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Particle evolution in Mg–Zn–Zr alloy processed by integrated extrusion and equal channel angular pressing: Evaluation by electron microscopy and synchrotron small-angle X-ray scattering

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Abstract

The evolution of intermetallic precipitate particles in Mg–Zn–Zr alloy ZK60 during thermomechanical processing by integrated extrusion and equal channel angular pressing was investigated in detail. Electron microscopy was employed to analyse individual particles and their orientation within the Mg matrix, while small-angle X-ray scattering (SAXS) was used for an assessment of global particle behaviour and statistical significance of their volume in the microstructure. A significant redistribution of prismatic rod-type and basal platelet-type precipitates, as well as their resolutioning followed by the formation of prismatic platelets, was found. The platelet-type precipitates lying on prismatic $\{2\,\bar{1}\,\bar{1}\,0\}_{\alpha}$ planes were hitherto unknown for the Mg–Zn–Zr system. These precipitates were present in a statistically significant amount detectable by SAXS. Such precipitates should favour an increase of critical resolved shear stress for basal slip in the Mg matrix, thus contributing to an improved performance of the Mg–Zn–Zr alloy. The possibility of formation of prismatic platelet-type precipitates in Mg alloys proven in this paper opens up a new avenue for the design of relatively inexpensive high-performance magnesium alloys.

Keywords: Magnesium; Intermetallic particles; Severe plastic deformation; Small-angle X-ray scattering; TEM

1. Introduction

Magnesium (Mg) products are becoming increasingly popular due to their high structural efficiency and have great potential for functional applications [1–3]. Since pure Mg has very limited applications due to insufficient strength and low-temperature deformability [1,4,5], Mg alloys are commonly used [6]. However, the ability to tailor the properties of Mg solely through alloying is often restricted by the limited solubility of alloying elements [2,3]. Another very efficient way to tailor the properties of various materials is deformation processing, see for

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instance Refs. [7–11] and a recent review [12]. However, due to the limited low-temperature deformability of Mg alloys, their deformation processing has not been widely employed to date [1,3]. Therefore, since the early 1920s until recently the market for Mg alloys has been dominated by cast Mg grades [13]. This situation began to change from the 1990s, and a number of new wrought Mg alloys emerged [14].

The principal alloying elements in the wrought compositions are aluminium (Al), manganese (Mn) and zinc (Zn), and the most popular wrought Mg alloys are M1A, AZ31, AZ61, AZ80A and ZK60 [15]. Among these, the magnesium–zinc–zirconium alloy ZK60 possesses the best combination of strength and ductility at room temperature [14]. Therefore, this alloy was selected for the present investigation. The addition of Al, Zn and Mn to Mg increases its strength through solution hardening as well as precipitation hardening, while zirconium (Zr) promotes grain refinement [15,16]. The latter can be further enhanced through deformation processing to large strains, particularly by severe plastic deformation (SPD) [11,12,17–19]. In the instances when creep resistance and high-temperature strength are of paramount importance, rare earth (RE) elements are added instead of Al or Zn (both of which can form low-temperature eutectics with Mg, thus reducing the operating temperature of final products) [1,15,16]. However, the resources of RE elements are scarce and geographically limited, and the addition of such elements to Mg alloys very substantially increases the cost.

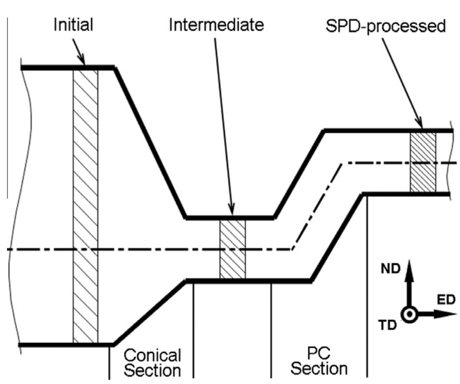

Fig. 1. Schematic illustration of the integrated extrusion and equal channel angular pressing also showing the designation of the principal directions and processed sample conditions.

In our previous investigations [20–22], an integrated process that combines extrusion and equal channel angular pressing (ECAP) to a single-step processing technique was introduced, see the schematic in Fig. 1. It was applied to the deformation processing of alloy ZK60, from which industrially viable 4 m long bars were produced. A simultaneous improvement of several characteristics of the material processed in this way was demonstrated. This shifts the Mg alloy ZK60 to a more favourable position in the property space. In particular, strength and ductility [20,23], along with corrosion resistance [21] and fatigue life [22], were shown to improve simultaneously. These properties were correlated with the grain structure and texture [20,22], and to some extent with the distribution of intermetallic precipitates [21] that result from the processing.

As was first demonstrated by Nie [24] and then confirmed by other researchers [5,10,16,25], the morphology of precipitates is among the most critical microstructure characteristics of Mg alloys. However, these aspects of the microstructure of the bars fabricated by the integrated process were not investigated in our previous studies in sufficient detail [20–23,26]. As explained below, the regime of thermomechanical processing and long-term storage at ambient temperature of the fabricated bars should significantly affect the precipitate state of the alloy. In addition, Takahashi et al. [27] and Mima and Tanaka [28] reported the formation of specific Guinier–Preston (GP 1) zones in Mg–Zn-based alloy systems due to natural and low-temperature ageing. Recently, Buha demonstrated further evidence of that with possible precipitate formation [29]. Hence, the presence and morphology of intermetallic precipitates in the ZK60 Mg alloy system also need to be considered.

Potential precipitates that can be present in the Mg–6Zn–0.5Zr (wt.%) system stoichiometrically [30] are summarized in Table 1 based on Refs. [27,30–32]. Usually, solution treatment of alloy ZK60 is carried out at temperatures above 400 °C and below 520 °C. Above 520 °C, partial melting can be expected, according to the specification of a commercial alloy manufacturer [33]. According to the phase diagram [30], the re-solutioning of Mg–Zn precipitates begins at temperatures above ~300 °C. The specification [33] also suggests the precipitation treated temper (T5) as heating to 150 °C for 24 h, followed by air cooling. In

Table 1
Possible phases in Mg–5.78Zn–0.44Zr (wt.%) alloy system at room temperature.

	Phase	Lattice				Morphological appearance	Ref.
		Type/space group	Parameters [nm]				
			a (α°)	b (β°)	c (γ°)		
1	α: Mg	Hexagonal/$P6_3/mmc$	0.3209		0.5211	Matrix	
2	β: MgZn	Base-centred monoclinic/$C1c1$ or $C1\ 2/c1$	1.610	2.579 (112.4)	0.880	Irregular-shape particles	[31,32]
3	β_1': Mg_2Zn_3 (Mg_4Zn_7)	Base-centred monoclinic/$B/2m$	2.596	1.428	0.524 (102.5)	Rods with long axis parallel to $[0001]_\alpha$	[30,32]
4	β_2': $MgZn_2$	Hexagonal/$P6_3/mmc$	0.5221		0.8567	Plates on $(0001)_\alpha$	[30,32]
5	$Zn_{14}Zr$ ($Zn_{22}Zr$)	Cubic/$Fd\bar{3}m$	1.4105			Standalone cuboids	[30]