MICROSTRUCTURAL CHARACTERIZATION OF Fe40Al5Cr (% at.) INTERMETALLIC ALLOY PRODUCED BY RAPID SOLIDIFICATION.

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ABSTRACT

In this work microstructural characterization of melt-spun intermetallic compound Fe40Al5Cr (% at.) produced by rapid solidification employing melt spinning technique at two different tangential wheel speeds (12, 16 ms^{-1}) is presented. Melt spun ribbons were characterized by Optical and Scanning Electron Microscopy (SEM) in order to observe morphology, grain size and ribbons thickness. EDS technique was employed to perform punctual and scan line chemical analyses on samples, X-Ray Diffraction (XRD) was utilized to determinate crystal structure and phases and Transmission Electron Microscopy (TEM) was employed to confirm crystal structure and also to characterize nano pores formed in the specimens by vacancy clustering. The grain size was reduced upon the wheel speed increment or rate of cooling, the punctual and scan line chemical analyses revealed that Cr enter in solid solution in FeAl matrix. The size and orientation of superficial meso pores as well as nano pores inside the ribbons were characterized by electron microscopy.

Keywords: Rapid-solidification, melt-spun-ribbons, meso-pores, nano-pores.

CARACTERIZACIÓN MICROESTRUCTURAL DE UNA ALEACIÓN INTERMETALICA Fe40Al5Cr (% at.) PRODUCIDA MEDIANTE SOLIDIFICACIÓN RÁPIDA.

RESUMEN

En este trabajo se presentan resultados concernientes a la caracterización microestructural de cintas de aleación Fe40Al5Cr (% at.) que fueron producidas mediante solidificación rápida empleando la técnica de "melt spinning" (rueda girante), a dos diferentes velocidades tangenciales de rueda (12, 16 ms^{-1}). Las cintas solidificadas rápidamente fueron caracterizadas mediante microscopía óptica y electrónica, con el propósito de observar morfología y tamaño de los granos y espesor de las cintas. Así mismo, se realizaron análisis químicos puntuales mediante EDS y de barrido en línea, también se realizaron análisis de rayos x (DRX) con el objeto de determinar la estructura cristalina, constantes de red y fases. De la misma manera, los especímenes se observaron mediante microscopia electrónica de transmisión (MET) con la finalidad de confirmar la estructura cristalina y caracterizar nano poros formados en los especímenes originados por la aglomeración de vacancias. Se observo que el tamaño de grano y el espesor de las cintas se redujeron con el incremento de velocidad de rueda ó rapidez de enfriamiento. Los análisis químicos puntuales y de barrido en línea revelaron que el cromo entra en solución solida en la matriz FeAl. Los meso poros superficiales, así como también los nano poros observados en el interior de las cintas fueron caracterizados mediante microscopia electrónica.

INTRODUCTION

Iron aluminides exhibit a great number of desirable properties that include low density, resistance at high temperature, stiffness, oxidation and sulfidation resistance [1, 2]. However, these intermetallics have limited applications because of its low ductility at room temperature and low impact failure resistance [3]. In order to reduce embrittlement mechanism and improve the ductility of these materials, methodologies have been developed, such as macro and micro alloying with elements that induce passivity that include (Ti, V, Nb, Ta, Cr, Mo, W, Si y Ni) [4].

Conventional ingot production route suffer of insufficient ductility at low temperature to allow adequate fabrication methods. Powder metallurgy is an alternative methodology where the molten metal is atomized by a gas

or liquid flow at elevated velocity in order to produce a rapid solidified powder that can be subsequently consolidated. The "chill block melt spinning" technique [5] is a convenient method employed to study the potential of rapid solidification where little amounts of alloy are prepared and converted to metallic ribbons under controlled atmosphere with cooling rates of about 10^6 K/s. The production of intermetallics by rapid solidification has the following potential advantages: Refinement of microstructure, so that any composition segregation occurs only through short distances needing short thermal treatments to achieve uniformity; The potential for extend solid solubility of ternary elements beyond equilibrium compositions.

MATERIALS AND METHODS

The alloy Fe40Al-5Cr (at. %) was produced by vacuum induction melting by pouring the liquid melt into a rectangular cooper mold. The produced ingot was crushed to form coarse particles, which were re-melted in a quartz tube nozzle and then rapidly solidified to room temperature using a melt spinning apparatus under argon atmosphere. The melt spinning experiments were performed under an argon atmosphere, the quartz crucible has showed a 1 mm orifice diameter, employing an eject Ar pressure of 4 Psi, with a rotating cooper wheel speeds of 12 and 20 ms^{-1} that produced cooling rates of 0.715 x 10^6 and 1.38 x 10^6 K/s respectively.

