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REPORT

Ultrastrong Mg Alloy via Nano-spaced Stacking Faults

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Mg alloys are among the lightest alloys but they are usually weak. Here we report a new mechanism to make them ultrastrong while maintaining good ductility. Stacking faults with nanoscale spacing were introduced into a Mg–8.5Gd–2.3Y–1.8Ag–0.4Zr (wt%) alloy by conventional hot rolling, which produced a yield strength of ~575 MPa, an ultimate strength of ~600 MPa, and a uniform elongation of ~5.2%. Low stacking fault (SF) energy enabled the introduction of a high density of SFs, which impeded dislocation slip and promoted dislocation accumulation. These findings provide guidance for developing Mg alloys with superior mechanical properties.

Keywords: Stacking Faults, Mg, Strength, Ductility, Dislocations

Magnesium and its alloys have attracted extensive attention in the recent years due to their abundance, low-density, good castability and recyclability [1–4]. However, the application of Mg alloys has been substantially hindered by their relatively low strengths (tensile yield strength: ~100–250 MPa for commercial casting Mg alloys [5,6]) and limited ductility (elongation: 2–8% [5–7]) at room temperature. Outside of traditional precipitation control, Mg-alloy strengthening typically relies on two general approaches: non-traditional, esoteric processing and grain refinement. An example of non-traditional processing for high strength is rapid solidification/powder metallurgy, which was used to obtain a yield strength of ~600 MPa in a Mg–Zn–Y alloy with uniform distribution of long-period ordered structures [8]. While the resultant properties are remarkable, such unconventional processing technologies limit potential industrial application. In addition, the ultra-high strength is usually accompanied by marked losses in ductility [8].

The second general approach of grain refinement for strengthening has been used to obtain ultrafine (<1 μm) grains in Mg alloys [9–12]. The high concentration of grain boundaries (GBs) in the ultrafine-grained

microstructure provides barriers to motion of dislocations and consequently promotes the strength improvement. Nevertheless GB strengthening mechanism alone provided limited contribution to macroscopic yield strengths, which, in these reports [9–12], are typically less than 400 MPa. More importantly, refining the grain size is reported to suppress the propensity of deformation twinning which, in addition to dislocation slip is an important mechanism for enhancing strength and ductility [13–18]. Ultrafine-grained microstructures also suffer from strength reduction via grain growth at a relatively low temperature (0.32 T_m) [19], thus limiting the potential for further shaping or processing.

In this letter, we report a new mechanism for inducing ultrahigh strengths (yield strength: ~575 MPa, ultimate strength: ~600 MPa) and maintaining moderate ductility (uniform elongation: ~5.2%) via conventional processing (hot rolling) of a Mg-alloy (Mg–8.5Gd–2.3Y–1.8Ag–0.4Zr (wt%)) with relatively large grain sizes (13 μm). We posit that the introduction of a high density of stacking faults (SFs) with nanoscale spacing provides for a high density of barriers to block and pin dislocations and retention of work hardening for enhanced ductility.

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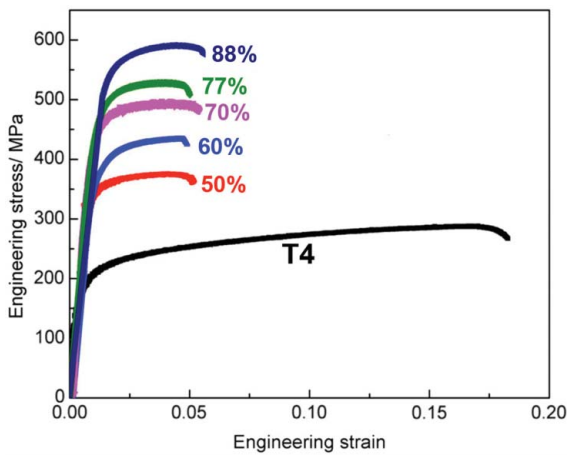


Figure 1. Engineering stress–strain curves of Mg alloy samples under T4 treatment and hot rolling with different thickness reductions (marked by the curves).

The casting procedure used to produce the Mg–8.5Gd–2.3Y–1.8Ag–0.4Zr (wt%) (hereafter referred to as “Mg alloy ingot can be found in reference [20]. The Mg alloy was then T4 treated (solution treatment at 500°C for 12 h in a vacuum furnace and quenched in room temperature silicon oil); the T4 conditions serve as a baseline for property assessment. Prior to each rolling pass, specimens were heated in a furnace at 450°C for 15 min and rolled on a conventional hot roller with a thickness reduction of <5% per pass to a total rolling reduction of 50%–88%. Rolled specimens were air-cooled to room temperature. Tensile test samples were cut on the rolling plane and pulled to failure in the direction parallel to rolling direction using a strain rate of $2.35 \times 10^{-3} \text{ s}^{-1}$. The gauge length and width of the testing samples are 7 and 1 mm, respectively. The surfaces of gauge region were polished to mirror-like surfaces before tensile testing and at least three samples for each rolling parameter were tested. For transmission electron microscopy (TEM) studies, specimens were gently polished and then ion milled to perforation on a cold stage with low angle and low-energy ion beam, and microscopy was conducted on a JEM-2000FX and a JEM-2010F operating at 200 kV.

