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Formation of Ti-matrix Composites for Lightweight Structural Components by Mechanical Alloying and Subsequent Annealing

B. Trindade[a], M. Caetano[b], N. Duarte[c]

ICEMS, Mechanical Department of Coimbra University, Polo II, Coimbra, Portugal

[a] bruno.trindade@dem.uc.pt, [b]marcocaetano@sapo.pt, [c]nelson.duarte@dem.uc.pt

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Abstract. In this work, a lightweight Ti-10Mg-5Al (wt. %) alloy for structural components was produced by mechanical alloying. A metastable α-Ti(Al,Mg) solid solution was obtained after 50h of milling. Diffraction peaks ascribed to the Mg phase were detected in the XRD pattern of the sample heat treated at 600ºC. This phase tends to oxidize with the increase of temperature giving rise to MgO. The structure of the mechanically alloyed and 900ºC heat treated sample consisted of a Ti(Al) solid solution with dispersed MgO particles in the matrix. Hardness and Young's modulus values obtained from ultramicrohardness tests confirm the strength improvement of the Ti-based alloy due to the MgO reinforcement.

Introduction

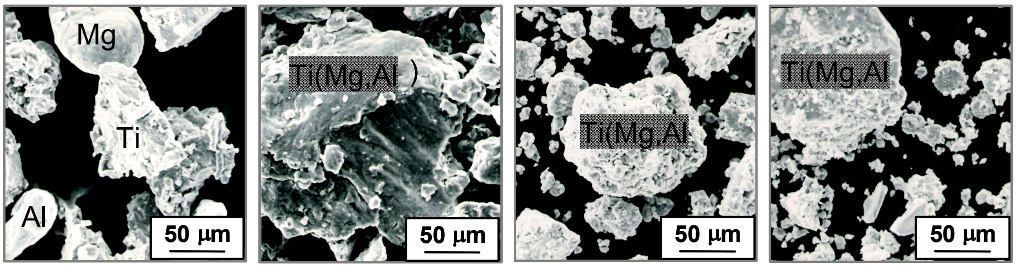
Titanium-based alloys have been attractive materials for different structural applications due to their good creep performance, high strength and toughness as well as excellent corrosion resistance. In last years, many attempts have been made to increase specific strength in order to obtain materials suitable for aerospace applications. This goal has been achieved by reducing the density and increasing the strength of the Ti-alloys. There are some elements that have been used for this purpose, e.g. magnesium, aluminum and silicon [1,2,3]. Titanium and magnesium are immiscible in the solid state and do not form intermetallic phases. On the contrary, aluminium and silicon form intermetallic compounds with titanium, which are responsible for the increase of strength. In a previous work on the production of Ti-based alloys by mechanical alloying (MA) with and without subsequent hot isostatic pressure (HIP), it was confirmed that the addition of in certain percentages of Mg and Si to Ti leads to the formation of lightweight Ti-matrix composites reinforced with titanium silicides (mainly Ti_5Si_3) [4]. In the present investigation we attempted to synthesize a Ti-10Mg-5Al (wt. %) alloy with the objective of producing a two phase material formed by a lightweight metallic matrix (for ductile phase toughening) and a discontinuous reinforcement intermetallic phase (for strength increase by impeding the dislocations motion).

Experimental Details

Ti-10Mg-5Al (wt. %) mixtures, which weighted 24.5g, were mechanically alloyed in a planetary ball mill. The ball to powder weight ratio was 20:1 and the rotation speed employed was 300 rpm. Hardened steel vials and balls sealed under argon atmosphere were used. Small quantities of the powder were withdrawn after 5, 10, 25 and final milling was performed up to 50h of milling. The powders were analyzed by means of electron probe microanalysis (EPMA), x-ray diffraction (XRD) and scanning electron microscopy (SEM). The thermal behavior of the 50h milled sample was analyzed by means of simultaneous differential scanning calorimetry and thermal gravimetry analysis (DTA/TG). Based on the DSC results the samples were heat-treated at 600 and 900ºC/1h in an argon atmosphere. The indentation Young modulus was calculated with a 100 mN load, according to the method described in [5]. Ultramicrohardness measurements were carried out on flat polished surfaces using a Vickers diamond indenter under the same loading conditions. Particle size distributions were determined by laser scattering (Coulter LS 130 equipment).

