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Tensile strength improvement of an Mg–12Gd–3Y (wt%) alloy processed by hot extrusion and free forging

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Abstract An Mg–12Gd–3Y (wt%) alloy was prepared by conventional casting method using permanent steel mold. Then this alloy was subjected to hot processing, involving hot extrusion and free forging. Tensile strength at room temperature can be improved, with the highest ultimate tensile strength (UTS) value of 390.2 MPa achieved by hot extrusion in comparison to that of as-cast alloy. Temperature dependence of tensile strength is distinguishable for the as-extruded alloy, while the relative stability in UTS values of the alloy after being freely forged should be ascribed to the inter-crossing among deformation bands located at various orientations and the accommodation effect of twining lamellas resulting from forging process on plastic deformation during tensile test at elevated temperatures. Further annealing after hot processing can only have adequate influence on the tensile strength of as-forged alloy. For the alloy freely forged and annealed at 523 K for 4 h, the highest UTS (441.1 MPa) at room temperature is found, which should be mainly related to an evolution from the original as-forged microstructure with subgrains to a more stable combination of large and refined grains through dynamic recrystallization during free forging, and the stress at offset yield YS (384.3 MPa) is also comparable to that relatively high value of 396.9 MPa after solution treatment and isothermal aging of the as-cast alloy.

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Introduction

Since pure magnesium's strength cannot meet the requirements for commercial application, scientists had to resort to the development of new magnesium alloys through the addition of rare earth (RE) elements to pure magnesium for the sake of different strengthening mechanisms, i.e. solution strengthening and precipitate strengthening, in addition to the traditional AZ and AM serial alloys. Among all recently recognized magnesium alloys containing RE elements, Mg–Gd–Y system alloys are thought to be more promising compared with other RE magnesium alloys [1–4], mainly due to its different precipitate morphologies [5]. Previous works by other researchers have demonstrated that magnesium alloys containing 10–12Gd and 0.4–0.5Y (wt%) showed a finally precipitated strengthening phase of platelet shape after serial phase transformations [5–8]. The relatively high strength demonstrated by this kind of alloy can be mainly ascribed to the triangularly arranged precipitates that precipitate initially and habitually at prismatic planes of magnesium matrix [1, 2, 9, 10]. Besides the second dispersed phase strengthening of RE magnesium alloy, solution strengthening and plastic processing effect should not be underestimated, because over 10 wt% RE elements in matrix will attain considerably recognized strengthening effect [11, 12] and non-equilibrium structure resulting from plastic deformation can usually perform dynamic recrystallization (DRX), thus grain refinement undergoes to some extents [13, 14].

In most cases, thermal plastic processing can considerably improve the mechanical strength of metallic materials, mainly due to the elimination of casting defects and grain refinement. Therefore, a further plastic processing of RE magnesium alloy was deemed as an efficient way to obtain a new type of magnesium alloy with enough high strength, based on both the original high dissolution of RE elements into magnesium matrix and the subsequent microstructure improvement brought about by thermal plastic processing. Among various thermal plastic processing techniques, hot extrusion and free forging can be regarded as two more feasible ways in comparison with other techniques, e.g. rolling and equal channel angular pressing, when it comes to the batch production of structural parts for commercial application. Previous works had been greatly concentrated on the precipitation sequence confirmation, characterization of various intermediate phases and strengthening mechanism analysis, but strengthening effects particularly after hot extrusion and free forging had never been accomplished for Mg–Gd–Y alloys [15–23]. Because characterization of strengthening phase had been clearly studied [1, 2, 9, 10], this paper deals mainly with tensile property improvement of this kind of alloy through hot extrusion and free forging. In addition, deformation mechanism of the alloy subjected to the two processing methods will be summarized and compared with the help of surface observation of specimens deformed under elevated temperatures.

Experimental procedures

The material used in this work is an Mg–12Gd–3Y (wt%) alloy, which can be obtained through conventional casting process using permanent steel mold. Smelting temperature is kept at 1,023 K in an electric resistance furnace with a protective atmosphere containing 99.9% (v/v) high-purity nitrogen and 0.1% (v/v) SF_6. Pouring into permanent mold is performed at 963 K, with mold temperature kept at 503 K after preheating. Real chemical composition by inductively coupled plasmas is Mg–12.37Gd–2.79Y–0.45 Zr (wt%), implying that the alloy studied hereinafter can be simply designated as Mg–12Gd–3Y alloy for the convenience in narration. Ingots obtained are subjected to roughly machining to remove surface oxidation and a suitable dimension of 200 mm in height and 150 mm in diameter for hot extrusion and free forging processing. Ingots are then at first solution treated at 803 K for 24 h in a steel tube sealed with sulfur powder for combating oxidation and ignition. After homogenization at this temperature, ingots are quickly taken out from the furnace and laid down on the hammering block, in order to keep ingot temperature not lower than 750 K. Free forging in this experiments is only done on the side face of each ingot, with 135 mm in final diameter (forging ratio roughly equals to 1.2), hammering head velocity at zero load is 2.6 m/s. Hot extrusion was performed also after that the as-cast ingots had been solution treated and air-cooled to room temperature. A horizontal hot extrusion machine was employed in this experiment, with the temperature of extrusion chamber kept at 633 ± 1 K and the pushing speed of cylinder plunger maintained at 3–4 cm/s. Extrusion ratio calculated from the transverse areas before and after extrusion is 9.8:1.