Figure 1 shows the tensile engineering stress–strain curves of the Mg alloy samples after T4 treatment, and after hot rolling to different thickness reductions. Both the tensile yield strength and ultimate strength of the rolled samples increase as the rolling strain increases. At 88% rolling reduction, the grain size was refined to $\sim 13 \mu\text{m}$, and the maximum tensile yield strength and ultimate strength reached 575 and ~ 600 MPa, respectively, which are more than two times the values of the T4 treated baseline. Compared with the yield strength and uniform elongation values of various Mg alloys [9,11,21–54] as shown in Figure 2, the current result is an extraordinary combination of ultrahigh strength and reasonably good ductility. Importantly, the elongation to failure of

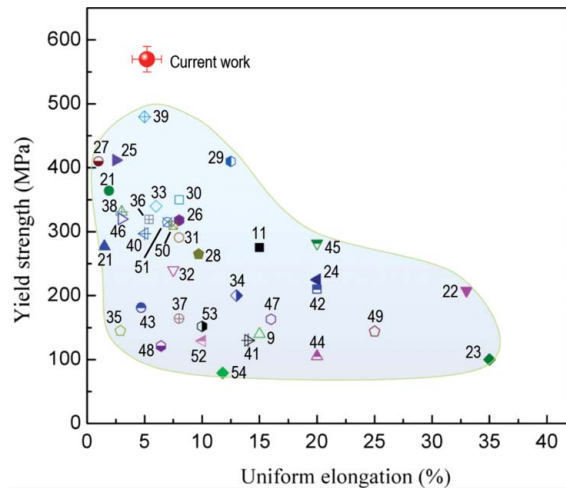


Figure 2. Summary of room temperature tensile yield strength and uniform elongation of previous reports and our current work. The criteria used to summarize and reproduce this figure are (1) only room temperature uniaxial tensile test results were included, (2) the gauge length of tensile test sample was larger than 5 mm and (3) the stress–strain curve must be indicated in the individual report in order to reproduce the uniform elongation. The reference number is adjacent to the data point. Note: Ref. [8] reported a Mg alloy with a yield strength of 600 MPa, but its uniform elongation is not available.

the processed Mg alloy retains a moderate level (5–6%) without diminishing during the hotrolling process.

The TEM analysis was performed to probe the main microstructural features that are responsible for the observed strengthening in the hot rolled Mg–8.5Gd–2.3Y–1.8Ag–0.4Zr (wt%) alloy. It was observed that beyond 50% thickness reductions, basal plane SFs become the primary crystalline defects (see Figure 3). The (0001) basal plane SFs are further verified by the presence of streaking along the [0001] direction in the selected area diffraction pattern [55] (Figure 3(d)). The average spacing between adjacent SFs, d , decreases with rolling thickness reduction. At 88% thickness reduction, nano-spaced SFs with an average spacing $d \sim 16$ nm were formed in most of the coarse ($\sim 13 \mu\text{m}$) grains. The formation of high density of SFs is due to the low SF energy (SFE) of this Mg alloy. It has been reported that both Gd and Y lower the SFE of Mg [56,57]. The SFs can be formed by the dissociation of a full dislocation into two partial dislocations on the basal plane during plastic deformation. The partial dislocations can be driven apart by applied stress to form a wide planar SF ribbon [58]. Our primary TEM observations also indicate that there was a transition stage in the rolling thickness reduction range of $\sim 30\%$ to $\sim 50\%$, in which deformation mechanism transitioned from dislocation slip to SFs-mediated process. Further study on this transition is underway and will be reported in the future.

Plotting the yield strength against the reciprocal of average spacing of SFs, $1/d$, reveals a linear relationship