Metallographic preparation of specimens was performed by grinding the specimens from 240 to 1500 grit paper and polished with 1 μm alumina powder. Then, the as spun ribbons were etched with a Keller reagent in order to observe grain size and morphology, photomicrographs of representative areas were obtained with an Olympus-zeiss optical microscope, with an interface to a PC equipped with image analyzer software (IMAGE PROPLUS). The grain size was determined by linear intercept method.

Microstructural characterization of intermetallic compounds was also carried out by scanning electron microscopy in order to perform punctual and scan line chemical analyses, also to observe surface meso pores formed by vacancy clustering.

X-ray diffraction analyses were performed to determine crystal structure, lattice constants, phases and orientation of specimens. The samples were scanned with a radiation filter CuK_α with a wavelength of λ = 1.5418 Å, employing a step of 0.02° / 0.6 s, in a range of (20 to 120° of 2θ). These analyses were carried out in a Siemens D5000 difractometer (30 kv, 20 mA). Lattice parameters were determined by least squares method.

Transmission Electron Microscopy (TEM) was employed to confirm crystal structure and also to characterize nano pores.

RESULTS AND DISCUSSION.

Figure 1 (a) and (b) shows melt-spun Fe40Al5Cr (at. %) ribbons produced at 12 and 20 m/s. Where the melt spun ribbon produced at 12 (m/s) exhibit three types of grains (chill, columnar and equiaxial). While the other ribbon produced at 20 m/s showed only equiaxial and columnar grains. The size of equiaxial grains of this ternary ribbons, exhibited a decrease from 24.7 μm to 6.61 μm as the wheel speed increased from 12 to 20 m/s. Besides, the thickness of the ribbons underwent a considerable reduction from 75.9 to 25.5 μm.

A line scanning chemical analysis performed in as spun Fe40Al5Cr (at. %) ribbons along a length of 83 μm (4 grains) revealed a uniform distribution of Al, Cr and Fe along the line as is shown in figure 1 (d). Thus it is evident that Cr is uniformly distributed in FeAl matrix. So it can be deduced that Cr still remains in solid solution after the rapid solidification. Besides, it is well known that rapid solidification processing produces an extended solid solubility of ternary element beyond equilibrium compositions [5].