Rectangular tensile specimens are directly taken by electric sparkle line-cutting from ingots after extrusion and forging, with 8-mm long, 1.8-mm thick and 3.6-mm wide in gauge section, respectively; specimens' annealing is performed in an electric resistance furnace with temperature kept at 523 K for the as-forged specimens and 573 K for the as-extruded specimens, and a deviation in temperature change lower than 1 K. For SEM observation (using microscope S3400n from HITACHI), tensile specimen is firstly polished, then strained to different strains under various temperatures ranging from 293 to 523 K and at an initial strain rate of 0.03 s^{-1} on a universal CSS-55100 testing machine, and finally sprayed with alcohol solution and unloaded quickly to preserve the microstructures under various conditions. Separate tensile tests were performed under 293, 373, 423, 473 and 523 K. Polished samples are etched with 5% (v/v) nital solution to get optical micrographs on an OLYMPUS BX-6 microscope.

Experimental results

Tensile strength improvement after hot extrusion and free forging

As shown in Fig. 1, hot extrusion and free forging influence dramatically the tensile behavior both at room temperature and at higher temperatures (523 K). For the alloy under three different states, hot extrusion brings about the highest strength (390.2 MPa) at room temperature, the freely forged alloy coming up with an ultimate tensile strength (UTS) value of 330.9 MPa in comparison to the

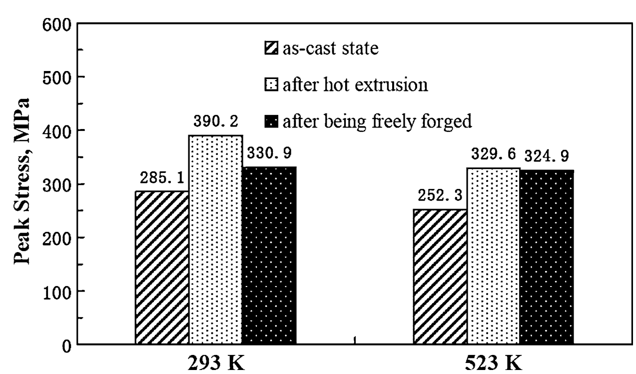


Fig. 1 Tensile strengths at room temperature and 523 K of the Mg–12Gd–3Y alloy before and after thermal processing

corresponding value (285.1 MPa) of the as-cast alloy. When tested under higher temperature (523 K), the UTS values of all three states decreased considerably, but states after being hot extruded and freely forged still give almost same and satisfactory results (329.6 and 324.9 MPa, respectively), suggesting that hot processing can make this kind of alloy much more suitable for real application under elevated temperatures.

Generally speaking, the microstructures of fine grains after hot processing will always present higher tensile strength and longer elongation compared with the as-cast alloy. This can be also confirmed from the experimental results that the alloy after hot extrusion underwent the largest elongation of 22.8% at room temperature in comparison with 11.0% of the as-cast alloy and 16.9% of the as-forged alloy. Improvement in plasticity after hot processing can be often easily observed, particularly when tested under elevated temperatures. This should be mainly ascribed to the fact that the finer and evenly distributed grains will facilitate grain boundary sliding (GBS) considerably, but the softening of the second strengthening phases exposed to elevated temperatures and the incapability of grains with different sizes and orientations in accommodating stress concentration during tensile test will most likely give rise to premature rupture. The largest elongation 30.1% at 523 K was achieved by the as-extruded alloy. However, for the as-cast alloy, it was interesting to find that the elongation decreased slightly from 11 at room temperature to 8.6% at 523 K. Whereas the as-forged alloy was observed to be strained longer at a higher temperature, thus elongation at 523 K (22.2%) was higher than that tested at room temperature (16.9%).

Since heat treatment involving solution and aging treatments can usually bring about relatively high strength compared with that under as-cast state due to precipitation strengthening of dispersed second phase and annealing after hot processing is often employed to obtained stable and fine microstructures which will significantly improve the tensile strength of metallic material, tensile strengths for alloys both under as-cast and hot processed states after heat treatment are compared in Fig. 2. In the case of as-cast alloy, the UTS value increased by 44.2% from 285.1 in as-cast state to 411.3 MPa after solution treatment at 773 K for 8 h and aging at 573 K for 10 h. Previous lots of trial experiments had proved that annealing of the extruded alloy either at higher temperatures for short period or at lower temperatures for long time will result in considerable grain growth, thus annealing of the extruded alloy was performed at 573 K for 30 min. But no significant improvement in strength of the alloy after hot extrusion and annealing is noticed. This can be briefly explained as that there is a precipitation sequence of Mg–Gd–Y alloy as assumed by some researchers, complete aging cannot be

