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Calcium and zirconium as texture modifiers during rolling and annealing of magnesium–zinc alloys

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Rolling experiments were carried out on a ternary Mg–Zn–Ca alloy and its modification with zirconium. Short time annealing of as-rolled sheets is used to reveal the microstructure and texture development. The texture of the as-rolled sheets can be characterised by basal pole figures with split peak towards the rolling direction (RD) and a broad transverse angular spread of basal planes towards the transverse direction (TD). During annealing the RD split peaks as well as orientations in the sheet plane vanish whereas the distribution of orientations tilted towards the TD remains. It is shown in EBSD measurements that during rolling bands of twin containing structures form. During subsequent annealing basal orientations close to the sheet plane vanish based on a grain nucleation and growth mechanism of recrystallisation. Orientations with tilt towards the TD remain in grains that do not undergo such a mechanism. The addition of Zr delays texture weakening.

1. Introduction

Conventional magnesium sheets, like those known from magnesium alloy AZ31, lack in enhanced formability, especially at room temperature. Numerous works have shown that a texture variation can help to overcome this issue [1–5]. The typical strong alignment of basal planes in conventional magnesium sheets limits the strain accommodation during deformation as well as the resulting work hardening ability [6]. If textures are weaker, higher ductility even at room temperature as well as improved formability can be achieved.

It has been shown that adding rare earth elements results in texture weakening characterised by a tilt of basal planes away from the sheet plane. Several approaches have been used to explain this behaviour which is very different compared to the same alloys without addition of rare earth elements [7]. The mechanisms which are considered to lead to such texture changes draw a line from changes in the active deformation mechanisms, such as slip modes [6,8–11], different types of twins [7,12] and shear bands [13,14] to changes during microstructure reformation as a result of recrystallisation [13,15–18].

In an attempt to overcome the necessity of including rare earth elements to improve sheet properties, actual works deal with alternative approaches, e.g. considering calcium (Ca) as an alloying element in magnesium. This can result in very similar effects in the texture development during rolling, e.g. [19]. Again, texture effects are those that significantly contribute to improved formability of such sheets.

Ca as an alloying element in magnesium has some features that compare well to those of rare earth elements, such as a size misfit in the magnesium lattice structure or a decrease of stacking fault energies [20,21]. Corresponding to this, it was found that Ca additions to Mg–Zn alloys lead to textures that have strong similarities with those of rare earth elements containing Mg–Zn sheets [5,22]. In these works a strong contribution of different twin types and subsequent static recrystallisation are emphasised as texture determining mechanisms.

In this work a Mg–1 wt.% Zn–0.5 wt.% Ca with and without Zr as a grain refiner in magnesium alloys is used and the developing microstructure during rolling is investigated. The sheets were analysed before and during annealing in order to track the texture development and reveal the significance of different mechanisms on the microstructure formation.

2. Experimental

Gravity die-cast billets of two alloys, ZX10 (Mg + 0.91 wt.% Zn + 0.52 wt.% Ca) and the zirconium containing modification ZXK100 (Mg + 0.86 wt.% Zn + 0.51 wt.% Ca + 0.08 wt.% Zr) were used to cut slabs (dimensions: 20 mm thickness, 280 mm width and 50 mm length) for rolling experiments. A solution annealing was carried out for 16 h at 450 °C prior to rolling. A 50 ton laboratory rolling stand with cold rolls of 400 mm diameter was used to carry out rolling experiments on both alloys at 400 °C. A rolling procedure with increasing

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degree of deformation per pass was applied, starting with 4 passes of 0.1 followed by 7 passes of 0.2 and 3 passes of 0.3. The same rolling schedule has been applied in earlier work [12].

In the case of ZXK100 two passes of 0.05 were applied instead of the first pass with 0.1 to avoid early cracking of the sample. The rolling speed was constant at 16 rounds per minute. Between the passes the samples were reheated to 400 °C for 15 min. The as-rolled sheets were used to apply annealing experiments at 400 °C for various times in order to track the microstructure development. For this purpose the samples were put into a circulating air furnace for very short periods of 15 s, 30 s, 45 s, 60 s, 90 s and 300 s. Obviously, the sample did not reach the targeted temperature at these short annealing times but the procedure is consistent with a technical annealing process. Furthermore, one sample was annealed for 1800 s (30 min). All samples were air-cooled after annealing.

