Journal of Alloys and Compounds 617 (2014) 253–264

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

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

Effect of aluminium and calcium on the microstructure, texture, plastic deformation and related acoustic emission of extruded magnesium–manganese alloys

K. Illkova [a], P. Dobroň [a], F. Chmelík [a], K.U. Kainer [b], J. Balík [a], S. Yi [b], D. Letzig [b], J. Bohlen [b,*]

[a] Charles University Prague, Department of Physics of Materials, Ke Karlovu 5, 12116 Prague 2, Czech Republic
[b] Helmholtz-Zentrum Geesthacht, Zentrum für Material- und Küstenforschung GmbH, Max-Planck-Straße 1, D21502 Geesthacht, Germany

ARTICLE INFO

Article history:
Received 7 March 2014
Received in revised form 15 July 2014
Accepted 4 August 2014
Available online 12 August 2014

Keywords:
Metals and alloys
Acoustic properties
Anisotropy
Mechanical properties
Microstructure
Scanning electron microscopy

ABSTRACT

Alloying modifications with Ca and Al of a binary Mg-1 wt.% Mn have been used to investigate the microstructure development during extrusion and the relation between these microstructures and the mechanical properties. Acoustic emission (AE) measurements were used to analyse dislocation or twin dominated deformation in differently textured fractions of the microstructure during tensile and compression testing. Ca appears to have a texture modifying effect and leads to the formation of unusual texture components with a tilt of basal planes out of the extrusion direction. This texture development has previously been described for rare earth containing magnesium alloys after extrusion. Al as an alloying element does not lead to the formation of such a component. Both elements have in common, that the alignment of basal plane normals perpendicular to the extrusion direction is reduced, having a substantial effect on the mechanical properties. Partially recrystallised microstructures obtained in Mg-1 wt.% Mn-0.3 wt.% Ca and Mg-1 wt.% Mn-1 wt.% Al after slow extrusion exhibit bimodal grain size distributions. Only in the case of Mg-1 wt.% Mn-8 wt.% Al a fully recrystallised microstructure is found, indicating differences in the grain nucleation rate during recrystallisation. In this alloy, the weakest texture and, correspondingly, the lowest asymmetry in mechanical properties was also observed.

1. Introduction

Magnesium alloys find widespread interest as a light-weight metallic alternative for conventional construction materials. Especially wrought magnesium (Mg) alloys, e.g. in the form of extruded profiles, exhibit finer grained microstructures and improved mechanical properties as compared to cast magnesium alloys [1,2] and may therefore be advantageous in taylored applications. An impediment for this is the typically pronounced texture which develops due to the thermomechanical treatment. It leads to highly anisotropic mechanical properties. Specifically, a large difference between the yield stresses in tension and compression is found for samples tested parallel to the extrusion direction [3]. The typical extrusion texture of Mg alloys orients grains such that the basal plane normals are oriented perpendicular to the extrusion direction. Such grains are therefore unfavourably oriented for basal slip [4,5] during loading parallel to the extrusion direction. If the texture is weaker, the angular distribution of the basal slip planes is broader and the grains are thus more favourably aligned for the activation of basal slip. As discussed in earlier work [3,6], this is likely to enhance the work hardening capability as well as to improve the ductility or formability of the material. On the other hand, a weakening of the texture also leads to a decrease in the yield stress. This motivates to derive enhanced understanding of texture weakening mechanisms of wrought magnesium alloys and a related understanding of the relation between microstructure and mechanical properties.