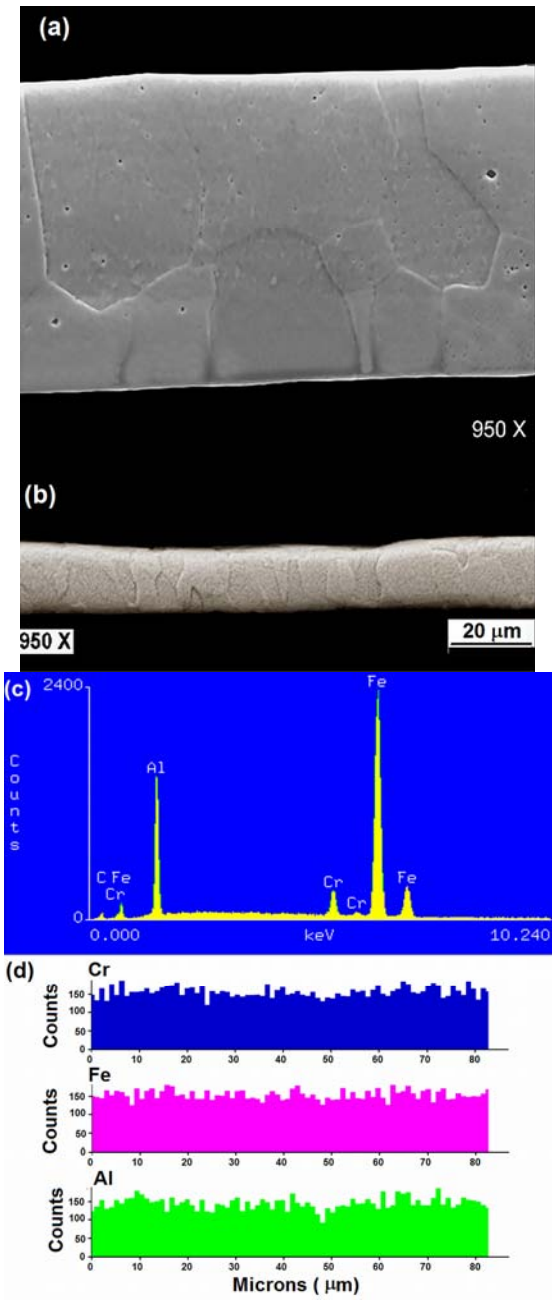

Fig. 1 Scanning electron micrographs of melt spun Fe40Al5Cr (at. %) ribbons, produced at: a) 12 m/s and b) 20 m/s, c) point chemical analysis and, d) scan line chemical analysis along a line of 83 µm (4 grains) performed both in melt spun Fe40Al5Cr (at. %) ribbon.

Figure 2 shows XRD profiles of melt-spun Fe40Al5Cr (at. %), where fundamental and super lattice peaks are shown, indicating that Cr does not modify the B2 ordered structure independently of wheel speed or cooling rate employed to produce the ribbons. Besides, additional peaks pertaining to second phases were not detected, indicating also that Cr enters in solid solution in the intermetallic matrix. Ternary FeAlCr ribbons exhibit all diffraction peaks without a notorious preferential orientation, related to a microstructure refinement.

In the same way, A. Argawal et. al. [4] observed that Cr and Ti additions were completely dissolved in a Fe_3Al intermetallic matrix, without forming second phases or precipitates.

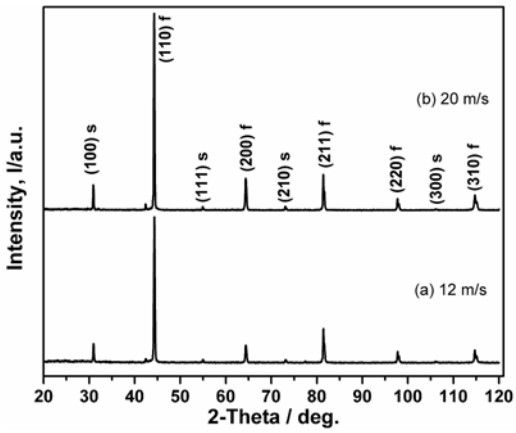

Fig. 2 X-ray diffraction patterns of melt spun Fe40Al5Cr (% at.) produced at (a) 12 m/s and (b) 20 m/s.

The lattice constants that correspond to the crystal structure B2 type of Fe40Al5Cr (at. %) ribbons produced at the wheel speeds of 12 and 20 m/s, resulted equal to 2.8898 and 2.893 Å respectively. These lattice constants present minor differences in comparison to those reported in binary Fe40Al (at. %) rapid solidified ribbons. R. A. Buckley et. al. [6] reported a lattice constant of 2.9 Å in one alloy Fe40Al (at. %) produced by rapid solidification. The values of lattice parameters of ternary FeAlCr ribbons reported in present work are smaller than those reported by R. A. Buckley et. al. [6], this is attributed to occupation of Cr in the vacancy sites that are produced during rapid solidification, producing thus a decrease of vacancy content and a subsequent decrease of lattice parameter in ternary FeAlCr ribbons.

Figure 3 a) shows an electron micrograph at higher magnification corresponding to Fe40Al5Cr (% at.) that was produced at a tangential wheel speed of 12 m/s, where triangular mesopores are shown with a pore size fluctuating between 240 to 940 nm. Figure 3 b) shows a

pore surface size distribution, where it can be observed that the highest percent of surface area, fluctuates among $60x10^3$ nm^2 to $100x10^3$nm^2. Several authors have reported pore or mes opore formation inside solid B2 intermetallics, such as NiAl, CoGa [7] and FeAl [8]. Pore formation inside melt-spun ribbons, can be originated from vacancy clusters generated during rapid solidification. Vacancy clusters must be stable even at high temperatures since the vacancy binding energy is positive in FeAl [8].