Standard metallographic sample preparation procedures were applied and an etchant based on picric acid was used [23] to reveal the microstructure in optical microscopy. Texture measurements were performed on samples ground and polished to their mid-planes. A Panalytical X-ray diffractometer with Cu Kα radiation was used to measure six pole figures up to a sample tilt of 70°. A computer code MTEX [24] was applied to calculate the orientation distribution function and recalculate full pole figures. Electron backscatter diffraction (EBSD) was used to measure local orientation patterns on a field emission gun scanning microscope (Zeiss, Ultra 55, EDAX/TSL) from longitudinal sections of the samples. Measurements were repeated at different sections of the samples to verify the reproducibility of results. An accelerating voltage of 15 kV and a step size of 0.3 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© was employed to analyse the EBSD measurements. Different data cleaning procedures were tested using TSL cleanup functions. A cleanup procedure consisting of a grain confidence index (CI) standardisation and a neighbour CI correlation was applied. The first function allows measured points with low CI but similar orientation like the surrounding measured points to be rated as properly indexed. A grain tolerance angle of 5° and a minimum grain size of 2 points were selected. The second function is applied on measured points with a CI lower than 0.04. In the case of a point with lower CI the neighbour with the highest confidence index is selected and the orientation is replaced. The combination of both cleanup functions allows a clear identification of boundaries. There has been no significant impact on the resulting textures of this work especially in comparison to other data processing approaches including a simple CI selection of points with different point CI, e.g. 0.03 or 0.05. EBSD data analysis especially included a function to separate grains with different grain orientation spreads (GOS). An additional limitation of a minimum grain size of 0.6 μm (again 2 measured points) was set. In this approach an average orientation is determined for a respective grain. The deviation for each measured point in this grain from the average orientation is then calculated and averaged as the GOS. The software then allows separation of grains with specified GOS.

3. Results

Fig. 1 collects micrographs after rolling as well as after annealing of the ZX10 (Fig. 1a) and ZXK100 (Fig. 1b) sheets. For both alloys significant changes in the microstructure are found even as a result of short-time annealing. In the as-rolled condition a deformed microstructure of originally equiaxed grains contains a high concentration of twins. The main difference in the grain structure of the two sheets is the grain size which is finer in the case of ZXK100 than in the case of

a)

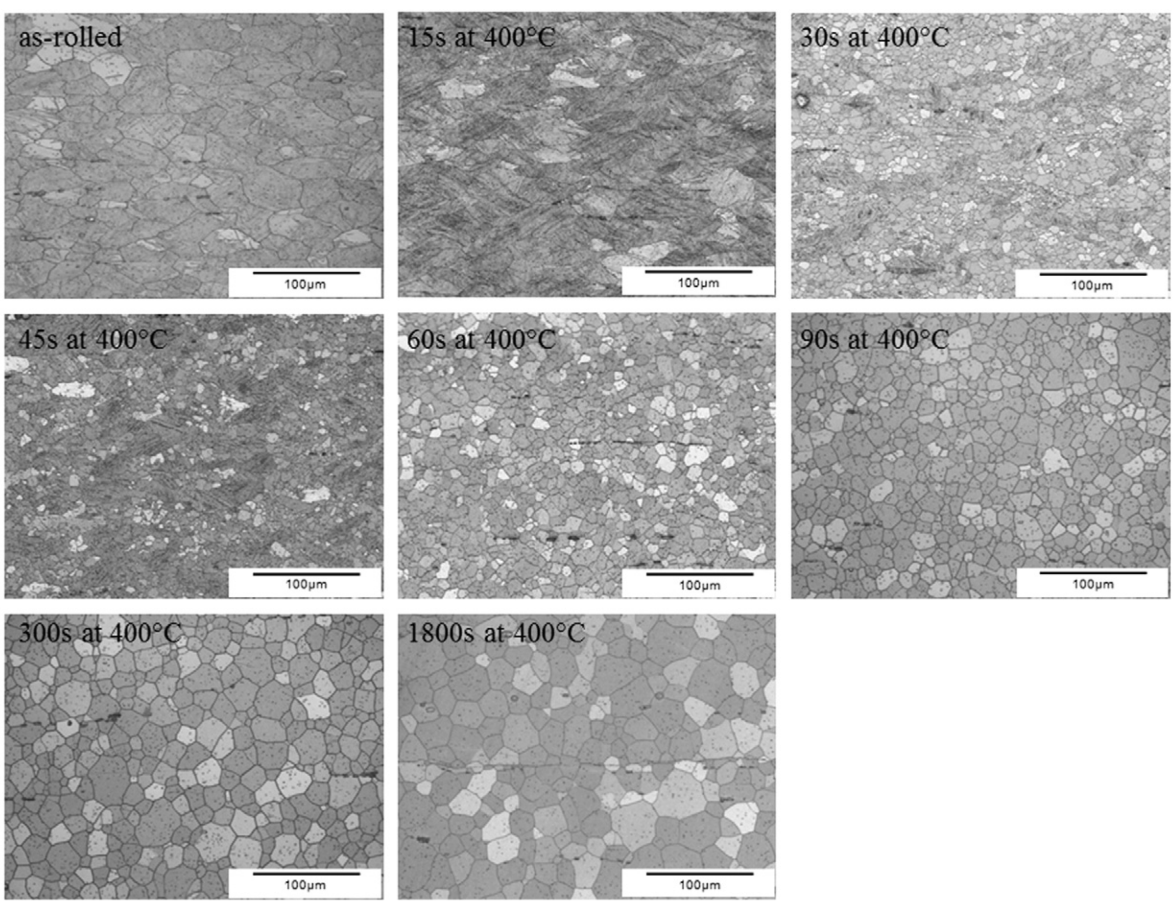

Fig. 1. Microstructure of as-rolled and annealed sheets after varied annealing times; a) ZX10, b) ZXK100 (RD horizontal, ND vertical), c) average grain size of recrystallised microstructures after annealing (60 s to 1800 s).