The microstructure of extruded Mg alloys can also be controlled by tailored alloying. Recent studies have focused mainly on the effects of rare earth (RE) element additions, which have been shown to influence the texture [7–11]. The beneficial effects of RE elements on the properties of Mg alloys have not yet been explained in detail. Changes in the activity of non-basal slip modes [12–14] or the formation of shear bands or deformation bands [8,15] accompanied by the formation of twins other than those on {10–12} planes have been suggested as contributing factors, as well as changes in the recrystallisation mechanisms [6,16] or the recrystallisation kinetics [9,17,18]. The latter effect is often attributed to changes in grain boundary mobility due to solute

* Corresponding author. Tel.: +49 4152 87 1995; fax: +49 4131 874 1995.
E-mail address: jan.bohlen@hzg.de (J. Bohlen).

http://dx.doi.org/10.1016/j.jallcom.2014.08.022
0925-8388/

segregation or particle pinning. Microalloying with RE elements also decreases the grain size and changes the texture of Mg–Mn-based alloys [19,20], which improves the ductility [21]. Mg–Mn alloys have potential for applications because of their good castability, tensile strength and hardness, as well as improved corrosion resistance.

In Ref. [10], a hypothesis linking the large atomic radii of these elements with their ability to weaken the texture was proposed. Consideration of other alloying elements with large atomic radii suggest that Ca could be a non-rare earth addition to Mg–Mn alloys which enables extrusion of profiles with weak textures and more isotropic mechanical properties. Furthermore, it has been shown that the addition of Ca to Mg alloys has a pronounced effect in refining the microstructure [22] and in increasing the high temperature strength and creep properties [23]. For example, the microstructure and tensile properties of Mg–Zn–Si and Mg–Al–Zn–Si alloys are significantly improved by alloying with 0.2 wt.% Ca [24]. In considering magnesium alloys with Ca for biomedical applications is has been shown that also a distinct grain refinement during extrusion can be obtained and attributed to the formation of fine precipitates [25,26], specifically in alloys that also contain Zn. Located at grain boundaries a particle pinning mechanism is rationalised as a grain boundary mobility restriction mechanism to cause such fine grained microstructures during extrusion. Furthermore, intragranular fine particles containing Ca are considered to cause material strengthening based on strengthening of specific slip systems such as basal slip by coherent or semicoherent particles [27,28].

Mn expands the extrusion window, i.e. higher extrusion speeds can be used without the occurrence of hot-cracking during the process [29], especially in wrought magnesium alloys with Al. Furthermore, higher ductilities of the extrudates were achieved. Al itself is known as an element leading to weaker textures of magnesium extrusions if the content is increased [30]. However, typical wrought alloys with higher content of Al such as AZ80 (without Mn) develop typical textures with an alignment of basal planes into the extrusion direction as described above [31]. It is presently not clear, if improved extrudability by using Mn as a substitution to Zn in a Mg–Al–Mn alloy would change the texture formation during extrusion as well as the related mechanical properties. Typical candidates for such alloys are known as magnesium cast alloys with 5 or 6 wt.% Al respectively (AM50, AM60). These effects of Al and Ca motivate investigation of the influence of both elements on the microstructure development and the related mechanical properties in Mg–Mn alloys.

A direct and real time correlation between the microstructure development during plastic deformation and mechanical properties can be determined by concurrent monitoring of acoustic emission (AE). The AE technique is a research tool, in which transient elastic waves generated within the material during deformation due to sudden localized structure changes are sensitively detected and evaluated. In metals, AE responds to dislocation motion and twinning, and therefore allows to identify the active deformation mechanisms [32,33]. Especially, the fast multiplication of dislocations and their propagation over larger distances (e.g. basal slip in coarse grained hexagonal materials), and twin nucleation are excellent sources of AE [33,34]. On the other hand, dislocation movement in material with a large density of sessile obstacles (e.g. forest dislocations), twin growth and thickening produce very weak if any AE signals [33,34]. Thus, the AE response depends on the grain size and grain size distribution, preferred orientation of the grains, the character and distribution of precipitates etc., as these mechanisms influence the activation and size of dislocation avalanches and twins. In recent works, it has been shown, that AE measurements can be used to investigate the dependency of twin nucleation on the grain size in extruded Mg alloys with bimodal microstructure [35], or, in combination with neutron diffraction, for the determination of the twinning activity during tensile and compression tests of cast Mg alloys [36]. Furthermore, the use of the so called Kaiser effect (described as the absence of detectable AE at a fixed sensitivity level, until a previously applied stress level is exceeded [37]) makes a study of the microstructure stability possible. The violation of the Kaiser effect indicates microstructure changes such as recovery or recrystallization, e.g. during unloading or stress relaxations [38].