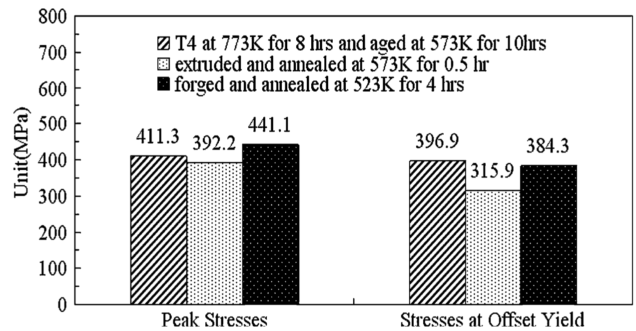


Fig. 2 Tensile strengths at room temperature of the Mg–12Gd–3Y alloy after thermal processing and heat-treatment

achieved after only a short period of annealing, but increase in both temperature and length of isothermal treatment can only result in grain growth but no improvement in tensile strength. For the alloy freely forged and annealed at 523 K for 4 h, the highest UTS is found which should be only related to the change in microstructure of the alloy subjected to free forging and annealing, and the stress at offset yield (YS, 384.3 MPa) is also comparable to that relatively high value of 396.9 MPa after solution treatment and aging of as-cast alloy (Fig. 2).

Temperature dependence of tensile strengths before and after hot processing

Temperature dependence of tensile strengths of all three states is apparent as demonstrated in Fig. 3. The UTS falls rapidly for the as-cast alloy, from 285.1 at room temperature to 239.3 MPa at 373 K. It is interesting to find that the UTS increases slightly under temperatures ranging from 373 to 473 K. Further increase in temperature from 473 to 523 K did not have significant effect on the UTS (Fig. 3a). The values of the YS show almost the same changing tendency as the UTS, although the lowest value (153.3 MPa) is found at 373 K for alloy in as-cast condition. This abnormal change in tensile behaviors can mainly attributed to the fact that slight increase in temperature will facilitate basal slip in as-cast Mg–Gd–Y alloy that contains triangularly arranged precipitate inhibiting non-basal slip at elevated temperatures. But ease in basal slip can also bring about dislocation piling-up and stress concentration at grain boundaries, so pre-mature fracture becomes inevitable in this case. Nevertheless, further increase in temperature will activate prismatic and pyramidal slip systems, making non-basal slip become much easier to some degrees. However, the unique precipitation placed at habit planes (initially at prismatic plane of magnesium matrix) of this Mg–Gd–Y alloy still plays an important role in preventing dislocation from further climbing and cross-slipping on non-basal planes, and this needs further investigation on deformation

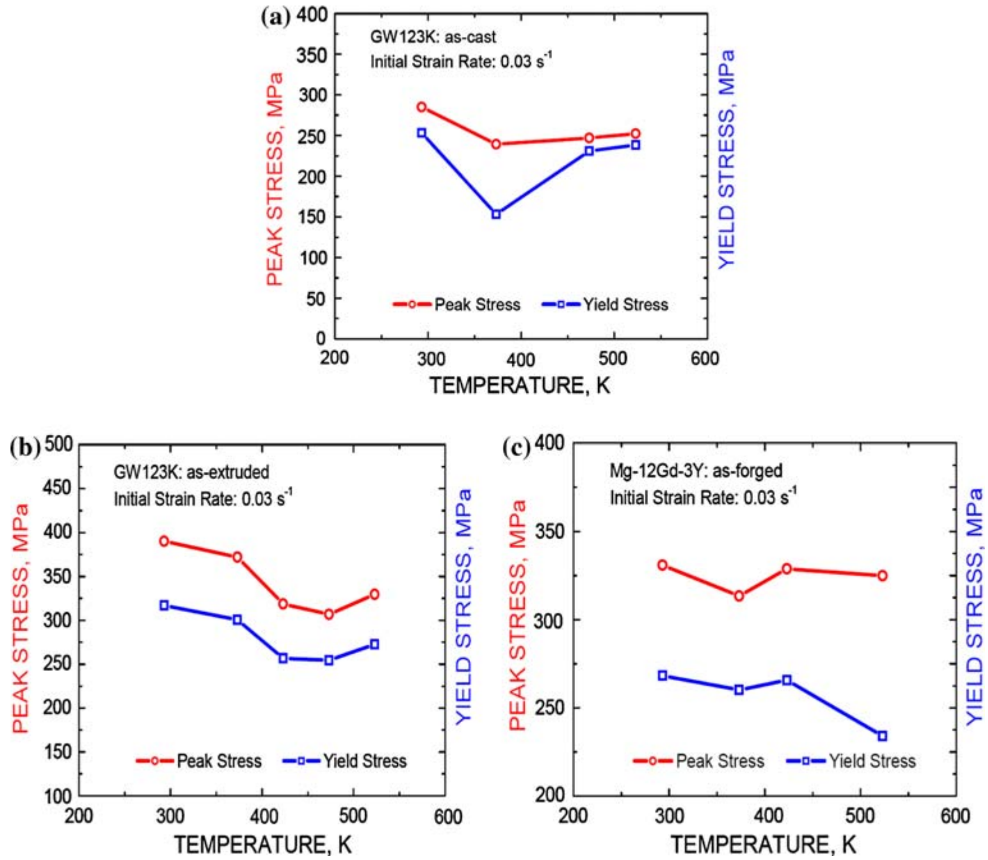


Fig. 3 Temperature dependence of tensile strengths of Mg–12Gd–3Y alloy before and after hot processing: (**a**) as-cast; (**b**) as-extruded; (**c**) as-forged

mechanisms of this kind of alloy under different experimental conditions.

