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Micromechanisms of grain refinement during extrusion of Mg–0.3 at.% Al at low homologous temperature

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ABSTRACT

Coarse grained Mg–0.3 at.% Al (0.33 in wt.%) alloy was processed by direct extrusion with a reduction ratio of 25:1 at a temperature of ~433 K. The extrusion remainder was removed from the die and analysed in three distinct zones: the cast billet, the conical zone of extrusion die, and the as-extruded rod. The zones were characterized by electron backscatter diffraction (EBSD) and light microscopy techniques to identify the processes responsible for grain refinement. Complex networks of {10–12} twins in practically all grains produced a noticeable microstructural fragmentation even before the material reached the conical zone of the die. Deformation twinning extended up to the entrance zone of the conical die where it was followed by a continuous dynamic recrystallization (CDRX) that gradually changed low angle boundaries to high angle boundaries. It is apparent that geometrically necessary dislocations play a crucial role in the formation of new grain boundaries. CDRX results in a bimodal structure with grain diameters ~3 and ~30 μm. As a material flows through the conical zone, the ratio of large to small grains is progressively decreased by CDRX in favour of fine grains. The as-extruded microstructure (a rod 8 mm in diameter), with an average grain diameter of ~2.1 μm, shows a strong texture where the vast majority of grains (99.99%) have the c-axis oriented at least 30° from the extrusion direction.

1. Introduction

Low density and high specific strength make magnesium alloys potentially good candidates for numerous structural applications [1]. However, significant technical barriers still limit a widespread use. These barriers include, for example, difficult formability at low temperatures, generally low ductility of finished components, and a limited alloy set [2].

It is well-known that the limited formability of magnesium alloys at lower homologous temperatures (<0.5 Tm, Tm melting point) often leads to cracking due to their hexagonal close packed structure [2]. Unlike most engineering materials which have highly symmetric cubic crystal structures with a sufficient number of active slip systems, Mg has a hexagonal structure with the only easy mode of deformation in the basal plane. Different studies have confirmed that the critical resolved shear stress for non-basal systems, especially <c + a> dislocations, is usually much higher than that for basal slip [3].

A lack of active slip systems at ambient temperatures facilitates abundant twinning [4]. Twinning is a particularly important feature in all hexagonal metals because it represents a basic mechanism of their plastic deformation, and

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is capable of partially accommodating plastic strain. Well-established twinning modes in magnesium occur on planes: $\{10\bar{1}1\}$, $\{10\bar{1}2\}$, $\{10\bar{1}3\}$, $\{10\bar{1}1\}$–$\{10\bar{1}2\}$ and $\{10\bar{1}3\}$–$\{10\bar{1}2\}$ [4,5]. Among these, $\{10\bar{1}2\}$ is the most common twinning plane that reorients the magnesium lattice around the <11–20> axis by an angle of 86°.

An insufficient number of active deformation modes available in Mg at ambient and elevated temperatures often lead to cracking. Therefore, Mg and its alloys must be processed at higher homologous temperatures, which in turn suppress grain refinement due to grain growth [6]. Among the various metal forming processes, extrusion plays an important role for processing magnesium alloys due to its technical and economical advantages in the production of structural components [7].

Most works dealing with microstructure evolution in the conical zone of an extrusion die were done at high homologous temperatures (>0.5 Tm), where the processes of grain refinement may substantially differ from those activated at T < 0.5 Tm [7–10]. At lower temperatures, thermally activated phenomena such as the mobility of grain boundaries (GBs) are limited, and high angle boundary structures must evolve during dynamic recrystallization (DRX) without any obvious nucleation and growth. In this respect, publications [11–13] are of interest because they are devoted to low temperature extrusion of magnesium and its alloys. Swiostek et al. [11] successfully realized industrial extrusion of several magnesium alloys down to 373 K and Tork et al. [12] showed the possibility of simple shear extrusion of pre-extruded magnesium even at room temperature (RT). In addition, Zhang carried out an in-depth crystallographic analysis of twinning in AZ31 magnesium alloy extruded in an aluminium shell at RT [13]. All of these studies proved the feasibility of low temperature extrusion, but their microstructural characterization mainly covers the as-extruded products. Despite significant progress in the low temperature processing of Mg alloys in the last few years, there is still a lack of detailed insight into the microstructure evolution during low temperature extrusion, even though understanding the recrystallization mechanisms is a key element in controlling the microstructure and, thus, in altering the final properties. To make up for this shortcoming, it is essential to investigate the processes taking place during the extrusion of magnesium at low homologous temperatures.

