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Enhancement of room temperature stretch formability via grain boundary sliding in magnesium alloy

Hidetoshi Somekawa [a,*], Akihito Kinoshita [b], Kota Washio [b], Akira Kato [b]

[a] Research Center for Structural Materials, National Institute for Materials Science, 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047, Japan
[b] Advanced Materials Engineering Division, Toyota Motor Corporation, 1200 Mishuku, Susono, Shizuoka 410–1193, Japan

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A B S T R A C T

The enhancement method of room temperature formability was investigated by controlling the rate-controlling mechanism. Wrought processed pure magnesium and its alloys (Mg-Al-Zn and Mg-Mn alloys), which have a basal texture, were used in this study. Pure magnesium had two kinds of average grain sizes ($\sim$7 μm and $\sim$30 μm), and the other alloys were 20–30 μm in average grain size. The forming load vs. displacement curves in Erichsen tests were not influenced by the forming rate in the Mg-Al-Zn alloy; however, the fine-grained pure magnesium and Mg-Mn alloy showed a high forming rate dependence. The limited dome heights were 4.7, 2.0 and 5.2 for the fine-grained pure magnesium, Mg-Al-Zn and Mg-Mn alloys, respectively. Deformed microstructural observations revealed that, in the meso-grained pure magnesium and the Mg-Al-Zn alloy, deformation twins formed and were closely related to crack propagation. On the other hand, the formation of such deformed structures was difficult to identify in the fine-grained pure magnesium and meso-grained Mg-Mn alloy. The good stretch formability and high rate dependence of the fine-grained pure magnesium and Mg-Mn alloy resulted from the major contribution of grain boundary sliding to deformation during Erichsen testing. The addition of an alloying element, which plays a role in the enhancement for grain boundary sliding, is quite effective to improve the room temperature stretch formability.

1. Introduction

Magnesium and its alloys are the lightest among all of the conventional metallic materials; thus, they have great potential as the structural materials for weight reduction. Although they have been applied to structural parts, the secondary formability at room temperature ranges is poor as compared with other light-weight metallic materials, i.e., aluminum. This poor formability results from a hexagonal closed pack structure, in which the critical resolved shear stress (CRSS) of the basal plane is much lower than that of non-basal planes. The dislocations with $<c>$-components, e.g., pyramidal slip, are difficult to activate; the von-Mises criterion is not satisfied under such a condition. Above all, since the basal plane aligns parallel to the wrought process, such as in the rolling- or extrusion-direction, wrought processed magnesium and its alloy have a limitation of deformation along the $<c>$-axis direction.

In recent decades, many papers have tried to solve this issue; that is too say, to find an improvement method for room temperature formability, essentially, stretch formability [1–21]. Control of the wrought process method and/or thermal treatment enables the creation of a tilted or split basal texture; as a result, these alloys show good stretch formability [1–8]. The limiting dome height (LDH) of the texture controlled Mg-3Al-1Zn (AZ31) alloy is several times larger than that of the conventional AZ31 alloy [1,2]. The addition of a rare-earth element is also known to effectively enhance formability [10–13]. The wrought processed alloys containing rare-earth elements weaken or broaden the basal texture due to its recrystallization mechanism. In addition, since the difference in the CRSS between basal and non-basal planes becomes small, dislocations on non-basal planes are activated with the addition of a rare-earth element [22,23]. Hence, these alloys show good stretch formability; for instance, the LDHs of Mg-Zn-Ce and Mg-Zn-Y alloys correspond to those of competitive light-weight alloys [10,12].

It is well known that superplastic behavior refers to the ability to exhibit a large strain, the so called large elongation-to-failure in tension [24]. In addition, superplastic forming combined with other forming is recognized as a viable technique for fabricating a hard-to-form material into complicated shapes [25]. It is also accepted that the dominant deformation process of a superplastic flow is grain boundary sliding (GBS). Many kinds of magnesium

* Corresponding author.
E-mail address: Somekawa.Hidetoshi@nims.go.jp (H. Somekawa).

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alloys have shown superplastic flow and have observed the occurrence of GBS [26–31]. These results imply that the use of such a deformation mechanism, i.e., GBS, has significant capability to improve the stretch formability of magnesium alloys. However, GBS generally occurs at temperature ranges over about $0.5T_m$ (where T_m is the melting point), which is approximately of 460 K for magnesium. These high or intermediate temperature ranges are unfavorable from an industrial point of view.

On the other hand, recent studies about the room temperature plastic deformation of pure magnesium have reported a high strain rate dependence in uniaxial tensile testing [32,33] and high strain rate sensitivity around grain boundaries in nanoindentation creep testing [34]. In addition, the elongation-to-failure in tension is influenced by the strain rate and grain size; in particular, the elongation-to-failure in tension of fine-grained magnesium is over 100% in the low strain rate regimes [33,35]. Furthermore, the Hall-Petch breakdown due to the dominant occurrence of GBS is observed in a grain size of less than about 5 μm [33]. These results indicate that control of the forming condition, which GBS acts as the rate-controlling process, is likely to improve the room temperature formability; however, to the best of our knowledge, such a systematic investigation has not been reported yet. Thus, in this study, we investigated the stretch formability under both quasi-static and low forming rates using wrought processed magnesium and its alloys. We considered the possibility of enhancement for room temperature formability by controlling the deformation mechanism, i.e., the major contribution of GBS to deformation.

