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# Superplastic microforming of Mg–9Al–1Zn alloy with ultrafine-grained microstructure

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Submicron grain size Mg–9Al–1Zn magnesium alloy was processed by severe plastic deformation through differential speed rolling (HRDSR), and exhibited excellent superplasticity and microformability at relatively low-temperatures. Beside the grain size, transition from superplastic flow to non-superplastic flow during microforming was proposed to be an important parameter greatly affecting the microformability. Under optimum forming conditions, the microformed components retained the fine grain size of the HRDSRed Mg alloy.

*Keywords:* Severe plastic deformation (SPD); Magnesium alloys; Superplasticity; Ultrafine-grained microstructure; Microforming

Technologies for scaling down the size of components to the microlevel have attracted recent attention. Much research is currently being conducted in metal-forming technologies for production of microparts, as this approach offers a major advantage for mass production with controlled quality and low cost. In microforming using polycrystalline metallic alloys, the most important factor is grain size since this limits the size of the microcomponents or the size of some of their geometrical features [1]. It has been shown that severe plastic deformation (SPD) techniques—forming processes that induce intense shearing during deformation—are effective in producing metals and alloys with submicron-sized grains. Ultrafine grains enhance mechanical properties at ambient temperature as well as superplastic forming ability at elevated temperatures by significantly lowering the processing temperature and flow stress, and increasing the upper limit of deformation rate for superplastic flow. Very recently, a new SPD method for introducing severe deformation in metallic alloys in the form of sheets has been developed. This method uses differential speed rolling with a high speed ratio (HRDSR) to introduce large shear straining [2]. As this process is continuous and does not require expensive dies, mass production of large surface-scale metal sheets with ultrafine-grained microstructure can be realized.

In the present study, the microformability of HRDSRed AZ91 magnesium alloy was evaluated. V-groove and pyramidal-type micropatterns of Si-based microdies were transferred to the HRDSRed AZ91 alloy using a microforging method. The relation between the micropattern size and grain size on microforming ability was examined. Finally, under the optimum microforming conditions found in the V-groove experiments, a complex shaped component composed of 1 and 16 μm size channels was fabricated to check its possible application for commercial use.

The initial material utilized in this study was 2 mm thick coarse-grained AZ91 alloy ($L = 22$ μm, where $L$ is the linear intercept grain size) prepared by hot extrusion. The alloy was subjected to HRDSR. The diameters of the upper and lower rollers were identical and the ratio of upper to lower roller speed was 3. The rolling was carried out at 473 K for a thickness reduction of 70% through a single rolling pass. After the HRDSR, the grain size was decreased to 0.5–0.8 μm [3]. AZ91 plates with a different grain size ($L = 3.6$ μm, EX AZ91) were independently prepared by hot extrusion and their mechanical properties and microformability were compared with those of the HRDSRed AZ91. Strain-rate change (SRC) tests and elongation-to-failure tests were conducted to examine the superplastic characteristics of the two materials. Tensile specimens of dog-bone geometry with a 5 mm gauge length cut along the planes coinciding with the rolling direction were used for the tensile tests.

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Microforming tests of the HRDSRed AZ91 alloys were carried out using a microforging apparatus with micro V-grooved and pyramidal shaped dies made of (1 0 0) silicon. Silicon dies with a base angle of 70.6° with three different dimensions were fabricated by anisotropic etching. The throat width ($W_d$) of the V-groove and the length of basal square of pyramids ($W_p$) were 30, 50 and 100, respectively ($W_d$ and $W_p$ are indicated in Fig. 2). A 5 mm × 5 mm Si die was placed on a 6 mm × 6 mm specimen with a thickness of 0.7 mm, and then the specimen and die were simultaneously heated to a forming temperature in the range 493–573 K by a small electric heater. It took about 400 s to reach the target temperature. A period of 180 s was given to stabilize the temperature. Then, the specimen was subjected to compressive loading by at a fixed punch speed of 0.02 mm min$^{-1}$ until the maximum forming force of 550 or 700 N was reached. The maximum force was maintained for 60 s before unloading. The microformability of the HRDSRed and EX AZ91 alloys were evaluated by using a microformability index, $R_f$ ($=A_f/A_g$) [4], where $A_g$ is the cross-sectional area of the V-groove, and $A_f$ is the filled area of the microformed sample. Additionally, the radius of curvature ($\rho$) of the microformed pattern tip was used as another index. The $A_f$ and $\rho$ of the deformed specimens were measured by using a three-dimensional (3-D) profiler that provided a 3-D profile of the surface based on the principle of scanning white light interferometry. Microforming of the pyramidal-type patterns was selectively conducted by employing the optimal process conditions found from the V-groove microforming experiments. Finally, a complex microcomponent composed of 1 and 16 μm deep channels was fabricated at 553 K.

