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Improving the mechanical properties of pure magnesium by three-roll planetary milling

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ABSTRACT

In this study three-roll planetary milling was used to trial its potential for enhancing the mechanical properties of pure magnesium. The material was subjected to several passes of planetary milling at progressively decreased temperatures, down to room temperature for the last pass. It was shown that with increased number of passes the grain size was reduced, eventually to the micron range. Concurrently, the microstructure became increasingly more uniform across the billet. After six passes pure magnesium showed an excellent combination of tensile properties at room temperature, with a yield stress of 116 MPa and a strain to failure of 13%.

1. Introduction

Magnesium is receiving a great deal of attention as a candidate material for biomedical applications owing to its advantages over other metallic materials, especially with regard to biocompatibility and biodegradability. In addition, magnesium has a low density, which means a better comfort for the patient with a Mg implant. Research into the suitability of magnesium and its alloys as implant materials has a long history [1,2] and is booming at present [3,4], but the benefits of Mg associated with its non-toxicity and biodegradability are not matched by its mechanical performance, which is an obvious obstacle to applications.

One of the limitations of pure magnesium with regard to its use in medical implants is its low mechanical strength and poor ductility [2], attributed to its hexagonal crystal structure. For use in load bearing applications high strength is required. If one makes a conscious choice to employ pure magnesium in order to eliminate any potentially toxic elements, the only path to strengthening is through grain refinement and texture control. Popular severe plastic deformation techniques, such as equal-channel angular pressing (ECAP), do lead to substantial grain refinement down to 1 μm, or even sub-micron scale [5], but the resulting texture promotes dislocation glide on basal planes [6–8]. This texture is helpful in terms of improvement of tensile ductility of Mg, but it negates the strengthening effect of grain refinement produced by ECAP. In a search for an alternative process, which is more amenable to industry scale production of ultrafine-grained pure Mg, we trialed a deformation process referred to as three-roll planetary milling or (in German literature) Planetenschrägwalzen (PSW) [9–11] or (in Russian literature) radial-shear rolling [12,13]. Colloquially, the process is also called screw rolling.

This technique has several advantages, such as high speed at which the material is processed, mass production ability, flexibility with regard to the workpiece dimensions, and a broad range of possible process parameters, including temperature and the rate of deformation [14]. Moreover, it was found that mechanical properties are substantially enhanced already after a single pass and no more passes would be needed in an industrial processing environment. The principal design of a three-roll milling machine includes three conical rolls and a mandrel that drives the billet between the rolls (Fig. 1). The rolls are inclined to the central axis of the rod-shaped billet in such a way that their axes intercept the axis at a fixed offset angle. When passing through the mill, the billet is subjected to a screw deformation that reduces its diameter and increases its length. The final diameter of the billet can be adjusted by modifying the positions of the rolls. Multiple passes with progressively decreased diameter of the processed rod are possible, so that the cumulative strain imparted to the rod can be

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very large. Publications on the use of this technique are rather scarce [9–17] and to the best of our knowledge it has never been used for processing of pure magnesium.

2. Experimental

The material used in the present study was extruded magnesium of commercial purity (99.9 wt%) with minor contamination with Zn (0.011 wt%) and Si (0.0108 wt%) received from Yirium Company (China). The material was machined and used in the form of rods 21 mm in diameter and 140 mm in length in order to fit the dimensions of the three-roll mill. Prior to planetary milling, rods were heated in a furnace in air for 30 min and subsequently pressed through the three-roll planetary mill at a speed of 80 mm/s. No lubricant was used. The first pass was conducted at 250 °C, and the rod diameter was reduced from 21 mm to 19.5 mm. The main objective here was to perform a first refinement of the microstructure so that the following passes would be able to be done at a lower temperature, thus producing cold working of the material. We targeted processing temperatures well below the recrystallization temperature, as deformation at or above the recrystallization temperature would not permit the desired grain refinement and associated enhancement of strength. As will be shown below, already the first pass produced a high degree of grain refinement and the concomitant increase in strength. Further passes were then conducted in order to show the potential of the process and to determine how far we can drive the strength of the material. A staggered reduction of the processing temperature for the consecutive passes (P1–P11) was used according to the following schedule: P1 250 °C, P2 230 °C, P3 210 °C, P4 190 °C, P5 170 °C, P6 150 °C, P7 130 °C, P8 110 °C, P9 90 °C, and P10 50 °C. The 11th pass, P11, was conducted at room temperature (RT). The final rod diameter obtained after 11 passes was 13 mm, corresponding to a total true strain of 0.96. After being pressed, the rods were air cooled.

