Materials Science & Engineering A 595 (2014) 124–130

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

Nanostructured Al–Zn–Mg–Cu–Zr alloy prepared by mechanical alloying followed by hot pressing

Amin Azimi, Ali Shokuhfar, Ashkan Zolriasatein *

Advanced Materials and Nanotechnology Research Laboratory, Faculty of Mechanical Engineering, K. N. Toosi University of Technology, P.O. Box 19395-1999, Tehran, Iran

ARTICLE INFO

Article history:
Received 27 August 2013
Received in revised form
31 October 2013
Accepted 29 November 2013
Available online 7 December 2013

Keywords:
Nanostructured Al–Zn–Mg–Cu–Zr alloy
Mechanical alloying
Hot pressing
Microstructure
Mechanical properties

ABSTRACT

Nanostructured Al–7.8 wt% Zn–2.6 wt% Mg–2 wt% Cu–0.1 wt% Zr alloy was mechanically alloyed (MA) from elemental powders and consolidated by hot press technique. The effect of the milling time and hot pressing process on microstructure was investigated by means of X-ray diffraction measurements (XRD) and analytical and scanning electron microscopy (SEM). Furthermore mechanical properties of samples with different MA time as well as pure aluminum were investigated by microhardness and compression tests. The results show that an Al–Zn–Mg–Cu–Zr homogenous supersaturated solid solution with a crystallite size of 27 nm was obtained after 40 h of milling time. Microstructure refinement and morphological changes of powders from flake to spherical shape were observed by increasing milling time. Phase and microstructural characterization of high density bulk nanostructured samples revealed that increasing milling time up to 40 h leads to formation of $MgZn_2$ precipitation in the alloy matrix. With increasing milling time, density of the samples and crystalline size decrease. Significant enhancement of hardness and compressive strength is observed in the aluminum alloy by increasing milling time up to 40 h which is much higher than pure aluminum. Crystallite size refinement in pure aluminum samples from micro- to nanoscales resulted in 107% and 100% improvement in compressive strength and hardness, respectively. Furthermore the compressive strength and hardness of Al–Zn–Mg–Cu–Zr alloy nanostructured samples increased to 179% and 172%, respectively, compared to nanostructured pure Al, which was produced as reference specimen. 40 h of MA was the optimum case for preparing such an Al alloy and more milling up to 50 h led to deterioration of mechanical properties.

1. Introduction

The 7xxx series (Al–Zn–Mg–Cu) aluminum alloys are well applied in an aeronautical field due to their high specific strength, lightweight, corrosion resistance and other features beneficial to superior performance [1]. Zinc and magnesium both have great solid solubility in aluminum and develop usually high precipitation–hardening capability. This is associated with forming fine precipitates of metastable η′-phase ($MgZn_2$). After extensive search, Al7075 alloy was introduced in 1943. This alloy is still successfully used in high strength structural materials and in subsequent years, alloys such as 7178 were developed by improving 7075 [2].

Al7068 alloy (Table 1) is one of the high strength 7xxx series aluminum alloys. These percentages of alloying elements provide the highest mechanical strength aluminum alloys which can be produced by ingot metallurgy. This alloy was developed in the mid 1990s by Kaiser Aluminum, and exclusively stocked and supplied in Europe by Advanced Metals International. 7068 Alloy was designed as a higher strength alternative to 7075 which provides the highest mechanical strength of all aluminum alloys and matching that of certain steels. This outstanding alloy combines yield strength (up to over 30% greater than that of 7075 alloy) and good ductility with corrosion resistance similar to 7075. Also mechanical properties of 7068 retained at elevated temperatures better than 7075. Finally 7068 significantly reduces the weight/cross section in transportation sectors [3].

Addition of copper and zirconium can improve the properties of 7xxx series alloys by solid solution strengthening. On the other hand, they can increase the stability of GP zones and contribute to $MgZn_2$ precipitate formation in the alloy matrix. In higher amounts, zirconium leads to formation of metastable Al_3Zr phase which is coherent with the matrix. This phase stabilizes the grain structure and prevents recrystallization [4,5].

In order to satisfy the requirements of aerospace industries for structural applications, increasing strength of these alloys is worthy to pay attention to. Reducing crystallite size to nanoscale, in other words, formation of nanocrystalline materials has attracted

* Corresponding author. Tel.: +98 21 84063304; fax: +98 21 88677274.
E-mail addresses: a.zolriasatein@dena.kntu.ac.ir, ashkan.zolriasatein@gmail.com (A. Zolriasatein).

0921-5093/$ - see front matter © 2013 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.msea.2013.11.094

Table 1
Chemical composition of Al7068 alloy (wt%).

Zn	Mg	Cu	Zr	Al
7.8	2.6	2	0.1	Balance

Table 2
Characteristics of initial powders.

Alloy elements	Al	Zn	Mg	Cu	Zr
Particle size (μm)	40–63	10–20	40–63	10–20	20–45
Purity (%)	99.9	99.98	99.98	99.98	99.9

considerable interest in recent years due to improving mechanical properties [6–11].

There are novel techniques such as mechanical alloying (MA) used for production of nanocrystalline aluminum alloys. Mechanical alloying is a solid-state powder processing technique covering the repetitive three mechanisms of severe plastic deformation, cold welding and fracturing of powder particle. Formation of the homogenous solid solutions with extended solubility limits from initial elemental powders can be attributed to the non-equilibrium nature of this process. Furthermore, in recent studies, it has been understood that existence of zirconium in this alloys can lead to formation of more homogeneous solid solution [6–11].