In the case of the alloy after hot extrusion, a steady decrease both in UTS and in YS is found when temperature increasing from room temperature to 473 K (Fig. 3b), except that a slight increase both in UTS and in YS occurs after temperature getting higher than 473 K, reminiscent of substantial contribution of precipitate to prevent non-basal slip at elevated temperatures. Moreover, hot extrusion is usually considered to give out fine grains and line textures in which basal planes of most grains are aligned parallel to the extrusion direction. Thus, GBS might perform easily, and it will not be difficult to understand why there is a steady decrease both in UTS and YS when increasing temperature from room temperature to 473 K. For the alloy subjected to free forging, the UTS shows the lowest value (313.4 MPa) at 373 K. Then it increases to 328.8 MPa at 423 K (Fig. 3c), showing almost the same changing characteristic as that of as-cast alloy. Further increase of 100 K in temperature will not affect the UTS considerably, but has heavy influence on the YS, which decreases from 265.7 at 423 to 234.0 MPa at 523 K. Because precipitates had been dissolved almost totally into matrix through solution treatment before free forging to avoid crack initiation at the interface between the matrix and the strengthening phases, tensile strengths of the alloy processed by free forging as shown in Fig. 3c should be mainly associated with the microstructure evolution as a result of free forging and the deforming mechanism of different deforming units within the matrix of as-forged alloy, such as deforming bands at various orientations and twining lamellas, etc.

Discussions

Fracture mechanism of as-cast alloy and necessity for hot processing

As discussed above, changes in tensile strength are closely related the microstructure evolution before and after hot processing. Thereafter, it is necessary and easier to perform SEM observations on the deforming behavior of all the three states when strained under room and elevated temperatures to explain different deforming mechanisms briefly and efficiently.

A typical microstructure of Mg–12Gd–3Y alloy in as-cast state is shown in Fig. 4a, within which some white second phases composed mainly of Mg_5Gd and $Mg_{24}Y_5$ (β phase) at random ratios [10] widely exist and fracture will happen to initiate from these sites under elevated temperatures (Fig. 4b). Thus, these β phases should be responsible for the comparatively lower strength under

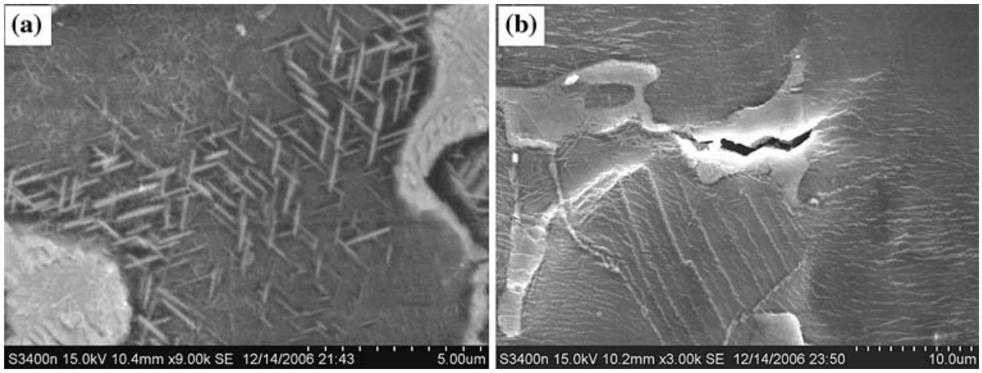


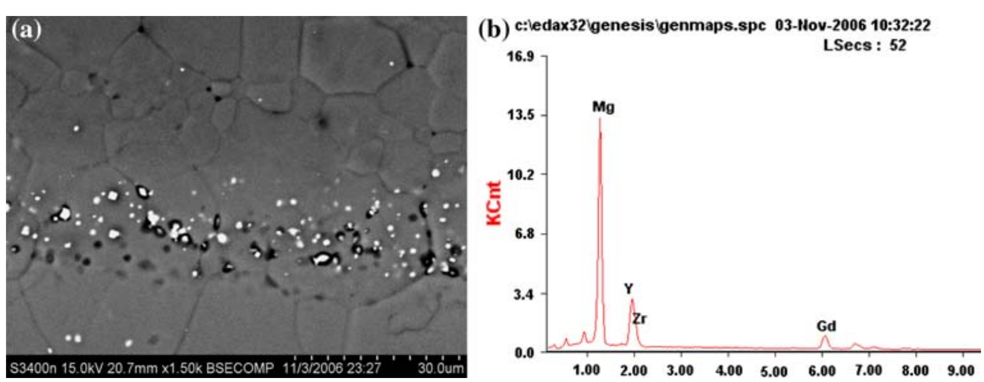
Fig. 4 Mg–12Gd–3Y alloy in as-cast state: (**a**) β phase and neighboring platelet-like precipitates; (**b**) slip lines on a sample strained under 523 K and an initial strain rate of 0.03 s^{-1}

room and elevated temperatures in comparison to the states after being hot extruded and freely forged [24–26]. This phenomenon can be explained as that these brittle β phases can easily precipitate through conventional casting method, but those platelet-like phases, which precipitate gradually afterwards from supersaturated solution (SSS), are selectively located on habit planes (initially at prismatic planes) and arranged triangularly, and they can really have much more legible strengthening effect for matrix enhancement compared with previously precipitated bulk β phases [27]. It can be seen that basal slip was performed smoothly in grains without either bulk β phases or platelet-like phases that precipitated afterwards from α matrix; whereas in grains embedded with β phases and or platelet-like precipitates, basal slip can only be seen far from strengthening phases and wavy non-basal slip lines emerged consequently, suggesting that theses phases prevent lattice deformation and moving dislocation from basal slipping, therefore non-basal slip (and or cross slip) will have to occur afterwards. But, if non-basal slip is heavily prohibited by strengthening phases finally located at non-basal planes of magnesium α matrix, stress concentration will be inevitable, causing some phases to break prematurely as demonstrated in Fig. 4b.