Results and Discussion

Elemental powders of Ti, Mg and Al were mixed in the desired composition (Ti-10Mg-5Al). Scanning electron micrographs of the mixture, as-blended and after different periods of MA, are shown in Fig.1.



a) b) c) d)

Figure 1 – Scanning electron micrographs of powder particles: (a) as-blended, (b) 5h, (c) 25h and (d) 50h of mechanical alloying

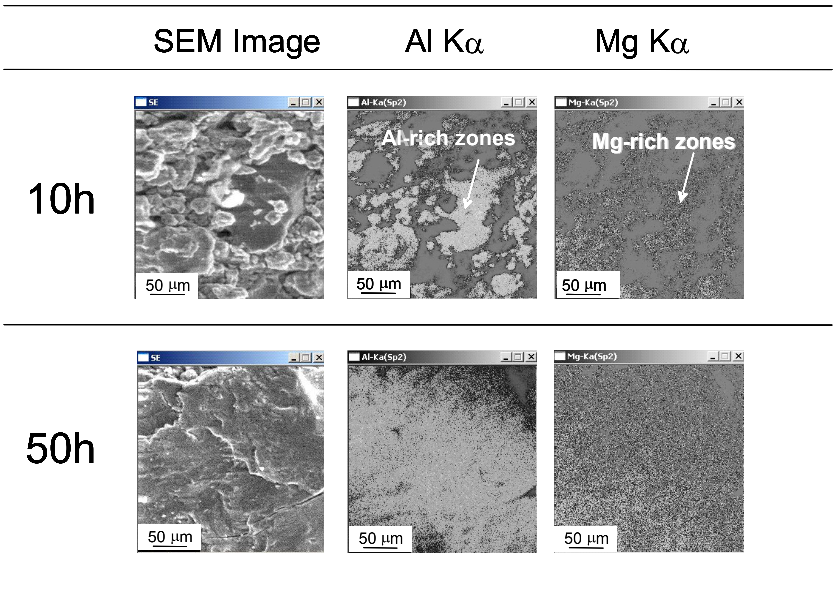


Figure 2 – SEM images and elemental X-ray maps (Al-Kα and Mg-Kα) of the Ti-10Mg-5Al mixture after 10 and 50h of milling.

At the beginning of the synthesis process, the particles are soft and their tendency to weld together is high, leading to an increase in particle size (Fig. 1b). A broad range of particle sizes develops, with some as large as three times bigger than the starting particles. With continued deformation the particles get harder (work hardened) and fracture occurs. At this stage the tendency to fracture predominates over cold welding.

Fig.2 shows SEM images and elemental X-ray maps for elements Al and Ti. As can be seen, zones rich in aluminum and magnesium still exists in the mixture after 10h of milling. For 50h, the mixture can be considered as chemically homogeneous at a micrometer scale.

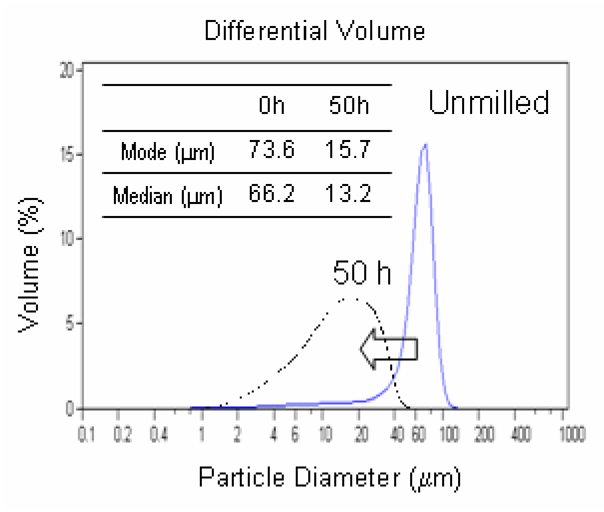


Figure 3 – Particle size distributions of the as-blended and 50 h milled mixtures

The particle size distribution after milling is shown in Fig.3. A broad distribution of particles was obtained after 50h of milling, characterized by median and mode values much lower than those of the as-blended mixture XRD analysis of the mille powder (Fig. 4) revealed the formation of a Ti(Mg,Al) solid solution. In fact, the peaks of the hcp-Mg and fcc-Al phases are no longer visible after 5h of milling, which means that these elements substitute for Ti in the α Ti lattice giving rise to an α-Ti(Mg,Al) solid solution. The incorporation of these elements in solid solution leads to an increase of the lattice volume i.e. increase of *a* and *c* lattice parameters.

