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Enhanced ductility in high-strength fine-grained magnesium and magnesium alloy sheets processed via multi-pass rolling with lowered temperature

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ABSTRACT

Ductility enhancement up to about 28–39% in fracture elongation with simultaneous strengthening was achieved in fine-grained magnesium and magnesium alloy sheets by multi-pass rolling with lowered temperature suitable for industrial fabrication. Specifically, high strengths at yielding following slip-dominated Hall–Petch relations were achieved owning to the naturally twinning's replacement by pyramidal<c+a> under intense basal texture. These Hall–Petch relations had a high slope of 190 MPa $\mu m^{1/2}$ and frictional stresses ranging from 40 MPa to 144 MPa. The variation in frictional stress matched well with solid solution hardening and/or precipitation hardening. Enhanced uniform elongation was strongly correlated with improved strain-hardening rate via introducing solid solution and/or fine precipitates in Mg alloys compared with pure Mg, and conversely post-necking elongation (local elongation) as well as cold-workability under compressive stress was strikingly weakened by the restriction of dynamic recovery.

1. Introduction

Wrought magnesium alloy sheets have great potential for structural applications for saving weight owing to their high specific properties, but impeded by the poor workability and relatively low strength at ambient temperature [1]. Tailoring texture, often represented by the inclination/weakening of {0002} pole intensity in the plane, has been shown to effectively improve the ductility of Mg alloys. For instance, excellent fracture elongation up to ~45% in an AZ31 alloy [2] was achieved via equal channel angular pressing (ECAP), and 31% in a Mg–Zn–Gd alloy [3] via cyclic extrusion and compression (CEC). However, such techniques often introduce a marked sacrifice in strength along processing direction (yield strength <150 MPa and ultimate tensile strength <250 MPa) [2–4], due to the profound texture planar anisotropy, and are still seriously limited to laboratory scale.

Comparatively, fine-grained structure is attracting large attention for better ductility while simultaneously enhancing strength owing to their high Hall–Petch slope k_y values [5,6], especially under an intense basal texture. This is believed to be readily achieved during industrial rolling via dynamic recrystallization, and is significantly different from the mutually exclusive relation between ductility and strength in tailoring texture, even at finer grain sizes. For example, Kim et al. [7] reported that a fine-grained AZ31 Mg alloy of 1.4 μm in average was attained after single pass high-ratio differential speed rolling (HDSR) with 70% thickness reduction at 413 K, leading to a maximum elongation of 24%, and high yield stresses as high as 317 MPa along rolling direction. Other examples include: accumulative rolling-bonding (ARB) in AM60 [8], and cross-rolling (CR) in AZ31 [9]. Additionally, lowering temperature is generally implemented to refine the grain size significantly due to the close dependence of grain size on Zener–Hollomon parameter [5,10], especially for multi-pass rolling. Our previous work [11] showed that improved properties in fracture elongation of 32% and strength larger than 310 MPa corresponding to a uniform fine–grained structure of 3.5 μm in average were achieved in ZK61 Mg alloy sheets by seven-pass hot rolling with lowered temperature from 673 K to 513 K.

Unfortunately, grain refinement is not always beneficial to uniform elongation (much less affected by the gauge length than fracture elongation [12]), and consequently not favorable to stretch forming operations, referring to the researches of Wu in a Mg-Gd-

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Zn alloy [13] and Chino in a Mg–Al–Zn alloy [14], and its mechanisms are not entirely clear yet. This gives a false impression of high ductility due to the contribution of large post-necking elongation (particularly for samples with very short gauge length), and goes against the potential wide application of Mg alloy sheets. Thus, in this paper we reported excellent ductility up to about 30% in fracture elongation while simultaneous strengthening was achieved in commercial Mg and Mg alloy sheets, which were efficiently fabricated by multi-pass rolling with lowered temperature. The microstructure and mechanical behaviors for the fine-grained Mg and Mg alloy sheets under intense basal textures were investigated to reveal the mechanisms on ductility enhancement, especially for uniform elongation, aiming to provide a beneficial reference in designing high-performance wrought Mg alloy sheets suitable for industrial application.