The double-log plots of flow stress vs. strain rate at different temperatures in the range 493–573 K obtained from the SRC results for the HRDSRed AZ91 and the EX AZ91 alloy are shown in Figure 1a. The plot indicates that there are two regimes, distinguished by different $m$ (i.e. the strain-rate-sensitivity exponent) values: high $m$ values of 0.5–0.8 and low $m$ values of ~0.2 are associated with the low and high strain-rate regimes, respectively. The HRDSRed AZ91 shows a higher upper limit of strain rate (associated with high $m$ values) than the EX AZ 91. For example, the strain rate above which $m$ becomes lower than 0.5 at 553 K is $8 \times 10^{-3}$ s$^{-1}$ for the HRDSRed AZ91, while it is $8 \times 10^{-4}$ s$^{-1}$ for the EX AZ 91. Another difference to note is that flow stresses of the HRDSRed AZ91 are lower than those of the EX AZ 91 in the low-strain-rate regimes. According to previous deformation-behavior analyses of the HRDSRed AZ91 [3], Coble creep and grain boundary diffusion-controlled grain-boundary sliding (GBS), which are grain-size sensitive, are competing at low strain rates and grain boundary diffusion-controlled GBS and pipe diffusion-controlled slip creep, which are grain-size insensitive, are competing at high strain rates. Figure 1b shows the elongations to failure (%) of the HRDSRed and EX AZ91 alloys plotted as a function of temperature at $3 \times 10^{-4}$ and $1 \times 10^{-3}$ s$^{-1}$. The values of $m$ measured from the data of the SRC tests in Figure 1a are also plotted in Figure 1b. Both alloys show a similar trend of total elongation increasing with decreasing strain rate and increasing temperature, except at 573 K where ductility begins to decline. This tensile behavior can be explained in terms of the $m$ value that increases with increasing temperature and decreasing strain rate but decreases at 573 K. By comparison with the EX AZ91 alloy, the HRDSRed AZ91 alloy with higher $m$ values shows noticeably larger elongations, including a maximum tensile elongation of 546% at $1 \times 10^{-3}$ s$^{-1}$ and 553 K. Furthermore, it shows low-temperature superplasticity by exhibiting large elongations over 400% at relatively low-temperatures (473 and 523 K).

The variation of $R_f$ and $\rho$ values for the HRDSRed AZ91 and EX AZ91 alloys deformed on the V-grooves with different sizes under the maximum loading force of 550 N are shown as a function of temperature in Figure 2. Two important findings can be inferred from the plot. First, for all the V-groove sizes, $R_f$ ($\rho$) increases (decreases) with temperature up to 553 K and then decreases (increases) at 573 K. This behavior resembles the tensile elongation behavior shown in Figure 1b. The grain sizes of the HRDSRed samples just prior to loading for microforming were examined by optical microscope to check for possible grain growth during the sample heating period. The sizes were ~1, ~1 and 13.5 μm at 523, 553 and 573 K, respectively, indicating that grain coarsening was markedly accelerated at 573 K. Therefore, the decrease in the $R_f$value at 573 K

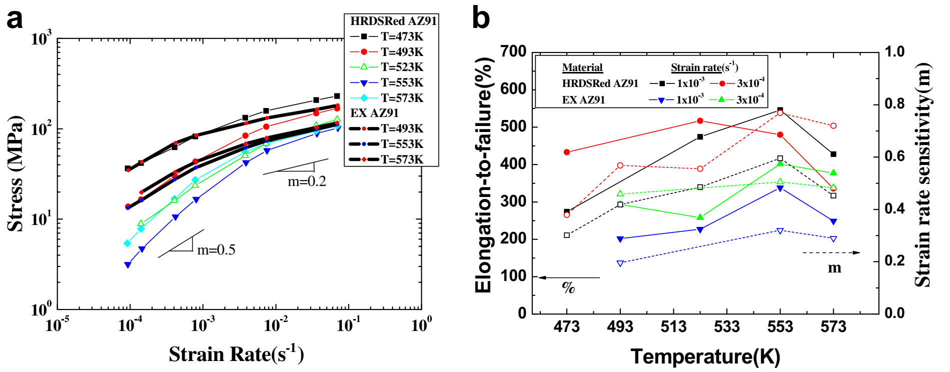

**Figure 1.** (a) The relationship between flow stress and strain rates for the HRDSRed AZ91 and EX AZ91 at various temperatures, (b) Tensile elongations and $m$ values of the HRDSRed AZ91 and EX AZ91 as a function of temperature at different strain rates.