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A novel study on mechanically alloyed Al–Mg system by X-ray diffraction technique

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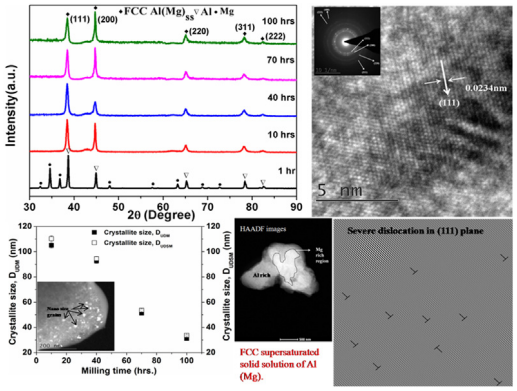
HIGHLIGHTS

- Fine grain supersaturated FCC Al(Mg) has been achieved by high energy ball milling.
- Dissolution of Mg atom has been revealed by determination of lattice parameter.
- High dislocation density was estimated from XRD technique during ball milling.
- Enhancement in stacking fault probability and dislocation density was observed.

GRAPHICAL ABSTRACT



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ABSTRACT

Ultra-fine grain supersaturated FCC Al (Mg) solid solution has been achieved by high energy ball milling of elemental Al and Mg. Dissolution of Mg atom has been revealed by determination of precise lattice parameter. Observation of high dislocation density ($\sim 4.5 \times 10^{15}$ m^{-2}) in (111) plane was manifest to severe plastic deformation during the mechanical alloying. Thermodynamic calculation by using extended Miedema model justifies the quantitative solubility measurement of Mg respective to grain size. Increase in stacking fault probability and dislocation density with milling time was also observed.

1. Introduction

Nanocrystalline Al–Mg system has great interest due to the beneficial features of high strength, good ductility and low density which make it more attractive for aerospace and automotive applications [1–4]. Among several synthesis processes, mechanical alloying (MA) is one of the solid state powders processing techniques which also of alloying results in nano-grain size. During ball milling, several collisions occur between powders with balls and the inner side of the milling vial. The basic mechanism of the milling process is severe plastic (SPD) through the fracture and cold welding of particles. Consequently, particles endure intermixing and interdiffusion, thus, yield supersaturated solid solution beyond the equilibrium solubility limit during MA [5–7]. X-ray diffraction (XRD) study was used for micro-structural characterization, crystallite size determination and defect structure analysis. During SPD several defects viz., dislocations, stacking faults induce into the structure of the materials. Such deformation mechanism plays an important role in structural evolution and fine grain formation [8,9]. Kenji Kaneko [10] showed up to ~40 nm crystallite size of

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Al–Mg alloy prepared by high-pressure torsion (HPT) where Mg was used more than 30 at. %. S. Scudino [11] suggests the lattice parameter of 4.07 A of the supersaturated solid solution of Al–Mg alloy. During MA, the solid solution of Mg increases and defects are introduced. Mg content plays an important role in the defect structure in Al–Mg system [12].

The present investigation is based on a detailed study of lattice parameter measurement followed by defect analyses through XRD. Moreover, the increase in lattice parameter further substantiates the dissolution of Mg atom into Al lattice.

2. Material and methods

Elemental Al and Mg powder (composition of Al-40 at. % Mg) were mechanically alloyed in Retsch PM200 planetary ball mill equipped with tungsten carbide balls and vials. The MA process was accomplished at ambient atmosphere with the ball (10 mm dia.) to powder weight ratio of 10:1 and milling speed of 300 rpm. The vial was filled one-third part of it by toluene which acted as a process control agent. XRD analysis was carried out using Philips PANalytical X-ray diffractometer equipped with the position-sensitive detector. Cu-K_α ($\lambda = 0.154078$ nm) radiation generated at 45 kV/40 mA with a step size of 0.05° and 1.35 s per step was used for diffraction. Crystallite size and micro-strain were determined by line broadening analysis considering all reflections in the 2θ range of 20°–90°. The instrumental broadening (β_{hkl}) was corrected, corresponding to each diffraction peak using the following relation [13,14].

$$\beta_{hkl} = [(\beta_{hkl})^2_{\text{Measured}} - (\beta_{hkl})^2_{\text{Instrumental}}]^{\frac{1}{2}}. \tag{1}$$

The crystallite size was measured by Williamson Hall (W–H) analysis following Uniform Deformation Model (UDM) [7].

$$\beta_{hkl}\cos\theta = \frac{K\lambda}{D} + 4\varepsilon\sin\theta. \tag{2}$$

A plot was drawn with $4\sin\theta$ along the x-axis and $\beta_{hkl}\cos\theta$ along the y-axis. The W–H equation was modified with the inclusion of Young's modulus concept for Uniform Deformation Stress Model (UDSM) in Eq. (1).

$$\beta_{hkl}\cos\theta = \frac{K\lambda}{D} + 4\sin\theta\frac{\sigma}{E_{hkl}}. \tag{3}$$