2. Experimental procedure

Magnesium with a purity of 99.96 wt%, Mg-0.65 wt%Mn ($\approx$ 0.1 at%Mn) alloy and Mg-3 wt%Al-1 wt%Zn (AZ31) alloy were used in this study. Two kinds of pure magnesium, which has a different grain size, were prepared; one was extruded at a temperature of 423 K into a plate shape with a thickness of 2 mm in an extrusion ratio of 32 (hereafter denoted as, the fine-grained magnesium). For the other, the extruded pure magnesium (fine-grained magnesium) was annealed at 573 K for 24 h to coarsen the microstructure (hereafter denoted as, the meso-grained magnesium). As for the Mg-Mn alloy, the extruded alloy with a diameter of 40 mm was cut to the size of $30 \times 75 \times 10$ mm by machinery. This plate shaped alloy was kept in the furnace at 573 K for 3.6 ks in air. Then, this alloy was rolled with a reduction of 10% each time, to a thickness of 1 mm. The AZ31 alloy was a commercial rolled sheet with a thickness of 0.9 mm.

The initial microstructures of all the materials were observed via optical microscopy and electron backscatter diffraction (EBSD, in a field emission gun scanning electron microscope (FE-SEM) equipped with an EDAX-TSL EBSD system). The observed areas taken via optical microscopy were in the middle parts of the extrusion-direction and transverse-direction (ED-TD) or the rolling-direction and transverse-direction (RD-TD) plane. The areas for EBSD observation were at the center of the cross-section containing the normal-direction and transverse-direction (ND-TD) plane. The samples for these microstructural observations were polished with fine SiC papers (#600 and #1200), diamond slurries and an alumina silica slurry. The polished samples were, then, etched for several seconds in an acetic solution of 10 mL HNO_3 and 30 mL H_2O.

The stretch formability was evaluated by the Erichsen test without a lubricant at room temperature. The Erichsen test was performed under setting an Instron-type machine to control the forming rate and to record the load vs. displacement curve. The punch diameter was 10 mm, and the forming rate, i.e., the cross head speed, was set to be 1×10^{-3} and 1×10^{-5} mm/s. The specimens for the Erichsen test that has a dimension of 30×30 mm were cut directly from each material. Before the Erichsen test, in order to remove residual strains and contamination on the surface, the surface was slightly polished using fine SiC papers (#1200 and #2400) to a thickness of approximately 0.85 mm. The deformed microstructures of the specific stretch formed and the fractured samples were observed by an optical microscopy and an EBSD method. The observed areas of these specific samples were in the ND-TD plane. When the displacement reached 2 or 4 mm, the Erichsen test was stopped to prepare the stretch formed samples. The fracture sample was taken after the appearance of a load drop in the Erichsen test, indicated by arrows in Fig. 3. The sample preparation for these deformed microstructural observations was the same as mentioned above for the initial microstructural observation.

3. Results

Typical initial microstructures of all the materials are shown in Figs. 1 and 2. Figs. 1 and 2 are the optical images of the ED-TD or RD-TD plane and the inverse pole figures of the ND-TD plane, respectively. These images show that the microstructures do not consist of the deformed structures, such as the deformation twins and the shear bands. The average grain size, which was measured by the linear intercept method [36] using the optical images, is added in Fig. 1. Except for the fine-grained magnesium (Fig. 1(a)), three kinds of materials are found to have a similar range in the average grain size of 20–30 μm. The results of EBSD observation (Fig. 2) show that the microstructure of the TD-ND plane in the Mg-Mn alloy slightly elongated to the TD, but most of the grains are blue- or green-colored in all the materials. The inverse pole figure maps obtained by EBSD analysis from the ND are inset in each figure. As expectation, peak intensity accumulates in the {0002} direction. This indicates that the basal plane is parallel to the RD or ED, as is well-observed in the wrought processed magnesium and its alloys [33]. Although the maximum intensity, I, of the present materials has some ranges in between 7.3 and 10.6, the tendency of the basal texture is not influenced by the alloying element.

The load and displacement curves in the Erichsen test are shown in Fig. 3 for (a) pure magnesium and (b) magnesium alloys. The x- and y-axes have the same limit in both figures for ease of comparison. The applied load for stretch forming increases with the displacement, and then the load drop occurs, as marked by arrows. The displacement at the load drop point corresponds to the LDH. It is found that the load and displacement response of the AZ31 alloy is likely to be independent of the forming rate, while the applied load of the others (pure magnesium and Mg-Mn alloy) tends to be large with an increase in the forming rate. A typical example for the forming rate dependence of an applied load is indicated by a dashed line at the constant displacement, e.g., 1.8 mm. Fig. 3(a) shows that the difference in the applied load, ΔP, between the cross head speeds (forming rates) of 1×10^{-3} and 1×10^{-5} mm/s of pure magnesium increases with grain refinement. This implies that a large applied load is not necessary to perform stretch forming, when the forming rate is low and/or the grain structure (grain size) is fine. Interestingly, Fig. 3 also clarifies that three kinds of materials (meso-grained magnesium, Mg-Mn and AZ31 alloys) have similar average grain sizes; however, the forming rate dependence on an applied load, ΔP, for the Mg-Mn alloy is the largest among them. These results reveal that several factors, i.e., the forming rates, microstructures (grain size) and alloying element, affect the load vs. displacement response in the Erichsen test.

The LDH obtained from Erichsen tests is summarized in Table 1,