The object of this work is to study alloys based on the Mg–Mn system, which were fabricated by indirect extrusion with different extrusion speeds. The study is focused on the relationship between microstructural development during extrusion and the resulting deformation behaviour in tension and compression of the extruded bars. Special emphasis is placed on the influence of the alloying elements Al and Ca and their potential to considerably reduce the anisotropy in the mechanical properties, whilst retaining fine microstructures and reasonable levels of tensile and compressive strength and ductility. Detailed studies by X-ray analysis and electron diffraction were combined with mechanical testing and concurrent AE measurements to establish the relationship between the microstructure after extrusion and the deformation behaviour in tension and compression.

2. Experimental

The alloys are based on the commercial alloy M1 (Mg-1 wt.% Mn), alloyed with Ca or Al, and are denoted as MX10 (Mg-1 wt.% Mn-0.3 wt.% Ca), AM11 (Mg-1 wt.% Mn-1 wt.% Al) and AM81 (Mg-1 wt.% Mn-8 wt.% Al). The alloys were gravity cast and then all annealed at 350 °C for 15 h in order to homogenise the cast materials and limit differences in the microstructures to the alloy composition. Billets were machined to a diameter of 93 mm and indirectly extruded to round bar profiles at 300 °C with an extrusion ratio of 1:30. The ram speeds were 0.5 mm/s and 5.5 mm/s, which correspond to extrusion rates (profile exit speeds) of 1 m/min and 10 m/min, respectively. For simplicity, these two extrusion rates will be referred to as "slow" and "fast" extrusion in the following.

The MX10 and AM81 alloys were annealed for 1 h at 120, 150, 200, 250, 350, 425, 475 and 500 °C, in order to investigate the microstructural stability after the extrusion process. After extrusion or annealing, a section from the centre of the extrudate was ground and polished for microstructure and texture analysis. Metallographic analysis using light microscopy was carried out on longitudinal sections of each profile. An etchant based on picric acid was used [39]. A Panalytical© X-ray diffractometer setup using CuK_{α} radiation was used to measure pole figures on polished cross sections up to a sample tilt of 70°. The (00.2), (10.0), (11.0), (10.1), (10.2) and (10.3) pole figures were measured to calculate the complete orientation distribution, which allowed for the recalculation of pole figures and inverse pole figures. A software code "Texture" of Panalytical© has been used to calculate inverse pole figures and intensity distributions.

Cylindrical samples were machined with diameters of 6 mm and gauge lengths of 60 mm for tensile tests and with diameters of 11 mm and lengths of 16.5 mm for compression tests. A universal testing machine Zwick®Z050 was used to carry out tests with a constant strain rate of $10^{-3}\,s^{-1}$ at room temperature (RT). A DAKEL-XEDO-3 computer controlled AE system with a miniaturized MST8S piezoelectric transducer was used to record AE on the basis of two-threshold-level detection [40]. This procedure allows for simple amplitude discrimination at appropriate settings. The transducer (diameter 3 mm, a flat response in a frequency band from 100 to 600 kHz) was attached to the specimen surface with the help of silicon grease and a spring. The AE signal sampling rate was 4 MHz. The threshold voltage for the total AE count N_{C1} was set above the noise level (to 725 mV) and that for the burst AE count N_{C2} to 1440 mV. The total gain was 90 dB.

Orientation imaging was performed on longitudinal sections of the extruded bars using the electron backscatter diffraction (EBSD) technique in a field emission gun scanning electron microscope (SEM). An accelerating voltage of 15 kV and a step size of 0.2 μm were used. Sample surfaces were prepared in the same way as for metallography and were then electropolished using an AC2 solution (Struers™). A software TSL Orientation Imaging Microscopy Analysis of EDAX© is employed to analyse EBSD measurements.

3. Results

3.1. Grain and phase structure after extrusion and annealing

The microstructures of the alloys after slow and fast extrusion are shown in Fig. 1. The slowly extruded MX10 and AM11 alloys