Mechanical improvement through hot extrusion

A typical microstructure of Mg–12Gd–3Y alloy after hot extrusion is shown in Fig. 5a, in which average grain size was estimated by linear section to be less than 10 μm. Meanwhile, like some magnesium alloys subjected to hot extrusion, lots of tiny white particles (termed by some researchers as K mass point) were found aligned parallel to the extrusion direction, and the composition of randomly selected particles was demonstrated in Fig. 5b and was considered to be a mixture of Mg_5Gd and $Mg_{24}Y_5$ (β phase) at random ratios, no second strengthening phases in bulk morphology were found to exist within the microstructure. Because it has been shown for pure magnesium and for other magnesium alloys [28–30] that hot extrusion serves to produce a texture in which a majority of the basal (0001) planes lie parallel to the extrusion direction, any changes in tensile strength is anticipated to be the result of small grain size and texture in basal planes. Thus, it will be easy to explain why the highest UTS and YS values among all the three different conditions were achieved for the material after hot extrusion (Fig. 1). As shown in Fig. 6a, no cleavages in large scale were discovered, indicating that small grains are solely responsible

Fig. 5 SEM of as-extruded Mg–12Gd–3Y alloy: (**a**) microstructure after hot extrusion with fine precipitates along extruding direction; (**b**) EDAX analysis of a randomly selected fine precipitate



for high strength of as-extruded alloy at room temperature. Decreases both in UTS and in YS with increasing temperature for the as-extruded alloy can be also ascribed to the relatively small grain size. When the temperature increased, cavities growth and coalition become much easier compared with the test conditions at room temperature, so fractures at elevated temperatures feature with coarse and shallow dimples (Fig. 6b). It is widely accepted that when the average grain size is less than 10 μm GBS can occur and steady flow stress can be attained meanwhile. For this alloy after hot extrusion, these two phenomena were observed when strained at 523 K and a lower initial strain rate of 0.03 s^{-1}, as indicated by the two true stress and true strain curves in Fig. 7a. When strained at room temperature, strain hardening is obvious and strengthening due to fine grains is the dominant deformation mechanism, even basal slip can result in pre-mature fracture of tensile specimen (Fig. 7b). However, increasing temperature will relieve stress concentration, and grain boundary sliding can perform to large strains since basal slip is easier at higher temperatures (see straight slip lines in Fig. 7c). In addition, prismatic and pyramidal slip systems might be activated at elevated temperatures, so plastic deformation is accommodated by the multiplicity of slip mode [26, 31, 32]. Long period of exposure to higher temperature can often bring about grain growth. It is also the case for the as-extruded alloy in this experiment, microstructure after annealing at 573 K for 30 min is a

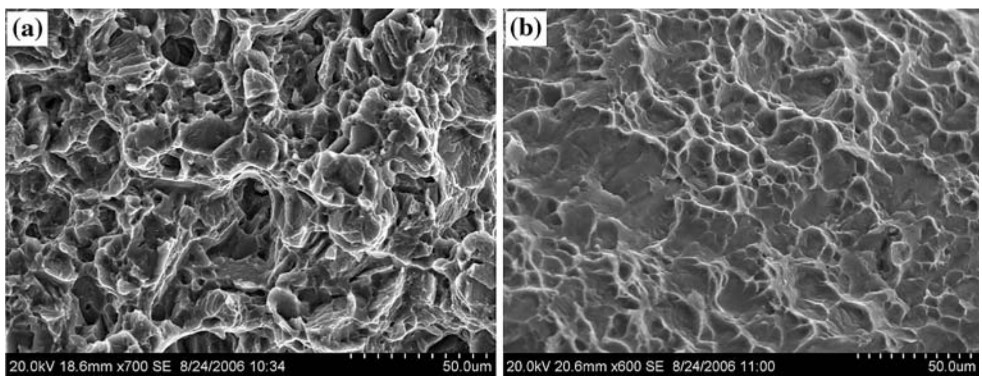


Fig. 6 Fractographs of as-extruded Mg–12Gd–3Y alloy strained at an initial strain rate of 0.03 s^{-1} and at: (**a**) room temperature; (**b**) 523 K