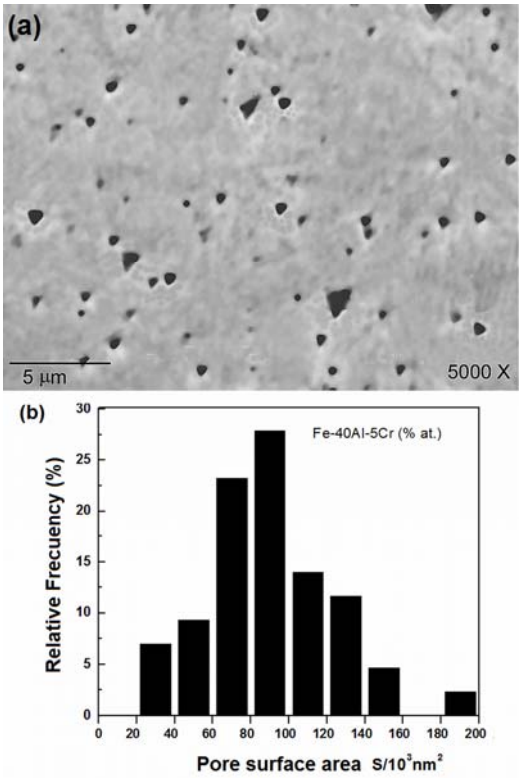

Fig. 3 (a) Triangular nano porosity observed in surface of melt spun Fe40Al5Cr ribbons produced at 12 m/s, (b) pore surface size distribution obtained by image analysis.

Figure 4 a) shows features observed inside grains of FeAl-B2 matrix that correspond to nano pores formation by vacancy clustering. The image of these nano pores was formed by the contrast originated from the thickness difference between pores and matrix. Figure 4 b) is an electron diffraction pattern viewed from [001] zone axis which was obtained from the matrix (next to the biggest nanopore). Besides, figure 4 a) also exhibits straight border dislocations with burgers vectors parallel to <001>. One side of the rectangles showed in figure 4 (a) is parallel to (100) plane and the other side is parallel to (010) plane.

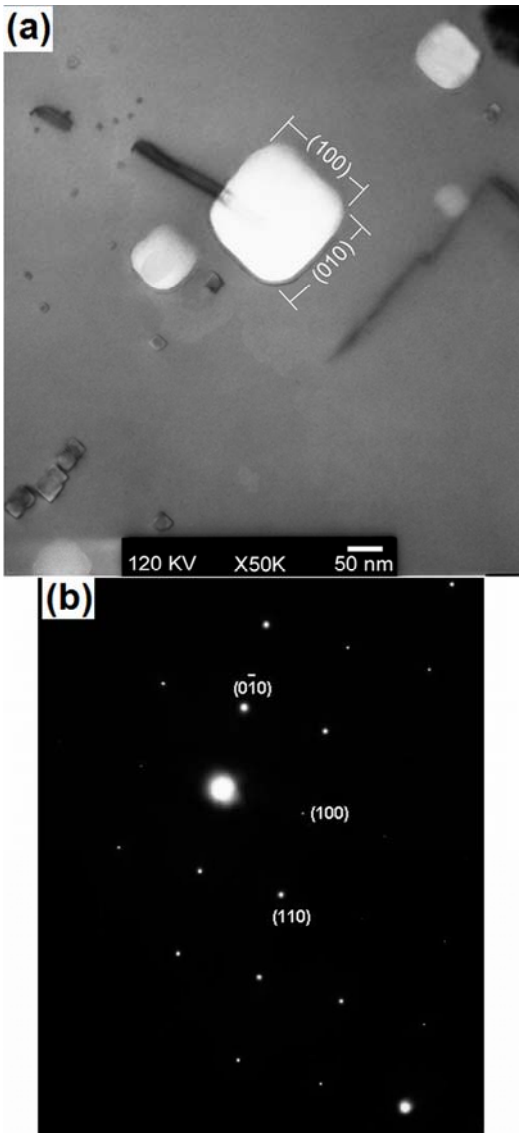

Fig. 4 Transmision electron micrograph of melt spun Fe40Al5Cr (at. %) ribbon produced at 20 ms^{-1} wheel speed. (a) micropore formation by vacancy clustering together with <001> dislocations, (b) electron difraction pattern viewed from [001] zone axis corresponding to a zone near to the biggest nanopore.

CONCLUSIONS

The grain size and ribbon thickness showed a considerable reduction as the wheel speed was increased from 12 to 20 ms^{-1}.

XRD analyses revealed that Cr element remained in solid solution, besides chromium is uniformly distributed among the whole melt spun FeAlCr ribbon.

Lattice constants of ternary FeAlCr ribbons resulted in lower values than those reported in rapid solidified binary Fe40Al (at. %) ribbons, this behavior suggests that chromium occupies the great quantity of vacancy sites that are produced upon rapid solidification processing.

Quasi rectangular nano pores were formed in the ternary FeAlCr ribbons, with one side parallel to (100) plane and the other side parallel to (010) plane.

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