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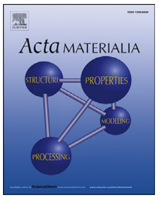

Full length article

# Microplasticity-based rationalization of the room temperature yield asymmetry in conventional polycrystalline Mg alloys

C.M. Cepeda-Jiménez, M.T. Pérez-Prado*

*IMDEA Materials Institute, C/ Eric Kandel 2, 28906 Getafe, Madrid, Spain*

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The aim of the present work is to assess the activity of the dominant deformation slip systems in the commercial cast alloy Mg-9%wt. Al-1%wt. Zn (AZ91) in the as-rolled, solution treated and peak-aged conditions. It is well known that the microstructure of this alloy is formed by $\alpha$-Mg grains, $\beta$-$Mg_{17}Al_{12}$ phases which precipitate mostly as plates parallel to the basal plane, and a smaller fraction of $Al_8Mn_5$ particles with a more equiaxed geometry. Earlier works have shown as well that even after peak aging treatments, very limited hardening can be achieved. Here, EBSD-assisted slip trace analysis is utilized to determine qualitatively the relative activities of basal, prismatic and pyramidal systems in the three microstructures under scrutiny following tensile deformation to a given strain level at ambient temperature and quasi-static rates. The incidence of the different systems is then related to the alloy grain size, texture, and precipitation state. Finally, the macromechanical behavior of the AZ91 alloy and, in particular, the strength, the ductility and the yield stress asymmetry are rationalized based on the measured slip activities. It is shown, in particular, that basal slip is the dominant deformation mechanism under in-plane tension at room temperature in all the microstructures investigated and that the yield stress asymmetry appears to be strongly related to the different stress levels required to activate basal slip (in tension) and twinning (in compression). Recommendations for alloy design are given on the light of these findings.

## 1. Introduction

Magnesium and its alloys have attracted extensive attention in recent years due to their abundance, low-density, good castability and recyclability [1]. Unsolved issues that are limiting the wide commercialization of these materials are the need to achieve superior strength levels and to decrease the tension-compression asymmetry without significantly increasing the production cost. Strengthening of Mg alloys has been pursued by placing obstacles to dislocation slip and by hindering twin nucleation and propagation. The nature of such obstacles might vary, the most common being individual atoms (solutes), second phase particles or precipitates [2], stacking faults [3], and grain boundaries [4]. It has to date not been possible to leverage the beneficial effects of precipitates in Mg alloys to similar extents to those achieved in Al alloys [2]. Reducing the yield stress asymmetry has been successfully achieved mainly by rare-earth addition, with the concomitant increase in cost [1]. Overcoming the currently existing knowledge barriers, which are preventing the full exploitation of these materials, requires an in-depth understanding of the slip and twin activities at a microscopic level, as well as of the interaction between dislocations and twins with obstacles, which, particularly for hcp metals, is still lacking.

There are a number of techniques that have been conventionally utilized to estimate slip activity. Transmission electron microscopy (TEM) [5], the only method that provides direct evidence of the presence of dislocations sliding on specific slip systems, suffers from relatively poor statistics and from potential biases due to sample damage during preparation. Other techniques provide only indirect evidence, which must subsequently be processed. They all have limitations that prevent a conclusive quantification of the activities of different slip systems. These techniques include: the observation of the change in the shape of grains, with the fraction of strain accommodated by slip being estimated from the elongation of grains along specific directions [6]; the analysis of the evolution of texture with deformation [7], which yields ambiguous information when several mechanisms are activated

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* Corresponding author.
*E-mail address:* teresa.perez.prado@imdea.org (M.T. Pérez-Prado).

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simultaneously and requires a relatively large strain level to give meaningful information; the observation of stress–strain curves [1,8,9], that only allow to roughly distinguish between tensile twinning (hereafter referred to as "twinning") and slip dominated flow upon yielding; and computational simulation and modeling methods [10–12] which, despite being very powerful, do not account for physically based microstructural phenomena such as grain boundary strengthening effects on different dislocation systems or the local distribution of orientations (clustering), which can critically alter the balance of deformation mechanisms.

Electron backscattered diffraction (EBSD) assisted trace analysis [13–16] provides direct evidence of the activity of the different slip systems, whether they act in a primarily isolated fashion or simultaneously. This technique, which involves a systematic and rigorous experimental approach, has been recently successfully utilized to shed light on unsolved issues regarding the microplasticity of pure Mg, such as the effect of grain size on slip activity at a wide range of temperatures, as well as on the transition between twinning and slip with grain refinement, with temperature and with strain rate [17–19]. The use of EBSD-assisted trace analysis to unveil the active deformation mechanisms in wrought or heat treated Mg alloys, and thus to assess the interaction between solutes and/or precipitates and dislocations, could provide key inputs for the design of alloys with higher strength and reduced yield stress asymmetry. Such studies are, however, still lacking.

The aim of this work is to estimate the relative activities of basal and non-basal systems during room temperature deformation of a commercial cast alloy Mg-9%wt. Al-1%wt. Zn (AZ91) in the as-rolled, solution treated and peak-aged conditions. With this purpose EBSD-assisted slip trace analysis is carried out in the three microstructures following tensile deformation to a true strain of 0.10. The incidence of the different systems is then related to the alloy grain size, texture, and precipitation state. Finally, the macromechanical response of the AZ91 alloy and, in particular, the strength, the ductility and the yield stress asymmetry are rationalized based on the measured slip activities. Our results highlight the importance of texture-grain size correlations as well as of basal slip in the macroscopic mechanical behavior of this alloy.