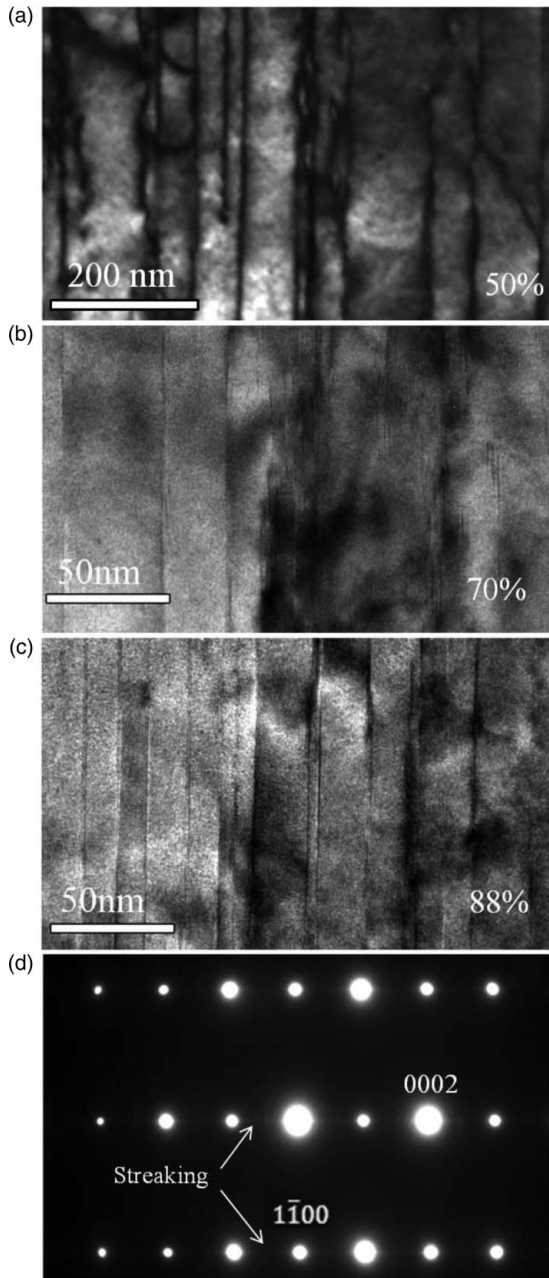


Figure 3. TEM images of Mg alloy samples with various rolling thickness reductions: (a) 50%, $d = 55$ nm, (b) 70%, $d = 25$ nm and (c) 88%, $d = 16$ nm. Image (d) is the SAD pattern of the 70% rolled sample in which the streaking verifies the basal plane SFs.

(Figure 4), i.e.

$$\sigma_{0.2} = \sigma_0 + \frac{k}{d}, \quad (1)$$

where d is the mean spacing between adjacent SFs, σ_0 is the total strength contributed by strengthening mechanisms other than SFs. This relationship is similar to the empirical Hall–Petch equation and its physical model is being developed and will be published in the future. In the current Mg alloy, a number of typical strengthening

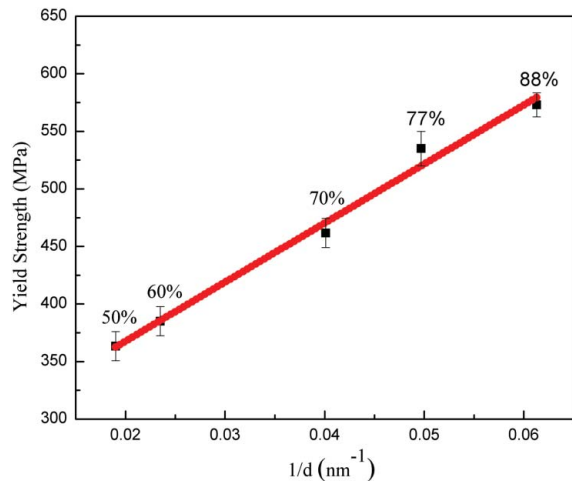


Figure 4. Yield strength vs the reciprocal of mean spacing between SFs of rolled samples with different thickness reduction. The number by each data point indicates the thickness reduction by hot rolling.

mechanisms such as solid solution strengthening, grain refinement, dynamic precipitation and textural strengthening are expected to be active [8,20]; however, strengthening contributions from these are not expected to be the primary cause of the ultrahigh strength observed.

The large slope of the linear correlation between the strength and $1/d$, the reciprocal of the SF spacing indicates that the SFs are very effective in improving the strength. Fitting the data in Figure 4 with Equation (1) yields $\sigma = 340$ MPa, and $k = 3780$ MPa nm. Therefore, introducing SFs with an average spacing of 16 nm increased the yield strength of the Mg alloy by 70% from 340 to 575 MPa. This indicates that SFs are very effective in improving the strength of Mg alloys. The fact that SFs increased the strength, but did not decrease the ductility (see Figure 1) makes SFs very attractive for improving mechanical properties of Mg alloys. This discovery is especially important for hexagonal close-packed (hcp) metals and alloys, because twinning, an effective approach for improving strength and ductility for face centered cubic (fcc) metals and alloys, becomes very difficult to activate in hcp metals when the grain sizes are below a few hundred nanometers [13,14], making it very difficult to combine the strengthening effects of refining grains and introducing twins.