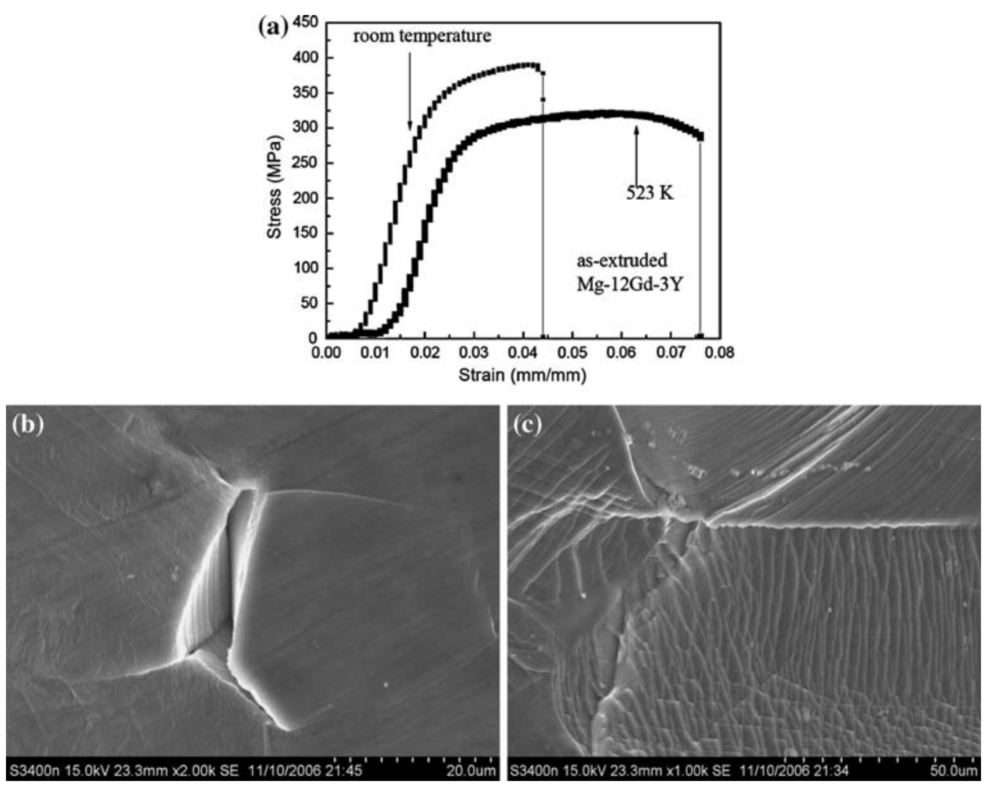


Fig. 7 (**a**) Stress–strain curves of an as-extruded Mg–12Gd–3Y alloy tested at an initial strain rate of 0.03 s^{-1} and recorded at room temperature and 523 K; (**b**) pre-mature fracture due to strain hardening at room temperature; (**c**) basal and non-basal slips under 523 K

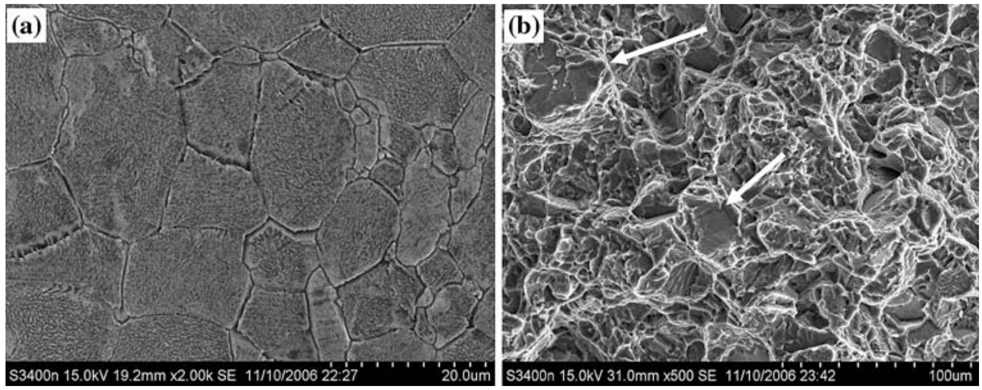


Fig. 8 Microstructure and fractograph of Mg–12Gd–3Y alloy after being extruded and annealed at 573 K for 30 min: (**a**) microstructure evolution after extrusion and annealing; (**b**) fractograph at an initial strain rate of 0.03 s^{-1} and at room temperature

mixture of fine and coarse grains as shown in Fig. 8a; fractograph consists of few lattice cleavage of some large grains with ductility decreasing as a result of grain growth (see white arrows in Fig. 8b).

Microstructure evolution after free forging and contribution of following heat treatment to further increase in tensile strength

As far as the Mg–12Gd–3Y alloy after being freely forged is concerned, its comparatively high strength should be ascribed to the inter-crossing among deformation (and or shearing) bands and the accommodation effect [22–24], which originates from lots of twining resulting from forging process, on plastic deformation during tensile test, as demonstrated in Fig. 9. Figure 9a is an optical micrograph of the Mg–12Gd–3Y alloy after being freely forged. Almost no formerly existing β phases and other strengthening particulates can be found within α matrix after long period of homogenization at 803 K. Clearly, adequate deforming (and or shearing) bands interact frequently with each other (Fig. 10a); twinning with characteristic long and straight lines also inter-cross with deforming bands besides that twinning of different orientations due to free forging

