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Development of new Al-based nanocomposites by mechanical alloying

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Abstract

Al-based binary (Al–Mg) and ternary (Al–Mg–Zr) elemental powder mixtures were mechanically alloyed to develop new Al–Mg–Zr nanocomposite materials. The phase evolution was studied in the as-milled and heat-treated powders by XRD and TEM/EDS analyses. For the binary Al–Mg alloy, the predominant phase was an Al(Mg) solid solution (SS) and an amorphous phase was not possible to be synthesized. Upon adding 5 at.% Zr to the Al–10Mg blended powder, some free Mg was present in addition to the formation of an Al(Zr,Mg)SS, which transformed to the Al_3Zr intermetallic after annealing. When the Zr content was increased a nanocomposite of a solid solution and an intermetallic was obtained with considerable improvement in terms of structural stability and hardness. The presence of an oxide phase at 35% Zr might be responsible for the increased hardness in this particular alloy.

Keywords: Nanocomposites; Mechanical alloying; X-ray diffraction; Transmission electron microscopy; Intermetallics

1. Introduction

The interest in developing new Al–Mg alloys has grown largely in the past years as part of the ongoing search for new advanced materials to meet the challenges and demands for higher performance alloys. Al–Mg alloys are interesting because of their high specific strengths (strength to weight ratio) in addition to good corrosion resistance granted by an adequate amount of Al. They are used in many applications which include automotive, aerospace and many other structural applications [1].

Al–Mg alloys can be processed by either equilibrium or non-equilibrium processing routes. Non-equilibrium techniques are favored as they yield improved materials with metastable structures, which can be beneficial in producing alloys with desired mechanical and physical properties [2,3]. Non-equilibrium techniques differ in the degree of departure from the equilibrium and the state of the produced material. Materials can be processed by gas condensation (GC), rapid solidification (RS) or mechanical alloying (MA); in MA no melting of the materials is required and alloying occurs in solid state. MA is mainly used due to its simplicity, versatility, and its economic viability [4]. It can also be used for the fabrication of novel alloys and allows for chemical reactions at low temperatures (mechanochemistry) due to the enhanced diffusivity, which is promoted by the introduction of structural defects [5]. Alloying is achieved by continuous cold-welding and fracturing of the powder material due to the transferred kinetic energy from the milling media.

On the other hand, the interest in nanocomposites has grown since the introduction of the Al–Y–Ni–M (M = Fe, Mn) nanocomposites comprising fcc-Al nano-dispersed particles in an Al-based amorphous alloy [6]. They were developed by RS showing improved tensile and hardness properties compared to their fully amorphous counterparts. Therefore, it will be interesting to develop nanocomposite materials with improved properties based on Al and Mg to add to the well-known advantages of these light weight elements. To promote the development of an amorphous matrix, Zr is chosen in the synthesis of the alloys, in addition to its ability to enhance fatigue corrosion cracking resistance, to prevent or minimize natural aging and to promote superplastic behavior.

Research on MAed and RSed Al–Mg alloys has shown that metastable phases can be produced [7–9] and solid solubility limits can be extended [2,7,10] beyond the equilibrium values. Moreover, research has been carried out to study the departure of the Al–Zr system from equilibrium by MA processing [3,11,12]. It has been shown that amorphous phase can be developed on ratios that depend mainly on Zr concentrations on alloys. With respect to Al–Mg–Zr system, only limited work has been done

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using liquid processing techniques [13,14]. On the other hand, MA has proved effective to produce Al–Mg–Zr alloys with Zr content varying from low percentages 0.6% Zr [15] to 5 at.% Zr [16] and 6 at.% Zr [17].

Therefore, the aim of this research was to investigate the possibility of fabricating Al-based nanocomposites with improved mechanical properties. The Zr content was varied from 5 to 35 at.% in Al–10 at.% Mg alloys to study the role of Zr in refining the microstructure and in promoting the nanocomposite formation.

2. Experimental procedure

Elemental powders of Mg, Al and Zr (purity >99%, −325 mesh provided by Alfa Aesar) were used as starting materials to prepare alloys of composition $Al_{90-x}Mg_{10}Z_x$ ($x = 0$, 5, 20, 35 at.%). The powders were loaded in a stainless steel vial and milled in a high-energy Spex 8000M mill under Ar atmosphere to reduce contamination. Stainless steel balls were used and the ball-to-powder weight ratio (BPR) was fixed at 10. The milling time was maintained at 9 h in all the experiments despite the fact that steady-state condition is reached at different times for different compositions. The milling was periodically stopped every 1.5 h for 3 h to avoid temperature build-up in the milling vial. Furthermore, to eliminate the accumulation of unprocessed powders on the internal walls, the vial was opened every 3 h of milling and the deposited powders were scraped out from the vial walls. Annealing was carried out on as-milled powders at 400 °C for 1 h to study phase stability and to examine possible transformations upon heating. The phase characterization of the resulting powders was carried out by XRD with Cu Kα radiation and with a 200 kV TEM/EDS. TEM studies were particularly useful to characterize the nanostructures obtained in the resulting alloys in terms of particle size and crystal structure. Additionally, the Scherrer equation [18] was used to calculate the crystallite size along with direct TEM observations, in order to assess the use of this procedure in measuring the crystallite size from XRD data. The resulting powders were cold pressed at 1 GPa for 10 min with the compaction die being kept inside the hot-press for 20 min at 400 °C, followed by pressing at 70 MPa for 10 min. This procedure was performed to facilitate hardness measurements, taken using a Clark® microhardness tester.