2. Experimental

Commercial pure Mg, AZ31 and ZK61 alloy ingots of about 250 mm in diameter were used as the starting material. These ingots were casted by semi-continuous casting and supplied by Shanxi Yinguang Huasheng Magnesium Industry Ltd, PR China. The compositions of the three materials were high purity Mg (Mg $\geq$ 99.95%, MgHR), Mg-3.28wt.%Al-0.46wt.%Zn (AZ31HR), and Mg-6.63wt.%Zn-0.56wt.%Zr (ZK61HR), respectively. The dimensions of samples prepared for hot-rolling amounted to about 150 mm width $\times$ 28–30 mm thick $\times$ 200 mm length. The length direction was in accordance with the ingot solidified direction and hence the subsequent rolling direction. The samples were homogenized at 673 K for 24 h (*as-homogenized*) performed in an electric furnace to improve component segregation and eliminate possible intercrystalline phases, and subsequently subjected to multi-pass rolling with lowered temperature to obtain fine-grained structure (*as-rolled*). Some preliminary experiments focusing on microstructure evolution and performance improvements have been conducted to maximize rolling thickness reductions (taking plate yield and efficiency into consideration for normal production) and to optimize rolling temperatures during multi-pass rolling [11,15–18]. As a result, the chosen hot-rolling processes in this work consisted of seven rolling passes of ~30% thickness reduction per pass with lowered temperature from 673 K to ~473 K for MgHR, to ~523 K for AZ31HR, and to ~533 K for ZK61HR, respectively. The process flow diagram is illustrated in Fig. 1 and the process parameters are in Table 1. The hot rolling was carried out on a two–high reversing mill, whose roll diameter and velocity were 220 mm and 5 m/min, respectively. Temperature was measured using infrared sensors installed at the inlet and outlet part of the mill, and was controlled by induction heating and force–air cooling devices. For comparison, a low-temperature prolonged annealing at 483 K for 72 h was performed on ZK61HR aiming to reduce supersaturation (denoted as ZK61LA). Tensile tests at ambient temperature were carried out on INSTRON5569 with a constant speed equal to an initial strain rate of 6.7 $\times$ 10^{-4} s^{-1}, and were repeated three times for each sheet. Dog-bone specimens with a gauge length of 25 mm and a width of 6 mm were machined out of the middle of the as-rolled sheets along the rolling direction. Microstructure and texture in the RD–TD plane were identified using electron backscattering diffraction (EBSD) performed on JEOL 733 electron probe equipped with TSL OIM Analysis system, and optical microscopy (OM) on OLYMPUS GX71. The calculation method of Harmonic series expansion with series rank of 16 and Gaussian half-width of 5° were used in OIM to generate pole figures and calculate corresponding maximal pole densities. Mean linear intercept method ($\overline{d}$, L is the linear intercept) was adopted to measure average grain size. TECNAI transmission electron microscopy (TEM) equipped with energy dispersive spectroscopic (EDS) system was applied to image microscopic features. The accelerating voltage used was 20 kV for EBSD and 200 kV for TEM.

3. Results and discussion

3.1. Microstructure characteristics before and after hot rolling

Fig. 2 shows the microstructural characteristics of as-homogenized Mg and Mg alloys and corresponding room-temperature stress–strain curves during tension. As shown, all the as-homogenized ingots consisted of obviously coarse, irregular grains of about 640 μm in average grain size for Pure Mg (a), 431 μm for AZ31 (b), and 278 μm for ZK61 (c), respectively, and profuse parallel {10–12} tension twins of ~5–20 μm in width were formed inside them after homogenization [19]. The representative crystallographic relationships of twin variants in ZK61 (for instance, the marked parent grain (P) with twin bands (T1 and T2)) are illustrated in Fig. 2d and e, where the normal direction (ND) corresponds to the crystal reference system in colored inverse pole figure map. Misorientations of ~86° were measured between the parent grains and twin bands as shown in Fig. 1f. As a result, these coarse-grained structures brought disappointingly poor mechanical performance in materials (Fig. 2g, YS: the 0.2% yield strength; UTS: ultimate tensile strength; UE: uniform elongation; FE: fracture elongation). Specifically, merely 92 MPa in yield strength (YS) and 13% in fracture elongation were observed in ZK61 and it appeared to be worse for others.

Fig. 3 displays the microstructural characteristics of the *as-rolled* sheets after multi-pass rolling in the RD-TD plane (RD: rolling direction, TD: transverse direction) (a–c: MgHR, d–f: AZ31HR, g–i: ZK61HR). Comparatively, it is clearly that evidently fine-grained microstructures together with better planar texture isotropy were achieved for all the materials after multi-pass hot rolling with lowered temperature. With relatively equiaxed grains and low grain size dispersions, the grain sizes of the recrystallized structures were strikingly refined to 7.3 μm for MgHR, 5.2 μm for AZ31HR, and 4.8 μm for ZK61HR, respectively, via multiple dynamic recrystallizations [11]. Effective refinement seems readily to be obtained in Mg alloys, although pure Mg had the lowest finishing rolling temperature. In turn, this means that pure Mg may possess a

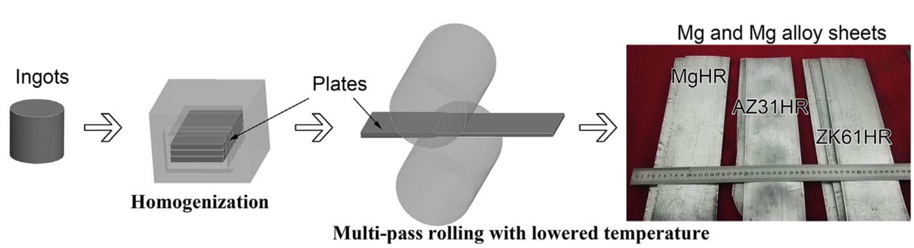

Fig. 1. Process flow diagram of Mg and Mg alloy sheets produced multi–pass rolling with lowered temperature.