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# Achieving high strength and high ductility in magnesium alloys using severe plastic deformation combined with low-temperature aging

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A novel method of fabricating age-hardenable magnesium alloys with high strength and high ductility was developed using high-ratio differential speed rolling combined with low-temperature aging. The ultrafine-grained Mg–9Al–1Zn alloy processed by this technique exhibited a high yield stress of >400 MPa and tensile elongations of 12–14%. The high strength could be attributed to grain size and particle strengthening effects, while the high ductility could be attributed to weakening of the basal texture component.

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There are recent reports on achieving a good combination of high strength and high ductility in ultrafine-grained metals produced by severe plastic deformation (SPD) [1–4]. In the case of Mg alloys with a limited number of slip systems, however, in addition to the grain size and the second phase, texture also strongly influences the strength and ductility.

The most effective method of improving the ductility of Mg alloys is through modification of texture by rearrangement of the distribution of basal planes since the critical resolved shear stress (CRSS) for basal plane slip in Mg is ~100 times lower than that for non-basal plane slip. Equal channel angular pressing (ECAP), in which shear straining is induced by deformation at a sharp die corner, has been demonstrated to be useful in improving the ductility of Mg alloys through the formation of textures with high Schmid factors for basal slip [5–7]. For example, ECAPed AZ31 and AZ61 alloys showed remarkably improved tensile elongations of 45–55% [5,6]. This is in sharp contrast to the typical cases of body-centered cubic (bcc) and face-centered cubic (fcc) metallic alloys with abundant slip systems at room temperature, where a considerable drop in ductility after ECAP is mostly due to the significantly reduced-work hardening ability [8]. Because of the asymmetric texture characteristics developed in Mg alloys by ECAP, however, the plastic response is highly anisotropic and some directions exhibit lower ductility than their conventionally processed counterparts [9].

One effective way to improve the strength of Mg alloys in bulk form is through grain refinement. This is because the grain-size strengthening effect in Mg alloys is high, compared with other metals with bcc and fcc crystal structures, since Mg has a large Taylor factor. The ECAP process has been shown to be effective in refining the microstructure of Mg alloys, as in other metals, but strength was often decreased rather than increased [5,7,9]. This was attributed to the texture-softening effect being dominant over the grain-size strengthening effect in the typical range of grain sizes (1–3 μm) obtainable by multipass ECAP [5]. For this reason, it is hard to simultaneously obtain high strength and high ductility in Mg alloys using the ECAP technique. This is also true when accumulative roll bonding (ARB) [10,11] or equal channel angular rolling (ECAR) [12] are used.

In the present work, we propose the high-ratio differential speed rolling (HRDSR) technique combined with low-temperature aging as a means to produce

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ultrafine-grained Mg alloy sheets with controlled texture, which exhibit a superb combination of high strength and high ductility. The HRDSR technique has the great advantage of being suited to cost-effective mass production of ultrafine-grained materials in sheet form. In the HRDSR process, large shear deformation can be uniformly induced along the thickness direction of a sheet during a single rolling pass for a large thickness reduction [13]. According to finite-element simulation, the effective strain accumulated in AZ31 alloy during HRDSR for a thickness reduction of 70% is high as ~3.5, which is comparable to that accumulated after four ECAP passes [13]. The concept of low-temperature aging used in this study is similar to that used for the ECAPed 6061 and 2024 Al alloys studied previously by the current authors [14,15], where it was shown that age-hardenable Al alloys could be strengthened markedly as they were severely deformed in solute supersaturated state and then aged at low-temperatures near 373 K.

The material used in the present study was the 85 mm wide and 2 mm thick AZ91 plate (9% Al–1% Zn–balance Mg) prepared by hot extrusion. The linear intercept grain size of the extruded material was 30–40 μm. In the HRDSR, the diameters of the upper and lower rolls were identical and the ratio of upper to lower roll speeds was set at 3. A plate was solid-solutionized at 688 K for 20 h and then immediately quenched in water. The size of grains in the water-quenched (WQ) material was about 75 μm. The WQ-AZ91 plate was preheated at 473 K for 10 min. and then deformed between hot rolls with a surface temperature that was maintained at 473 K for a thickness reduction of 65% in a single rolling pass. The sample obtained following the above procedures will be called HRDSRed B hereafter. The HRDSRed B was subsequently aged at two different temperatures, 373 and 438 K, for different aging times. The HRDSRed B samples aged at 373 K for 5 h and 438 K for 2 h will be named HRDSRed $B_1$ and HRDSRed $B_2$, respectively. The as-extruded material without any preliminary heat treatment was also subjected to HRDSR at 473 K for the same thickness reduction of 65%. This material will be called HRDSRed A.