Many different types of existing nanostructured materials such as nanocomposites, nanocrystalline metals or intermetallic compounds can be synthesized by mechanical alloying which exhibit better properties and higher performance than conventional coarse-grain materials [12–16].

There were many investigations about fabrication and characterization of nanostructured aluminum alloys by MA. It was revealed that the production of nanocrystalline structures with a large volume fraction of atoms in high energy grain boundaries contribute in formation of supersaturated solid solutions. The mentioned phenomena result in enhanced diffusion and solid solubility. Consolidation of these powders to bulk nanocrystalline materials is usually carried out by hot isostatic pressing [5,17].

Synthesis and characterization of nanostructured aluminum alloys by mechanical alloying such as 6061, 2024, 7050 and 7075 were investigated in recent studies [18–23], and to the best of the authors' knowledge there is no research on the synthesis of nanostructured Al7068 alloy by mechanical alloying.

The aim of the present study is to prepare nanostructured Al–7.8 wt% Zn–2.6 wt% Mg–2 wt% Cu–0.1 wt% Zr which matches the composition of 7068 aluminum alloy by mechanical alloying of mixed element powders. In the next step, production of bulk nanostructured alloys by the hot pressing method and then studying the effect of the milling and hot pressing on the microstructure of powders and bulk samples are considered. The density, Vickers microhardness and compressive strength of the Al alloy milled for different time were compared with consolidated as-received and nanostructured pure Al samples.

2. Experimental

Mixture of Al, Zn, Mg, Cu and Zr powders was applied to fabrication of nanostructured sample with an exact chemical composition of 7068 aluminum alloy by the powder metallurgy (PM) technique which was named PM-Al7068 alloy in this research. Specifications (purity and particle size) of initial powders are summarized in Table 2. MA was performed in a planetary high-energy ball mill Fritsch – P6 under an argon atmosphere using 10 mm diameter stainless steel balls. A ball-powder weight ratio of 15:1 and rotation speed of 250 rpm were used for ball milling. Moreover 0.5 wt% stearic acid was used as a process control agent (PCA).The milled powders were hot pressed in an uniaxial die under 500 MPa at 380 °C for 30 min. As received aluminum and 40 h-milled pure Al powders were consolidated under this same condition. The structural changes of powders during the milling and consolidated nanostructured PM-Al7068 powders were studied by means of X-ray diffraction measurement (XRD) using a Philips diffractometer (40 kV) with Cu-Kα radiation (λ=0.15406 nm). The XRD patterns were collected over 2θ range of 20–100° with step size of 0.031°. The Al crystallite size (d) and lattice strain (ε) were determined from broadening of XRD peaks using the Williamson–Hall formula [24]:

$$\beta \cos \theta = \frac{k\lambda}{d} + 2\varepsilon \sin \theta$$

where β is the full-width at half-maximum (FWHM) of the diffraction peak, θ is Bragg diffraction angle, λ is the X-ray wave length, d is the crystallite size, ε is the lattice strain induced by mechanical deformation and k is a constant (0.9). Furthermore the lattice parameters of powders during the milling were obtained from each reflection. The Al lattice parameter was determined according to the method which is given in Ref. [25]. The microstructure of powder particles and hot pressed powders were characterized by scanning electron microscopy (SEM) using Philips XL30 combined with energy dispersive X-ray (EDX). Density values of compact samples were measured applying Archimedes and theoretical approaches. Microhardness measurements were carried out on the cross section of the bulk samples using a Vickers indenter at a load of 50 g and dwell time of 10 s and mean value of 5 measurements are reported.

Compression tests were performed on the cylindrical shape specimens (10 mm in diameter and 15 mm in height) at room temperature with strain rate of 1 mm/s by an universal testing machine according to ASTM E9-89a standard.

3. Result and discussion

3.1. Characterization of powder samples

Fig. 1 shows the XRD spectra of the milled powder elements of PM-Al7068 at different milling times. The diffraction peaks of Al, Zn, Mg, Cu and Zr confirm the presence of these elements in mixed powder (0 h milling). After 5 h of milling, broadening and

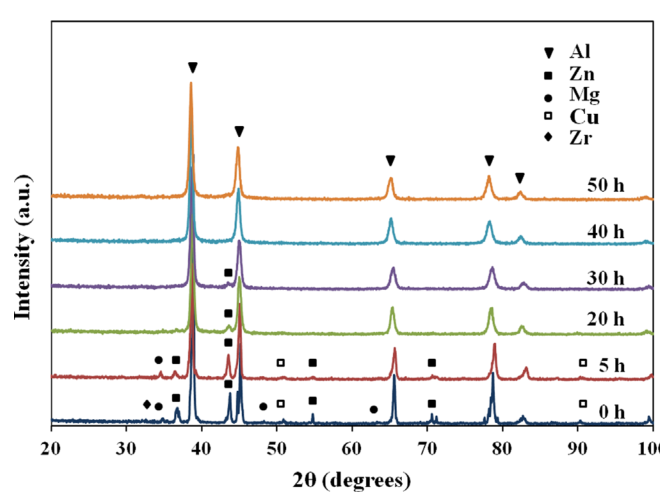

Fig. 1. X-ray diffraction pattern of PM-Al7068 milled powders for different milling time.