In fact, values of 35.54 and 35.74 $Å^3$ were obtained for the volume of the α-Ti lattice before and after 50h milling, respectively. This is in accordance with the atomic radius of the metallic elements as contrarily to aluminum, the magnesium atom is bigger than the titanium atom (r_{at} Ti = 1.47Å, r_{at} Mg = 1.60Å and r_{at} Al = 1.43Å). This means that these elements play different roles in the volume of the α-Ti lattice, the influence of magnesium being of great magnitude due to its higher percentage in the alloy. The grain size of the α-Ti(Mg,Al) phase milled for 50 h was measured by the Williamson-Hall method [6]. This method considers that the final width of an X-ray line is the result of its half height intensity, the residual strain broadening and the instrumental broadening associated with the X-ray spectrometer. The value obtained (6 nm) is typical of nanostructured materials.

The 50h milled mixture was submitted to DSC and TG simultaneous run followed by XRD analysis at room temperature after cooling. The results obtained are presented in Figs. 5a and b. Regarding the DSC curve, an endothermic peak could be detected with a minimum heat flow at about 645ºC (Tonset = 630ºC). The TG curve reveals a mass gain for temperatures higher than 400ºC. Comparing the XRD pattern of the mixture heat up to 900ºC with that of the as-mechanically alloyed mixture, the only difference to notice is the formation of MgO. No Ti aluminides peaks were detected in the XRD pattern. EPMA analysis of the heat-treated mixture revealed, in some particles, a significant decrease of the magnesium amount. It should be noted that the boiling point of magnesium is 1090ºC (190ºC over the maximum temperature achieved in the DSC + TG run), which might explain this loss of magnesium. Taking into account these results, the DSC endothermic peak might be ascribed to the melting of and Mg (660ºC). In spite of the lower melting point of aluminum (650ºC), it is not probable that its melting had occurred since the solubility of this element in the α-Ti phase is 10 wt.% at 900ºC, which is higher than its percentage in the mixture. Concerning the TG curve the gain of mass must be attributed to the oxygen incorporation in the α-Ti lattice as well as oxidation of Mg.

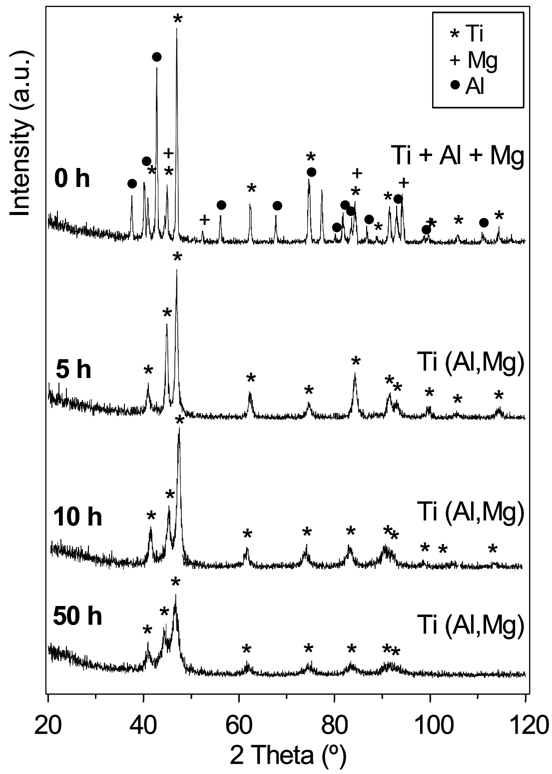
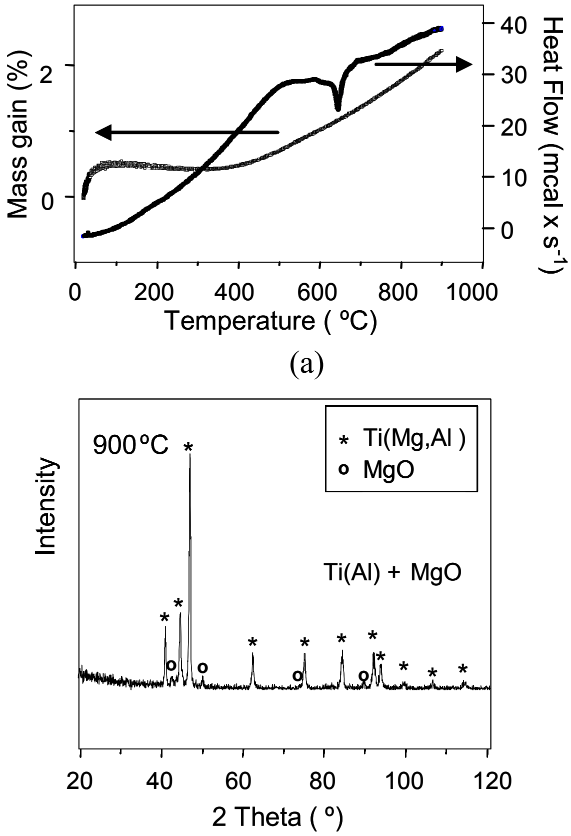