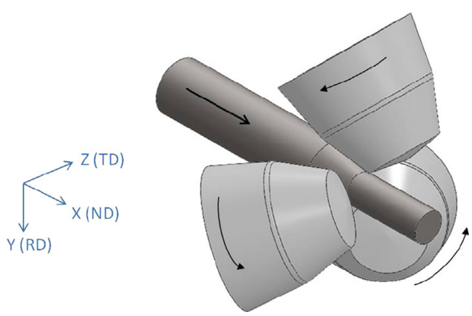

Fig. 1. Schematic of the three-roll planetary milling process. The arrows indicate the directions of the roll rotations and the translation movement of the billet. The coordinate system used is also shown.

For microstructure and texture characterization, specimens were cut from a rod normally to its axis. The microstructure and the texture were observed in this plane (*yz*). The sections were mounted in epoxy, polished following the ASTM standard [18] and finally etched with picric acid solution (5 ml acetic acid, 6 g picric acid, 10 ml distilled water, 100 ml ethanol) [19] for 5–15 s. The microstructure was observed in an optical microscope Olympus BX51M (Japan) and the uniformity of the sample was checked in terms of the variation of the grain size in radial direction, measured in 1 mm steps by the standard Mean Lineal Intercept method following the ASTM standard E112-96 [20]. The grain size was also measured by means of TEM observations using a JEM-2100 transmission electron microscope. Thin foils for electron microscopy were prepared by ion polishing with a GATAN 600 unit.

Texture analysis was carried out using X-ray diffraction in a DRON-7 goniometer with the $Co_{k\alpha}$ radiation in the reflection mode. Six incomplete pole figures, {00.4}, {10.0}, {10.1}, {10.2}, {10.3}, {11.0} were obtained with a step of 5° in the radial angle α and the azimuth angle β on a pole figure (maximum inclination angle $\alpha_{max}=70°$). The orientation distribution function (ODF), $f(g)$, was calculated from the measured pole figures presented as a superposition of a large number (1000) of standard distributions with equal small scatter [21]. Quantitative texture analysis was carried out on this basis.

The mechanical properties were characterized by means of tensile tests performed on flat tensile specimens having a gauge

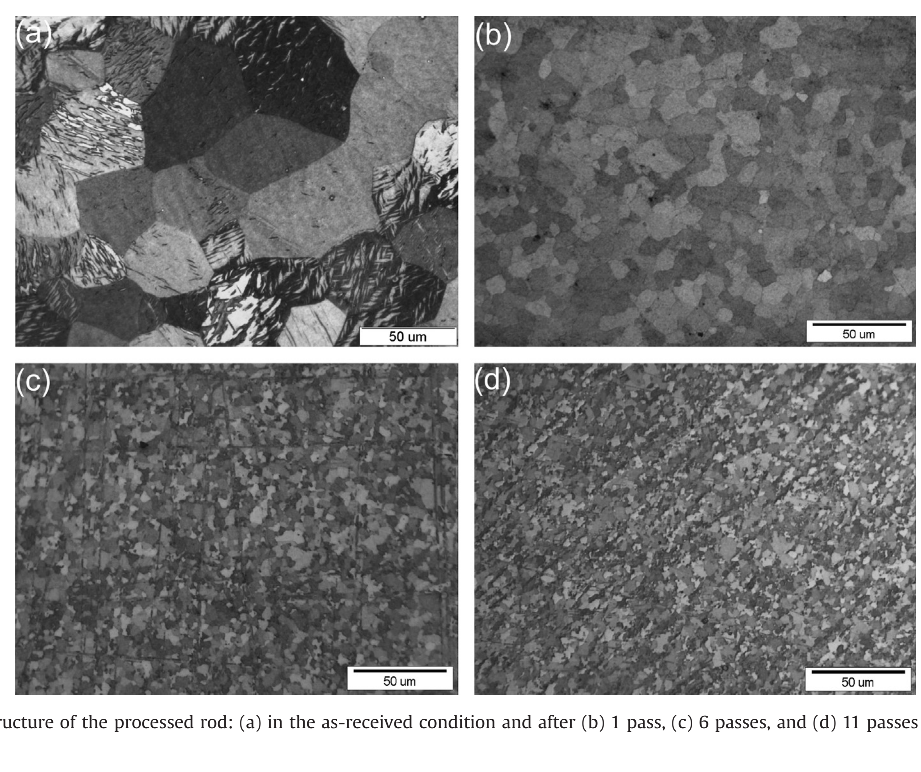

Fig. 2. Microstructure of the processed rod: (a) in the as-received condition and after (b) 1 pass, (c) 6 passes, and (d) 11 passes of screw rolling.