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Achieving excellent thermal stability and very high activation energy in an ultrafine-grained magnesium silver rare earth alloy prepared by friction stir processing

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ABSTRACT

Ultrafine-grained microstructure of a QE22 alloy prepared by Friction Stir processing (FSP) is isochronally annealed to study the thermal stability and grain growth kinetics. The FSPed microstructure of QE22 alloy is thermally stable under ultrafine-grained regime up to 300 °C and the activation energy required for grain growth is found to be exceptionally high as compared to conventional ultrafine-grained magnesium alloys. The high thermal stability and activation energy of the FSPed QE22 alloy is due to Zener pinning effect from thermally stable eutectic $Mg_{12}Nd$ and fine precipitates $Mg_{12}Nd_2Ag$ and solute drag effect from segregation of Neodymium (Nd) solute atoms at grain boundaries.

1. Introduction

Magnesium alloys have received significant attention in aerospace and automotive industries due to their high strength to weight ratio, high damping performance and better creep resistance [1,2]. However, due to their hexagonal closed packed crystal structure, these magnesium alloys exhibit low room temperature ductility, poor formability, yield strength anisotropy and comparatively lower strength [3]. It is well proven that the aforementioned properties of these alloys can be significantly enhanced by two ways such as (i) refinement of grain structure to the ultrafine regime which leads to enhanced strength and ductility and reduced tension-compression yield asymmetry [3] and (ii) addition of rare earth (RE) elements, which improve the formability and ductility by weakening the texture [4–13].

Recently, many researchers have developed ultrafine-grained (UFG) magnesium alloys using severe plastic deformation (SPD) methods [14–29] and found superior properties in the UFG material as compared to their base counterpart. But the practical applications of the UFG materials are limited due to low thermal stability of the microstructure at elevated temperatures that complicates the processing of final products. Thermal stability depends on many variables, such as compositional parameters, processing microstructure, porosity, impurity, grain size distribution and texture [22–24,30–32]. Efforts have been made to develop new UFG and nano-crystalline magnesium alloys having improved thermal stability at higher temperatures by adding reinforcement and other alloying elements [30–33]. However, extensive grain growth have been observed above 200 °C temperature in various nano-crystalline and UFG magnesium alloys [14–18,20,21,25,34].

The formability and ductility of magnesium alloys can also be improved by weakening the recrystallization textures via addition of RE elements to wrought magnesium alloys. Even though many researchers reported that the weakening of texture happens because of segregation of RE atoms to grain boundaries, but the clear mechanism is not well understood. [4–13]. Grain boundary segregation is an extremely sensitive phenomenon that may significantly affect the material properties like corrosion resistance, mechanical behavior or the thermal stability. Also the role of RE elements on thermal stability of UFG magnesium alloys is not well understood. The main objectives of the present work are to first develop UFG microstructure in a RE contained Mg-Ag alloy (QE22) and then to study and correlate the role of solute drag and Zener pinning effect on thermal stability and grain growth kinetics of the developed UFG QE22 alloy.

2. Materials and methods

The QE22 alloy is procured from Magnesium Elektron North

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America which is having chemical composition of Ag, 4.08 wt%; Nd, 2.07 wt%; Zr, 0.6 wt%; Mg, balance. The as-received QE22 plates of 6 mm thickness were FSPed in two passes with a tool rotation rate of 800 rpm and 600 rpm respectively and a traverse speed of 100 mm/min. A custom designed FSP tool was used for FSP passes with shoulder diameter of 12 mm and tapered threaded pin of 3.4 mm length and 6 mm root diameter. In order to investigate the thermal stability and grain growth kinetics of the UFG material, the FSPed plates were subjected to isochronal annealing for 60 min at different temperatures in the range of 100–450 °C, since most of the industrial and superplastic forming operations are conducted within this range at sufficiently high strain rates to avoid grain growth due to the prolonged thermal exposure [14,34].

Microstructural examination was conducted using optical microscopy (OM), transmission electron microscopy (TEM) and scanning electron microscopy (SEM). For OM and SEM characterization the as-received QE22 alloy samples were subjected to mechanical polishing by **using different emery grit sizes**, followed by diamond polishing and etched with picric acid solution. An inverted metallographic optical microscope Quama 5000 series and a Quanta 200 SEM were used for the microstructure observation. For TEM characterization a 500 μm thin foils of FSPed samples are sliced from the processed zone using precision low speed cutting machine and mechanically grounded to less than 80 μm. These thin foils are punched in to 3 mm disc and were ion milled with an incident angle of 6° and an energy beam of 5 eV in an argon environment till a transparent region is formed. Microstructural observation by TEM was undertaken by using Phillips-CM12 electron microscopy operating at an accelerating voltage of 120 kV. To calculate the average grain size of the FSPed samples, a mean linear intercept method was used by considering at least 50 unique grains in each annealing condition. The polished FSPed samples were taken to micro-hardness testing to determine the hardness value. The test was carried out on Future Tech FM-707 Vickers Micro-hardness testing machine with a load of 200 g for 10 s.