For precise measurement of lattice constant and conscientious correction to remove the possible instrumental offset, Nelson–Riley (N–R) parameter $[(\cos^2\theta/\sin\theta) + (\cos^2\theta/\theta)]$ had been used. Precise lattice parameter was calculated by extrapolating the plot of lattice parameter and N–R parameter. Peak displacement occurred with the course of the ball milling due to the formation of stacking faults. For the (111) and (200) reflections, the changes in peak separation due to stacking faults was calculated by [13].

$$\Delta\,(2\theta_{200} - 2\theta_{111})\,(^\circ) = -45\sqrt{3}\frac{2\tan\theta_{200} + \tan\theta_{111}}{2\pi^2}\alpha \tag{4}$$

where α is stacking fault probability. After determination of stacking fault probability (α), the positions of all reflections were corrected due to peak shifts according to

$$\Delta(2\theta) = \frac{360}{\pi}G_{hkl}\alpha\tan\theta \tag{5}$$

G_{hkl} depends on (hkl) reflections. The corrected lattice parameter was determined as per relation mentioned in Ref. [13]. Dislocation density (ρ) was measured using the value of crystallite size and RMS strain [15].

$$\rho = (\rho_D\rho_S)^{\frac{1}{2}} \tag{6}$$

$$\rho_D = \frac{3}{D^2} \tag{7}$$

$$\rho_S = \frac{K\langle\varepsilon^2\rangle}{b^2} \tag{8}$$

where, ρ_D and ρ_S are dislocation density due to domain size and strain broadening, respectively. For Gaussian strain distribution, $K = 6\pi$ and b is the Burgers vector with magnitude $a/\sqrt{2}$ for [1 1 0] direction for FCC structure. The strain excess free volume at the grain boundary according to a simple geometrical model was reported in [15].

$$\text{Excess free volume} \quad (\Delta V) = \frac{((D + \frac{t}{2})^2 - D^2)}{D^2} \tag{9}$$

where, t is the grain boundary thickness.

Further microstructural and compositional analysis of the milled alloys was carried out in detail by High-Resolution Transmission Electron Microscope (HRTEM) (TECNAI T F 30 G2SUPER TWIN Made by FEI) equipped with FISCHIONE High Angle Angular Dark Field (HAADF) detector (Model M-3000). The powder alloy Al–Mg system was ultrasonicated in acetone sufficiently and then carefully put on carbon-coated copper grids of 300 mesh size for TEM analysis.

3. Results and discussions

Fig. 1(a) shows the XRD patterns of Al–Mg ball milled sample in different milling duration (1, 10, 40, 70 and 100 h). Lattice parameter, crystallite size, stacking fault and dislocation density [16,17] were calculated precisely by rigorous XRD analysis. The precise lattice parameter of the milled alloy was calculated using N–R parameter [18]. Fig. 1(b) shows the variation of precise lattice parameter (a') as a function of milling time. The plot shows that with the progress of milling time the value of the lattice parameter increases, i.e. MA of the Al–Mg system gradually displays a tendency of formation of substitutional supersaturated solid solutions of FCC Al (Mg) phase. As the larger atom Mg (160 pm) dissolves into the Al lattice, the strain is induced, and lattice parameter increases. The 100 h milled sample exhibits highest precise lattice parameter value which shows in Fig. 1(b). Determination of precise lattice parameter (a') in faulted materials requires correction as deformation stacking faults result in diffraction line shifting [13,17]. After determining the stacking fault probability the all reflection peak positions were corrected. Consequently, precise lattice parameter values were corrected by using the N–R parameter. Highest lattice parameter value of 100 h milled alloy indicates the maximum solid solution of Mg in Al lattice. Expansion of lattice parameter (%) occurs as the milling time proceeds depicted in Fig. 1(c). Fig. 2(a) shows a comparison between lattice parameter with and without stacking fault correction as a function of milling time (10, 40, 70 and 100 h). It is observed that both values vary linearly with milling time. Primarily, MA induces SPD through dislocation generation. To understand the mechanism, dislocation density was calculated and is enlisted in Table 1. Comparatively larger Mg atoms accommodated themselves along the dislocation line of Al lattice due to high dislocation density through the pinning effect of Mg atoms. Essentially, like pure Al dislocation structure of Al–Mg alloy was rearranged in a way to minimize energy [16,19]. Due to the substitution of the Al atoms by the larger Mg atoms, preferential diffusion on dilation side of the edge dislocation occurred. A spherical symmetry of strain field along the dislocation core formed due to dislocation solute interaction. This is the prime reason behind low energy dislocation structure (LEDS) of the Al–Mg system. Whereas, rearrangement, interaction and tangling of dislocation result in fine grain size [9,19]. In this manner, during milling, high dislocation density reduces the chemical potential and enhances the solubility of Mg atoms [20,21]. Fig. 2(b) shows that crystallite size (measured by both UDM and UDSM methods) decreases with milling time. The system was work hardened due to prolonged milling up to 100 h which facilitated the nanostructure formation. Thus, defect density and diffusion process enhances and