This work aims to identify the microstructure evolution in binary Mg–0.3 at.% Al alloy during direct extrusion at 433 K (0.46 Tm) where thermally activated phenomena are limited. Particular attention is paid to finding a sequence of grain refinement mechanisms in the extrusion remainder and to twinning, which often controls deformation mechanisms in metals with hexagonal structure.

2. Material and Methods

Binary magnesium alloy Mg–0.3 at.% Al was employed as a model case in the present work. An as-cast ingot was homogenized at 723 K for 6 h. The homogenized microstructure was twin-free and the grain size was a few mm with partially columnar structure as a result of die casting. As expected from the Al–Mg phase diagram, second phase particles (e.g. precipitates) were not observed. A cylindrical billet of 39.5 mm in diameter and ~100 mm high was machined from the homogenized ingot. Direct extrusion was conducted on a well-lubricated billet at a temperature of ~433 K and at a relatively low ram speed of 0.2 mm/s to avoid significant adiabatic heating. The initial and exit diameters of the conical extrusion die were 40 mm and 8 mm, respectively, which corresponds to a reduction ratio of 25:1. The microstructure was analysed by electron backscattered diffraction (EBSD) and light microscopy techniques. EBSD measurements were performed using a FEI Quanta 3D FEG scanning electron microscope (SEM) fitted with a TSL/EDAX Hikari camera. Sample preparation for microscopic techniques consisted of grinding with progressively finer SiC papers and mechanical polishing with diamond suspensions of particle size 3 and 1 μm. Final polishing was performed with a solution of colloidal silica followed by etching in Glycol (1 ml HNO_3, 24 ml water, 75 ml ethyleneglycol) for light microscopy and ion milling (PECS by Gatan) for EBSD measurement. The percentage of the material that could be indexed by EBSD was 80–90% and the mapping step was selected to be 0.2 or 0.5 μm. The identification of the twin and grain boundaries relied on EBSD measurements on either side of a boundary. Average grain size was determined by the intercept length routine in cleaned EBSD maps by TSL OIM 5.3 software and verified by the evaluation of SEM micrographs (linear intercept method with coef. 1.74). Each EBSD map for grain size calculation contained about 5×10^3 grains.

3. Results and Discussion

3.1. Extrusion Remainder

The metallographic section of the extrusion remainder is shown in Fig. 1b together with typical microstructures before (Fig. 1a) and after extrusion (Fig. 1c). For the sake of clarity, the characteristic zones of the extrusion remainder are labelled as I, II and III, where I depicts the unextruded (cast) billet, II is a conical part and III represents the as-extruded rod. These zones, including transitions I–II and II–III, will be discussed separately but in given order to chronologically describe microstructure evolution during direct extrusion.

3.2. Zone I: Unextruded billet

The inverse pole figure (IPF) map in Fig. 1a shows a typical detail of an unextruded billet with estimated grain size ~3 mm. The most distinctive aspects of the microstructure in zone I are the deformation twins which occur with a non-uniform distribution in practically all grains. Abundant twinning throughout zone I is caused by expansion of the billet to fill an extrusion container by the ram since twinning is capable to accommodate (at least partially) the imposed plastic strain. Besides twinning, slight dislocation activity was also detected. This can be observed as a gradual colour change in the untwinned matrix and as the formation of a few low angle boundaries (Fig. 1a). These dislocations can originate either from plastic deformation occurring concurrently with twinning or from compensation of shape changes in grains caused by twinning.

As confirmed by EBSD, the vast majority of twins belong to the <11–20> 86.3° {10–12} twinning system, independent of