys. This has allowed us to notice that depending on the Al concentration the magnetism of these alloys changes, presenting a ferromagnetic phase when this concentration is lower than 35 at.% Al and a change to paramagnetic state for concentrations higher than 35 at.% [6,7]. These properties depend on the intermetallic order and can change with mechanical deformation [8]. Another interesting property obtained in these alloys with high Fe

concentration is their magnetostriction [9]. This property opens the application of these materials to the field of magnetostrictive sensors. Some years ago, the melt spinning technique was used to produce ribbons of FeAl with different composition [10–13], achieving even larger values of the magnetostriction.

Intermetallic based alloys have some desirable properties but, as mentioned before, they also exhibit brittleness at room temperature which restricts their use. Liu et al. [3] have shown that moisture-induced environmental embrittlement is the main source of this room temperature brittleness; another contribution to this is the effect of the grain boundaries [14]. To overcome this limitation, different works have been devoted to studying the influence of the addition of B in the mechanical properties of these alloys [15,16]. These studies have shown that the addition of small quantities of B increases the ductility and the amount of transgranular fracture. In the case of rapidly solidified FeAlB ternary alloys obtained by copper mold casting, a considerable improvement of the strength is achieved [17]. However, it has also been shown that rapidly quenched Fe–Al–B alloys containing 20–40 at. % Al and 5–7 at. % B are extremely brittle [18]. Nevertheless, the possibility of obtaining amorphous materials based in Fe–Al with a suitable quantity of boron could be of great interest due to the

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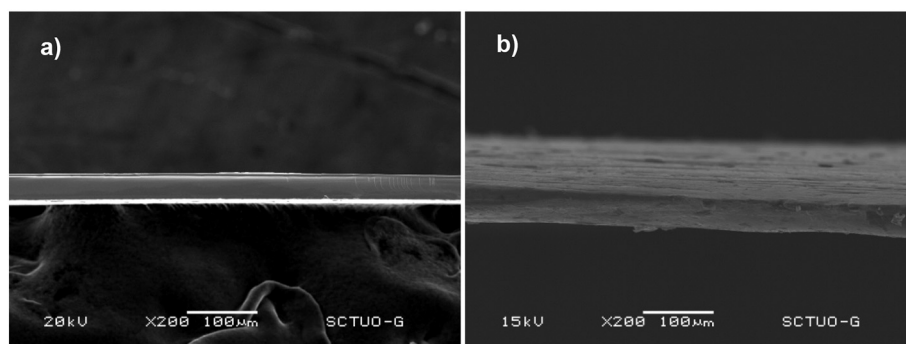


Fig. 1. SEM images of the ribbons thickness: a) $\text{Fe}_{81}\text{Al}_{19}$ and b) $\text{Fe}_{72}\text{Al}_{14}\text{B}_{14}$.

magnetoelastic coupling and mechanical properties that this kind of materials present. In this way, some of the authors of this article have shown that it is possible to obtain an amorphous ribbon by the melt spinning technique of composition $\text{Fe}_{72}\text{Al}_{14}\text{B}_{14}$ with reasonable magnetostriction [19].

In this work, a study of the microstructure, mechanical properties and deformation fracture of $\text{Fe}_{81}\text{Al}_{19}$ nanocrystalline and $\text{Fe}_{72}\text{Al}_{14}\text{B}_{14}$ amorphous ribbons obtained by the melt spinning method is presented. These alloys were chosen due to their magnetostriction, which makes them of great interest for their use in sensor technology and the possibility of comparing the mechanical properties of Fe–Al based alloys in the crystalline and amorphous state.

2. Experimental method

Ingot of nominal composition $\text{Fe}_{81}\text{Al}_{19}$ and $\text{Fe}_{72}\text{Al}_{14}\text{B}_{14}$ were prepared by melting appropriate amounts of high purity Al (99.96%), Fe (99.98%) and B (99.8%) in vacuum atmosphere in a high-frequency induction furnace. They were re-melted three times in order to make the alloy homogeneous. The melting of the alloys was carried out in a graphite crucible closed at the end with a tape of boron nitride in order to minimize the contamination in the master alloy during the melting. A rapid solidification process of the master alloy was carried out by planar flow casting in vacuum atmosphere with copper wheel of 20 cm diameter at a linear speed of 17.5 m/s. The $\text{Fe}_{81}\text{Al}_{19}$ and $\text{Fe}_{72}\text{Al}_{14}\text{B}_{14}$ melt-spun ribbons are 4 mm wide and 60 μm thick and 3 mm wide and 33 μm thick respectively.