The development of textures during HRDSR and annealing processes was analyzed at the top and bottom surfaces of the rolled sheets ($s = 1.0, -1.0$). Three pole figures of {0 0 0 2}, {1 0 1 0} and {1 0 1 1} were measured up to a tilt angle of 75° using the Schultz reflection method, and the orientation distribution function (ODF) was calculated using LaboTex 2.1 commercial software.

Dog-bone shaped tensile specimens with gauge length of 10 mm were cut along planes coinciding with the rolling direction (0°) and at angles of 45° and 90° (transverse) to the rolling direction. Tensile testing was conducted at room temperature under constant cross-head condition at an initial strain rate of $10^{-3}$ $s^{-1}$. Foils for transmission electron microscopy (TEM) observations were mechanically polished to a thickness about 0.1 mm, and then thinned to perforation by an ion milling method. The thinned foils were examined using a JEM 2010FX transmission electron microscope operating at 200 kV and with a double-tilt stage.

Vickers hardness measurement on the TD (transverse direction) planes shows a significant increase in hardness after HRDSR. The hardness increase was more pronounced in the HRDSRed B ($H_v = 110$) compared with the HRDSRed A ($H_v = 95.2$). The HRDSRed B samples were aged at two temperatures: one is the normal aging temperature for AZ91 (i.e. 438 K) and the other is 373 K. The peak aging ($H_v = 113$) was achieved at 438 K after 2 h, while peak aging ($H_v = 117$) was achieved at 373 K after 5 h, indicating that low-temperature aging is more effective in strengthening the HRDSRed B, though a longer aging time is required. Further increase in aging time at 373 K decreased the hardness but the high hardness of $\sim 110 H_v$ could be maintained up to an aging time of 30 h.

Figure 1a shows the representative engineering stress–strain curves of the WQ-AZ91, the HRDSRed A, the HRDSRed B, the HRDSRed $B_1$ and the HRDSRed $B_2$ tested along the rolling direction (0°) and at angles of 45° and 90° to the rolling direction. The table in Figure 1a summarizes the tensile properties of the HRDSRed B aged at 373 and 438 K for different aging times measured in the 0° direction. For each aging condition, three tensile tests were conducted and the measured values were averaged. The following can be inferred from the plot and the table. First, the HRDSRed materials exhibit a good planar isotropy in strength and ductility compared with the WQ-AZ91. Second, the HRDSRed B (yield strength (YS) = 362 MPa, ultimate tensile strength (UTS) = 410 MPa) has a higher strength than the HRDSRed A (YS = 327 MPa, UTS = 394 MPa). Third, when the HRDSRed B was aged at 438 K, its strength increased with aging time until 2 h related to peak aging (YS = 384 MPa, UTS = 431 MPa). When the aging temperature was lowered to 317 K, the maximum strengthening was obtained after 5 h (YS = 410 MPa, UTS = 467 MPa). These tensile results are in good agreement with those from the hardness measurements. It is noteworthy that the strength of the HRDSRed $B_1$ is markedly high compared with those of AZ91 alloys processed by extrusion (YS = 130–364 MPa, UTS = 274–370 MPa) [16,17], ECAP (YS = 220–277 MPa, UTS = 298–425 MPa) [16,17], ARB (YS = 320 MPa, UTS = 378–405 MPa) [10] and powder-metallurgy (YS = 376 MPa, UTS = 432 MPa) [18]. While yield strength significantly increased after HRDSR, ductility largely decreased due to a severe loss of work-hardening capability. Aging, however, restored some ductility. The HRDSRed $B_2$ and the HRDSRed $B_1$ exhibit tensile elongations of 7–12% and 12–14%, respectively; these values are substantially larger than those obtained for AZ91 alloys processed by ECAP (2.5–6.5%) [16,17], ARB (2%) [10] and powder-metallurgy (6%) [18]. It is important to note that the current ductility improvement after aging was attained simultaneously with the strength improvement. As seen in Figure 1b, the extraordinary combination of high strength and high ductility in the HRDSRed $B_1$ clearly sets it apart from other SPD processed and conventionally processed counterparts.

The microstructures of the HRDSRed samples were examined by TEM and are shown in Figure 2a–d. All the HRDSRed samples exhibit equiaxed grains of submicron size. Round particles identified as β-$Mg_{12}Al_{17}$ preferentially exist at grain boundaries in the HRDSRed B, $B_1$ and $B_2$, while their distribution is rather random in the HRDSRed A. The mean linear intercept grain sizes

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