3. Results and discussion

3.1. Phase evolution studied by XRD

X-ray diffraction patterns of the resulting as-milled and annealed powder alloys are presented in Figs. 1 and 2, respectively. As mentioned previously, the Mg content was fixed at 10 at.% for all alloys, whereas the concentration of Zr was varied to produce one binary and three ternary alloys. For the binary alloy, the XRD plot shows primarily the presence of an Al(Mg) solid solution (SS). This was inferred by the clear shift of the α-Al peaks toward lower angles, due to the dissolution of the larger size Mg atoms in the Al matrix. Broadening of the diffraction peaks is another feature that can be noticed and this is indicative of reduction in crystallite size and accumulation of strain in the material. However, neither the presence of Al–Mg intermetallics nor unalloyed Mg was revealed from the diffraction pattern, which suggests only complete solid solubility. These results agreed with previous findings [9] in which only α-Al solid solution was present in alloys up to 30 at.% Mg. Furthermore, upon annealing the Al(Mg) SS was the only phase present; however, peaks were slightly narrower due to stress relaxation and grain coarsening.

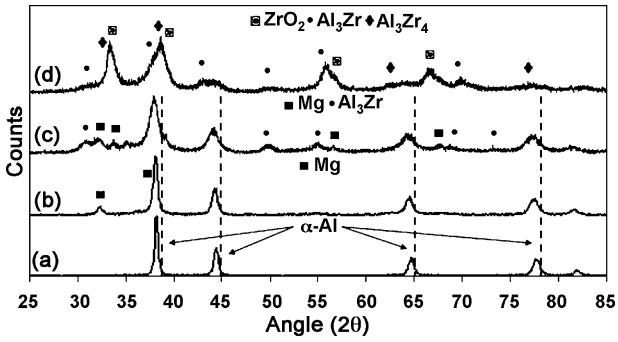

Fig. 1. X-ray diffraction patterns of resulting as-milled powders: (a) 10Mg–90Al, (b) 10Mg–85Al–5Zr, (c) 10Mg–70Al–20Zr, and (d) 10Mg–55Al–35Zr.

To study the phase evolution further and the possibility of forming nanocomposites as a result of adding a glass-forming ternary element, a series of alloys with different Zr concentrations was prepared. In the 5 at.% Zr alloy, some free Mg was present along with the Al(Mg)SS, suggesting that the solid solubility of Mg in Al was less in the presence of Zr. This might be due to the substitution of some Zr for Mg and the formation of an Al(Mg,Zr) SS. Upon annealing, however, traces of Al_3Zr were detected with a cubic ordered structure Ll_2 [19]. It is considered as a high temperature phase that is present in a metastable state at room temperature. This phase can be synthesized directly by MA in higher Zr contents or by annealing the solid solution containing Zr at low Zr contents. This resulted from annealing and the high affinity between Al and Zr. An additional advantage of the non-equilibrium processes is that it is possible to retain the Al_3Zr phase in the cubic ordered structure $L1_2$ rather than the equilibrium tetragonal DO_{23}, which causes embrittlement of the alloy

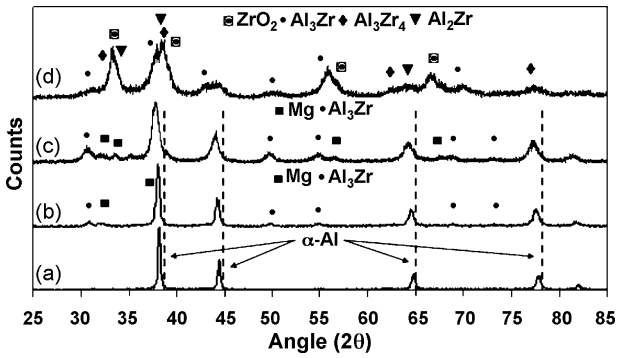

Fig. 2. X-ray diffraction patterns of alloy powders annealed at 400 °C: (a) 10Mg–90Al, (b) 10Mg–85Al–5Zr, (c) 10Mg–70Al–20Zr, and (d) 10Mg–55Al–35Zr.