Planar flow casting is one of the most popular techniques of rapid solidification for ribbon manufacturing. This processing method has a large number of variable parameters such as ejection temperature, orifice diameter, Argon pressure, the angle formed by the jet and the tangential surface to the wheel at the point of contact, wheel speed. Thus, depending on the parameters and alloy composition, amorphous or nanocrystalline ribbons can be obtained. For a given composition, the structure depends basically on the ribbon thickness and the melt temperature.

The chemical composition of the ribbons was determined by inductive coupled plasma (ICP) and by an electronic microprobe. Phases and crystallinity analyses were performed by X-ray diffraction with a Siemens D-50 diffractometer working in Bragg-Brentano configuration and using $\text{Cu-K}\alpha$ radiation with $\lambda = 1.3518 \text{ \AA}$. The structural details of the crystalline samples have been analyzed by transmission electron microscopy (TEM) with a Jeol 2000 microscope. In order to observe the samples by TEM they were previously thinned using the ion milling technique (Gatan Duomill 600 DIF).

The micro-mechanical behavior was studied by the conventional indentation technique and instrumented micro-indentation tests (P-h curves) [20]. In both cases, a Vickers pyramid diamond tip was used. The first test was carried out in a micro-durometer (Buehler - Model 1600.6100), at a load of 0.3 N (5 s dwell-time), on both sides along the

width of the ribbons. On the other hand, P-h curves were performed in a MICROTTEST device (MTR3/50–50/N1), at a load rate applied of 1 and 2 N/min and holding times of 10 s. In addition, the potential anisotropic mechanical behavior of ribbons, longitudinal and transverse direction, was evaluated. To perform the measurements, the samples were fixed to a steel cylinder. The surface of the samples was prepared using fine abrasive papers, the final polishing was performed with diamond particles in suspension and colloidal silica. At least three measurements were made for each type of material and test condition. The hardness, Young's modulus and the recovery elastic deformation from the resulting load–penetration curves were evaluated using the Oliver and Pharr analysis [21–24]. Finally, a Zwick 3212 hardness testing machine was used to corroborate the previous results (ASTM Standard E92).

The tensile testing was performed in a universal tester MTS400/M, with a load cell of 2 kN and flat grips. The strain was measured by an extensometer (MTS 634.25F-854) with a sensitivity of 1 μm and the load-strain tracks were automatically recorded. Samples for tensile testing had a gauge length of 13 cm and were tested to failure at room temperature. A constant cross-head speed of 0.05 mm/min was employed to conduct uniaxial tension experiments along the longitudinal direction of the ribbon. These measurements were performed in five ribbons of each composition to determine the statistical errors. The fracture feature of surfaces of the ribbons was observed by scanning electron microscopy (JEOL JSM-5600).

3. Results and discussion

Fig. 1 shows SEM images of the $\text{Fe}_{81}\text{Al}_{19}$ and $\text{Fe}_{72}\text{Al}_{14}\text{B}_{14}$ melt spun ribbons of 60 μm and 33 μm thickness, respectively. As can be seen in Fig. 1 both samples exhibit uniform thickness along their length. Fig. 2 shows the aspect of the surface roughness and microstructure, average size and morphology of the grains, for both sides of the ribbons. In this context, the influence of the different cooling rate in the size of the equiaxed grains, 4 μm in the wheel surface and 9 μm in the air surface in the ribbon $\text{Fe}_{81}\text{Al}_{19}$, and the absence of them in the amorphous ribbon, is confirmed.

Fig. 3 shows the XRD patterns of the air side of the ribbons. The observed reflections at $2\theta = 44.2^\circ$, 64.4° and 81.3° in the $\text{Fe}_{81}\text{Al}_{19}$ pattern can be indexed as the three peaks (110), (200) and (211) indicate the presence of the A2 and B2 phases but the (111) peak corresponding to the DO3 superstructure is not detected. These results suggest a bcc crystal structure and indicate that the non-equilibrium fabrication process restricts the formation of the DO3 ordered Fe_3Al intermetallic phase, which normally exists in Fe–Al-based alloys produced under equilibrium conditions [25,26]. Li et al. [17] have also found the same restriction of the formation of the DO3 ordered phase due to the non-equilibrium fabrication process in Fe–Al–B ternary alloys rapidly solidified in a copper mold. The amorphous state of the ribbon $\text{Fe}_{72}\text{Al}_{14}\text{B}_{14}$ has been confirmed by XRD studies, as can be seen in Fig. 3b, where the XRD pattern of the ribbon is shown. A broad

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