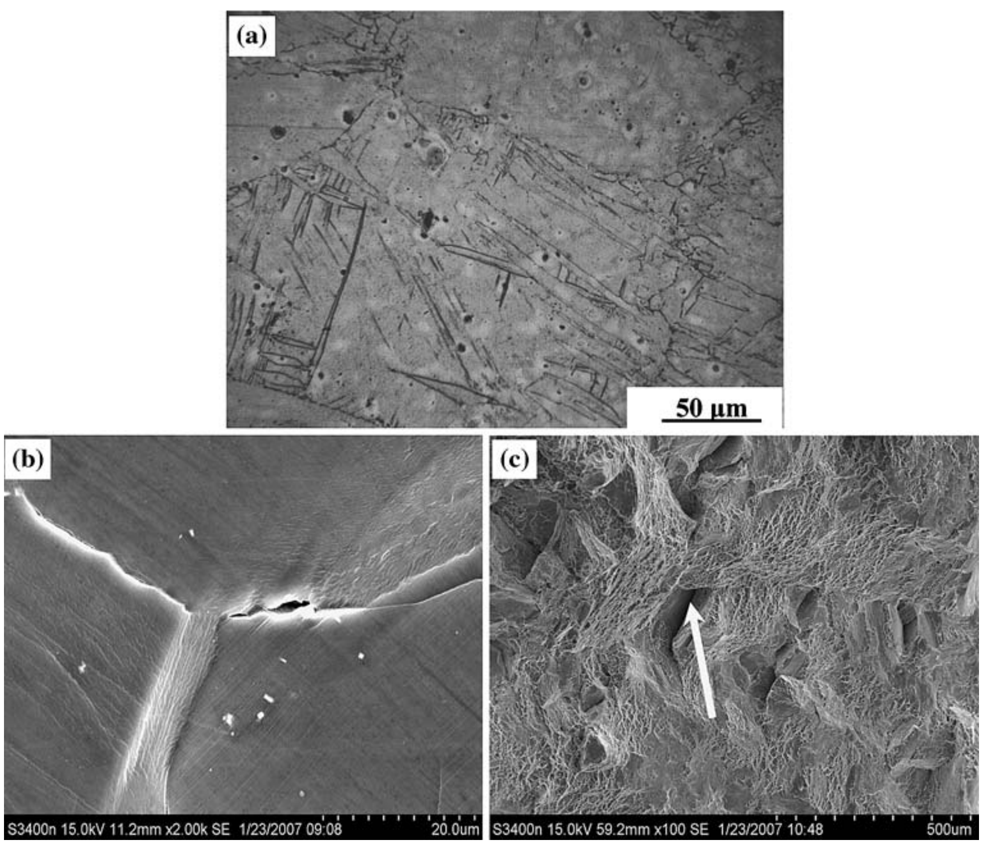


Fig. 9 (**a**) Optical micrograph of Mg–12Gd–3Y alloy after being freely forged; (**b**) deforming bands preventing or accommodating slip; (**c**) fractograph of specimen strained at 523 K

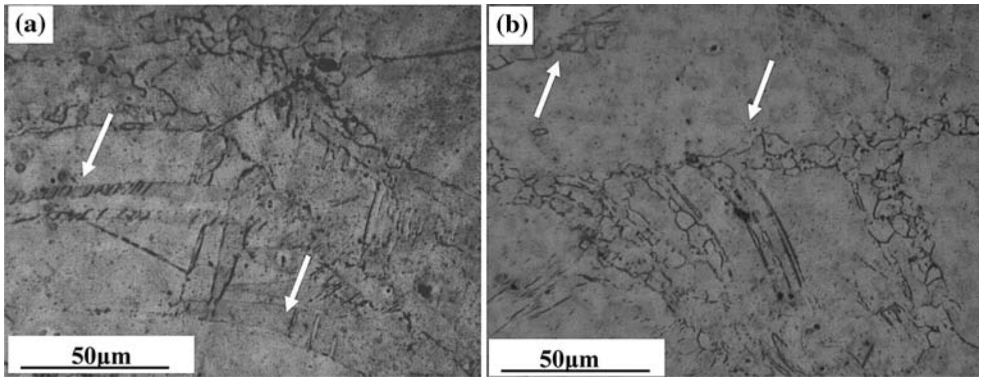


Fig. 10 Optical micrograph of the as-forged Mg–12Gd–3Y alloy after annealing at 523 K for 4 h: (**a**) bamboo structure indicative of inter-crossing of deforming bands or twinning lamellas; (**b**) DRX through grain boundary bulging and refined grain boundary mantle

also interact among themselves [23, 24]. Meanwhile, few grains of apparently small sizes emerge along grains boundary, it should be partially due to the annealing procedures after being forged, and it will facilitate plastic deformation to some extents under the mechanism of DRX (Fig. 10b). As revealed in Fig. 9b, deforming bands can act self-contradictorily, having influences on tensile behavior of the alloy studied. First of all, deforming bands at soft orientation can enhance lattice deformation; secondly, they will also result in premature fracture if located at hard orientation, though lattice slip can still performed adequately at moderate and high temperatures. Advantageous effect of deforming bands and twinning lamella on deformation at 523 K can be also apparently verified by the fact in Fig. 9b where fracture crack often initiates from grain boundaries, but not from the interface between two deforming units or between deforming unit and α matrix. Whereas, ductile fracture might be the controlling mechanism at elevated temperatures (523 K) when strain softening is not very difficult and particularly when some deforming bands (or twinning lamellas) are located at soft orientation to bridge the gaps among deforming bands, although there are really some fracture crack initiations at the interface between deforming units of different orientations (as shown by whiter arrow in Fig. 9c).