SFs on basal planes are expected to provide similar effect on impeding dislocation movement as reported for nano-twinned electrodeposited Cu [17] As dislocations slip and encounter SF boundaries, they can either (1) cut or interact with SFs in order to move and facilitate plastic deformation or (2) accumulate around SF boundaries and accommodate a moderate strain hardening [59]

In order to probe the dislocation interactions with SFs, postmortem TEM observation (Figure 5) was conducted on tension-tested samples that were hot rolled

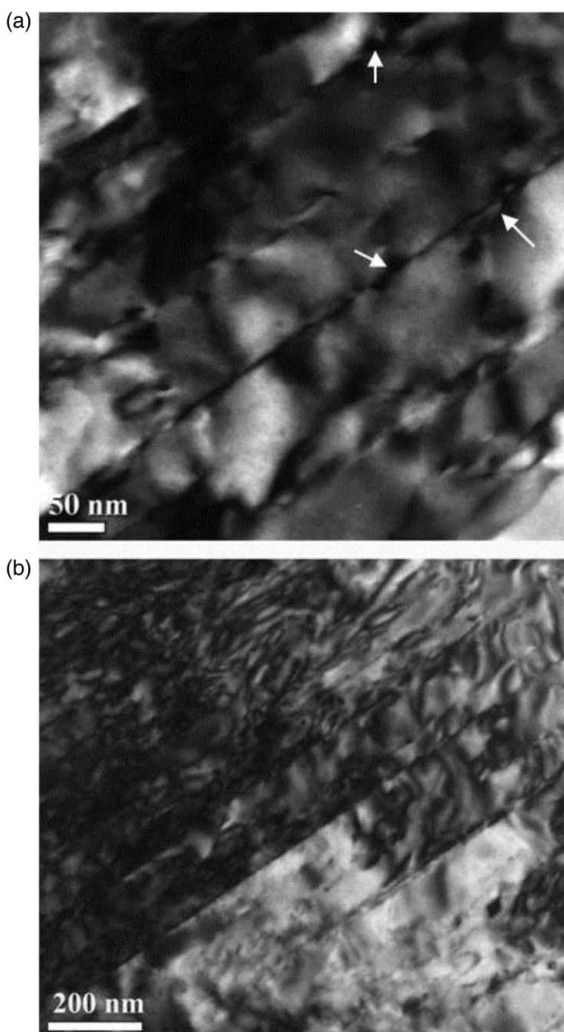


Figure 5. TEM images of 88% hot rolled sample after tensile test. (a) Fragments of SFs cut by the dislocations marked by white arrows (b) High density of dislocations was trapped between SFs.

to 88% to produce nano-spaced SFs Figure 5(a) shows the distinctly different features of fragmented SFs as compared to Figure 3(c). Some SFs have been broken into short segments (Figure 5(a)), and a high density of dislocations are trapped between SFs (Figure 5(b)). As shown in Figure 5(a), some SF segments are well aligned along a line, suggesting that they were most likely formed from a single SF that was cut by dislocations (marked by white arrows) slipping on pyramidal or prismatic planes.

Since the slip systems in hcp Mg are limited at room temperature, it is necessary to activate $\langle c \rangle$ and/or $\langle c + a \rangle$ dislocations to slip on the pyramidal or prismatic slip planes. Such dislocation slip will encounter SFs and cut SFs into segments, which are additional pinning sites to block dislocation motion. These dislocation-SF interactions are similar in some way to the dislocation-twin interactions in an fcc system [59] More investigation is needed to study these interaction details.

The blocking of dislocations by SFs and the cutting of SFs by dislocations will hinder the slip of dislocations, which consequently increases the yield strength. The high density of dislocations between SFs after tensile testing indicates that SFs are effective in blocking and accumulating dislocations, which will enhance strain-hardening rate and consequently help with ductility retention. This explains why no ductility reduction is observed with increasing strength when a high density of SFs is introduced into the Mg alloy.

In summary, conventional hot rolling was conducted on a T4treated Mg–8.5Gd–2.3Y–1.8Ag–0.4Zr (wt%)Mg alloy with thickness reduction up to 88%. Unprecedented strength and moderate ductility (YS \sim 575 MPa, UTS \sim 600 MPa and uniform elongation \sim 5.2%) were observed in the resultant coarse-grained alloy. TEM studies show that a high density of nano-spaced SFs are the main defects inside of the grains and their density increased as rolling thickness reduction increased. The strength of the processed Mg alloy was found to increase as the mean spacing between adjacent (SFs) decreased. Nano-spaced SFs are found to be tremendously effective in impeding the movement of dislocations and retaining strain hardening. Activation of nonbasal dislocations during tensile testing accounts for the detected moderate ductility, in addition to the capability of retaining strain hardening. It is expected that optimization of approaches that introduce a high density of nano-spaced SFs will enable other Mg alloys with concurrent high strength and good ductility.

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