## 2. Experimental procedure

The material employed in the current work is a commercial AZ91 alloy. The starting material was a 10 cm diameter ingot in the as-cast condition with a composition of Mg- 9.00 wt.%Al-0.70 wt.% Zn- 0.25 wt.%Mn-0.04 wt.% Si-0.001 wt.%Fe. Slabs of the as-received material, approximately 8 mm in thickness, were first solution treated at 420 °C for 23 h and then hot rolled at 420 °C using four passes, each of ~17% reduction, with an inter-pass annealing of 10 min at 420 °C. A final thickness of ~4 mm was obtained. The resulting microstructure is named throughout the entire text as the "as-rolled" condition. A set of as-rolled workpieces were then solution treated at 420 °C for 18 h followed by water quenching; the resultant microstructure will hereafter be termed the "solid solution" condition. Finally, some of the solution-treated specimens were aged at 210 °C for 8 h in order to generate additional microstructures, which will be referred to as the "peak aged" condition [20,21]. The current study was designed to investigate the effect of these three different starting conditions on the cumulative activity of the different deformation systems of the AZ91 alloy during tensile tests.

The microstructure of the as-rolled and heat treated samples was examined by EBSD and by X-ray diffraction (XRD). EBSD microtexture measurements were carried out using a Helios NanoLab 600i FEI field emission gun scanning electron microscope (FEG-SEM) equipped with a NordlysMax detector, a CCD camera, the AZtekHKL data acquisition software and the Channel 5.0 post-processing analysis package. The FEG-SEM operating conditions were an accelerating voltage of 15 kV and a current of 2.7 nA with a step size of ~1.7 µm. Average grain size values were calculated from inverse pole figure maps (IPF) in the normal direction to the rolling plane (ND) considering only GBs with misorientation angles greater than 15° (Brandon criterion) [22]. The average grain size was calculated by the linear intercept method corrected by a shape factor of 1.74 [23]. Sample preparation for EBSD included mechanical mirror-polishing using diamond pastes of increasingly finer particle sizes and a colloidal silica slurry finishing. The macrotexture of the three samples under investigation was measured by the Schulz reflection method in a Philips X'pert-Pro Panalytical X-ray diffractometer, furnished with a PW3050/60 goniometer and filtered Cu $K_\alpha$ radiation. The surface area examined was about 1 cm$^2$. XRD data were corrected for background and defocusing using the Philips X'pert software. From the incomplete measured pole figures, the orientation distribution function (ODF), and then the complete calculated pole figures were constructed using the MTEX package [24].

A series of dog-bone tensile samples with 10 mm gage length and transversal section of 2 × 2.5 mm$^2$ were electrodischarge-machined out of the as-rolled and heat treated sheets with the tensile axis parallel to the rolling direction (RD). Tensile tests were then carried out at 50 °C and at initial strain rate of $10^{-3}$ s$^{-1}$ using a screw-driven tensile stage (Kammrath and Weiss, Dortmund, Germany). Two tests per sample were performed to failure with the aim of characterizing the full macromechanical response (yield and maximum strength and ductility). Additional tests were stopped at a strain of ~10% in order to evaluate the slip activity by the methodology EBSD-assisted slip trace analysis [14,15]. The estimation of the slip activity was carried out as follows. First, large areas within the gage length of the tensile samples are mapped by EBSD prior to testing. Next, the evolution of the microstructure within such areas was followed during straining by SEM imaging. In particular, special attention was paid to detecting the appearance of as many slip traces as possible in a large number of grains. Subsequently, post-mortem EBSD examination of the same selected large areas was

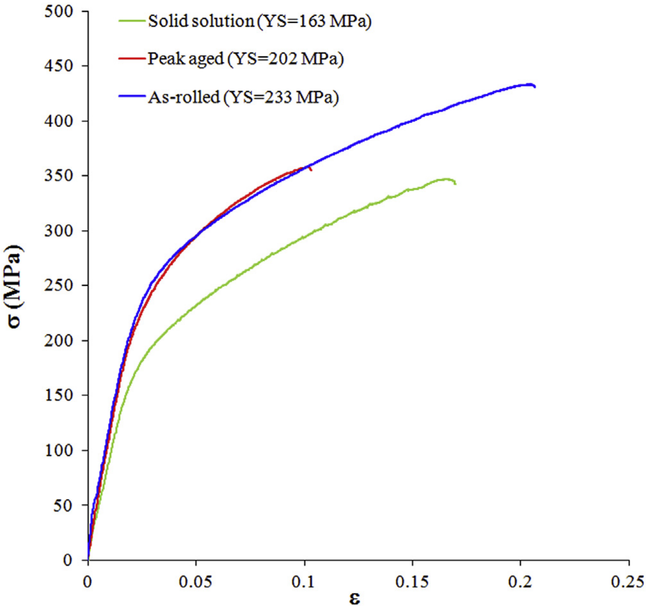

**Fig. 1.** Tensile true stress-true strain curves corresponding to the solution treated, peak-aged and as-rolled AZ91 samples, tested at 50 °C and at an initial strain rate of $10^{-3}$ s$^{-1}$.