Further enhancement in tensile strength can mainly be related to the subgrain conversion, through annealing at elevated temperatures [11, 12, 21], which could often be associated with DRX during free forging [33, 34] and dislocation recovery (DRV) to some extents at 523 K. As shown by the white arrows in Fig. 10a, inter-crossing of deforming bands (or twinning lamellas) results in bamboo structures, which will accommodate plastic deformation due to grain refinement [14, 24, 26, 35], and small necklace grains are also discovered along boundaries of large grains. Figure 10b (see the white arrows) also demonstrates the effect of DRX by grain boundary bulging into α matrix and the apparent refined mantle composed of small grains acting as "lubricant" during GBS [14]. In consideration of the DRX effects on grain refinement, the inter-crossing among various deforming units (either deforming bands or twinning lamellas, as indicated by the bamboo structures within grains in Fig. 10a) during forging and annealing can be classified as discontinuous DRX or directly as twin DRX

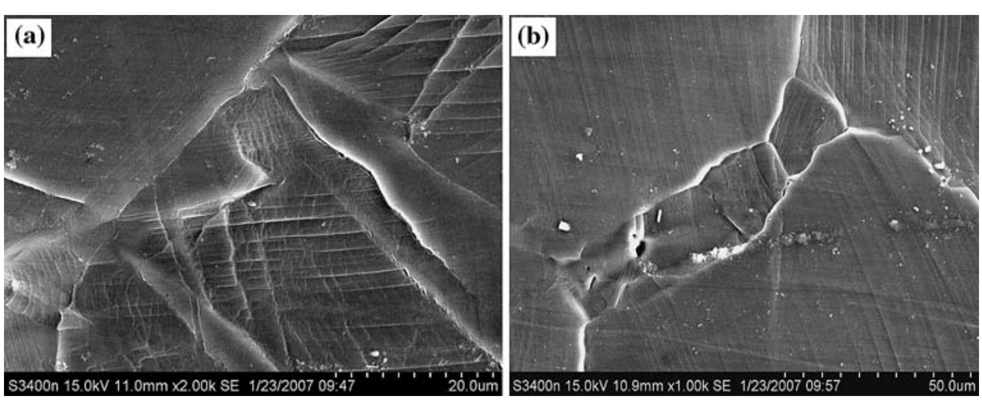


Fig. 11 SEM surface observation of the forged and annealed Mg–12Gd–3Y alloy when strained at 523 K: (**a**) DRX through multi-system slip; (**b**) refined grain boundary mantle in accommodation of stress concentration

[24, 26], and this mechanism can also be proved in Fig. 11a, in which small grains are formed partially due to multi-system slip in large grains strained under elevated temperatures, and deforming bands and twinning lamellas terminated by grain boundaries will help the formation of these small grains by preventing slip from mono-system slipping. While, the bulging process of grain boundaries usually associated with dislocation movement is thought to be continuous DRX (Fig. 10b) [31, 32, 35], when taking the nucleation place (usually at grain boundaries) and the rearrangement of different slip systems into consideration. The lubricating effects of grain mantle can be proved clearly in Fig. 11b, but cavities form also among small grains when deformation proceeds to a relatively large strain even at elevated temperatures, suggesting that grain orientation plays a very important role when the alloy is severely strained under elevated temperatures and DRX effects might be overcome by grain growth and cavity coalition under uniaxial straining.

Conclusions

1. Tensile strength of a cast Mg–12Gd–3Y (wt%) alloy can be improved through hot extrusion and free forging. The highest tensile strength at room temperature is attained by hot extrusion, through which grain refinement is achieved considerably, and the predominant strengthening mechanism at room temperature is refined microstructure. Tensile strength of as-forged alloy at room temperature is ranked at the second place in comparison with as-cast and as-extruded states, but stress at offset yield under room temperature of this alloy is comparable to that of in as-extruded condition.
2. Decrease in tensile strength of as-extruded alloy at elevated temperature is thought to be the result of combination of small grain size and texture in basal planes, which originate mainly from hot extrusion and make GBS much easier under higher temperatures.
3. For the Mg–12Gd–3Y alloy after being freely forged, its comparatively high strength and the relative stability in values of UTS should be ascribed to the inter-crossing among deformation (and or shearing) bands and the accommodation effect of twining lamellas resulting from forging process on plastic deformation during tensile test at elevated temperatures.
4. Following annealing after hot processing brings about apparently different influences on the tensile strength of alloy subjected to hot extrusion and free forging. Long period of exposure to higher temperature will only result in grain growth. Annealing of as-extruded alloy even at the optimal condition has almost no considerable effect on tensile strength at room temperature; whereas further enhancement to tensile strength of the Mg–12Gd–3Y (wt%) can be obtained through annealing at 523 K for 4 h. This improvement in strength is related to the subgrain conversion, which could often be associated with DRX and DRV to some extents at 523 K.

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