3. Results and discussion

3.1. Microstructural characterization of an FSPed UFG QE22 alloy

Fig. 1(A) shows the as-received cast QE22 magnesium alloy which is composed of magnesium solid solution with equiaxed grains surrounded by thermally stable eutectic network of $Mg_{12}Nd$ (Fig. 1(B)) and precipitates of $Mg_{12}Nd_2Ag$ (Fig. 1(C)) inside the magnesium matrix. The average grain size and $Mg_{12}Nd$ eutectic size **is** observed as 38 ± 12 μm and 8.3 ± 3.6 μm respectively **and bearing an** average micro-hardness of 83 ± 4 HV [35]. Fig. 2 shows the TEM microstructure of the QE22 magnesium alloy after FSP at the center of the stirred zone. The FSP **resulted** (i) complete transformation of cast microstructure to uniformly distributed equiaxed dynamic recrystallized UFG microstructure with average grain size of 0.63 ± 0.1 μm because of SPD during multi-pass FSP and (ii) fragmentation of large eutectic $Mg_{12}Nd$ network which was observed along the grain boundary in the cast microstructures in to small particles with average particle size of 0.2 ± 0.05 μm and uniform distribution of these particles along the grain boundaries as well as at triple point junctions of the grain boundaries **and** (iii) dissolution of $Mg_{12}Nd_2Ag$ precipitate in magnesium matrix.

Fig. 3 shows the TEM microstructures after isochronal annealing treatment for 60 min at different temperatures. During annealing process $Mg_{12}Nd_2Ag$ precipitates are formed like a continuous chain along the grain boundary as shown in Fig. 3(B-D). The average grain size, particle size and micro-hardness are plotted against the annealing temperature in Fig. 4. At low-temperature region (100–300 °C) of annealing treatment, there is almost negligible increase in grain and eutectic particle sizes ($Mg_{12}Nd$). A slight increasing trend of both grain size and eutectics is observed at high annealing temperature region (300–450 °C). The grain size retained in UFG regime upto 300 °C and increases upto ~3.8 μm at 450 °C temperature shown in Fig. 5.

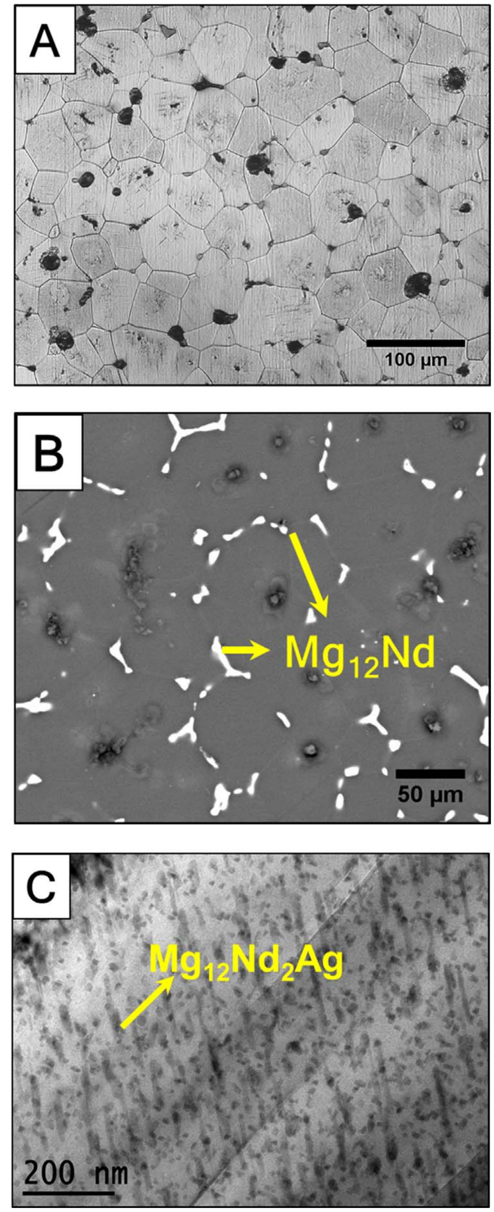

Fig. 1. Microstructure of an as-received cast QE22 magnesium alloy. (A) Optical micrograph showing equiaxed microstructure (B) Back scattered electron (BSE) micrograph; arrow indicates the $Mg_{12}Nd$ eutectic (C) TEM micrograph showing $Mg_{12}Nd_2Ag$ precipitates.

The micro-hardness of UFG QE22 sample is slightly decreased when annealed in the range of 100–300 °C, although microscopy studies did not reveal noticeable grain growth in the specimen (Fig. 3). The softening at low-temperature region may be a consequence of reduction of dislocation density due to static recovery and relaxation of internal residual stresses resulting from the annihilation and rearrangement of dislocations. At higher temperature range (300–450 °C), a significant reduction of hardness is observed with marginal increase in grain growth which may be due over ageing effect (Fig. 4).