Figure 4- XRD patterns of the Ti-10Mg-5Al mixture as a function of milling time.



(b)

Figure 5 (a) DSC and TG curve and (b) XRD pattern of the Ti-10Mg-5Al mixture.

Hardness and Young´s modulus of the mechanically alloyed and heat-treated samples were determined by hardness tests. An average hardness value of 4.5 ± 0.4 GPa was obtained which is in accordance with the value measured by Wilkes et al. [7] in a Ti-9 wt.% Mg milled for 48h but much higher than the value reported in the literature for Ti [8]. This hardness increase is due to the presence of a second phase (MgO) finely dispersed in the matrix as well as to the incorporation of some oxygen in the α-Ti matrix. However, this incorporation must be of slight magnitude since a dramatic increase in hardness (> 7 GPa) would be expect for high oxygen concentrations in the α-Ti lattice [7] which wasn't the case of the present study. Moreover, the presence of aluminum in the α-Ti lattice might also contribute for the increase in hardness. The Young´s modulus of the heat-treated particles was calculated from the indentation curve. The value obtained (70 ± 0.4) is not so high as expected and is similar to the one of titanium reported in the literature. It is likely that the high concentration of pores existing in the green compact affected the measurements.

Conclusions

A lightweight Ti-10Mg-5Al (wt. %) alloy was produced by mechanical alloying. During milling the elements Al and Mg substitute for titanium in the α-Ti lattice. A metastable α-Ti(Al,Mg) solid solution was obtained after 50h of milling. The DSC curve of the mechanically alloyed mixture showed an endothermic peak at about 650ºC corresponding to the melting of Mg. This element reacts with oxygen at high temperature to give rise to MgO. The structure of the mechanically alloyed and 900ºC heat treated sample consisted of a Ti(Al) solid solution with a fine dispersion of MgO particles in the matrix. Hardness and Young's modulus values obtained from ultramicrohardness tests confirmed the strength improvement of the Ti-based alloy by the MgO reinforcement.

References

[1] L. Dias, C. Coelho, B. Trindade and F.H. Froes: Key Engineering Materials, vol. 230-232 (2002), p.283.
[2] J.B. Zhou and K.P. Rao: J. Alloys and Compd. vol. 384 (2004) p. 125.
[3] F. Simões, B. Trindade, J.A. Santos and F.H. Froes: Materials Technology vol. 18/2 (2003), p. 98.
[4] L. Dias, C. Coelho, B. Trindade and F.H. Froes: Mater. Sci. Eng. A vol. 364 (2004), p.273.
[5] J.M. Antunes, A. Cavaleiro, L.F. Menezes, M.I. Simões and J.V. Fernandes: Surf. Coat. Technol. vol. 149 (2002), p.27.
[6] G. Williamson and W.H. Hall: Acta Met. vol. 1 (1953), p.22.
[7] D.M.J. Wilkes, P.S. Goodwin, C.M. Ward-Close, K. Bagnall and J. Steeds: Materials Letters vol. 27 (1996), p.47.