



# Precipitation behavior of bulk nanocrystalline GW103K alloy induced by surface mechanical attrition treatment



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## ABSTRACT

Precipitation hardening plays a significant role in improving the strength of Mg-RE (RE = rare earth) alloy due to the formation of precipitates. It is vital to control the precipitation behavior in order to optimize the mechanical properties of Mg-RE alloy. The present study explored one new way to promote the precipitation behavior of GW103K alloy by surface mechanical attrition treatment (SMAT). As compared with the conventional heat treatment, more precipitates could be formed under a shorter aging duration after SMAT, which results in finer grain size and higher micro hardness than the as-extruded one. The promotion of precipitation behavior is attributed to the high dislocation density induced by SMAT, which leads to a low nucleation energy and a high nucleation rate for the formation of precipitate.

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## 1. Introduction

After the precipitation hardening in Al-Cu was first discovered by Guinier [1] and Preston [2], a lot of heat treatment ways were studied to control the precipitation behavior in order to adjust the mechanical properties of metallic alloys. During aging treatment, the solutes in the matrix precipitate in the form of secondary phase due to the decrease of solubility of solutes, which could result in the effectively obstacle of dislocation motion and lead to the reinforcement of alloy [3,4]. Generally, the strengthened effect of aging under low temperature is better than the one under high temperature. The precipitate with homogeneous distribution and fine dimension is beneficial to the mechanical properties of alloy according to Orowan strengthening mechanism [5]. It requests the aging temperature should be as low as possible in order to reduce the diffusion rate and growth rate of precipitate, at the expense of

prolong duration. In addition, the grain size also hardly grows under low aging temperature, which won't hamper the effect of grain refinement strengthen [6,7]. Therefore, the precipitation hardening of alloy was always obtained by aging at low temperature with a long period.

However, the aging time at low temperature is too long due to the low dynamic driving force, which increased the energy consumption and the cost of production indirectly. In addition, the grain is easily getting coarsen when the aging period is too long, which reduces the strength of alloy [8–10]. Thus, it comes to be an important issue to find solution to accelerate the formation of precipitate under low temperature. Generally, according to the classical nucleation theory [11], The nucleation energy  $\Delta G$  for a spherical nucleus lists as follows:

$$\Delta G = \frac{16}{3} \pi \frac{\gamma^3}{\Delta G_v^2} - 0.8 \mu b^2 \frac{\gamma}{\Delta G_v} \quad (1)$$

Here  $\gamma$  and  $\Delta G_v$  is the surface energy and volume free energy of the precipitate, respectively.  $\mu$  and  $b$  is the shear modulus and Burgers vector, respectively. For the alloys without dislocation,  $\Delta G$  could be the maximum value since  $b = 0$ . It could be found that the  $\Delta G$  is reduced by the introduction of dislocation. Thus, the

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precipitates should be easier to nucleate with dislocation due to a lower  $\Delta G$ .

In addition, the nucleation rate is also obtained from the classical theory of nucleation as follows:

$$N = \rho^{1.5} \frac{8\pi\gamma^2 DC_{Gd}}{a_0^4} Z \exp\left(-\frac{\Delta G}{kT}\right) \cdot \exp\left(-\frac{\tau}{t}\right) \quad (2)$$

Where  $\rho$  is the dislocation density,  $D$  is the diffusion coefficient of solute,  $C_{Gd}$  is the instantaneous concentration of Gd in the matrix of Mg,  $a_0$  is the lattice constant of Gd,  $Z$  is the Zeldovich factor ( $<1/20$ ) and  $k$  is Boltzmann constant. Precipitation on dislocations is assumed to be instantaneous and therefore, the incubation period ( $t$ ) is assumed to be zero. It could be clearly found that  $N$  increases with the augment of  $\rho$ . Thus, the aging precipitation behavior could be accelerated by introducing dislocations or grain boundaries due to the heterogeneous nucleation effect. Generally, grain refinement could improve the strength of alloys as a result of the acceleration of grain boundary and dislocation pile.

Alternatively, the optimization of the surface structure of materials is currently of great concern since most failures occur on the surface, such as fatigue, fretting corrosion, corrosion, wear, etc. As a result, the improvement of the surface properties would greatly enhance the overall behavior of materials. Surface mechanical attrition treatment (SMAT) can realize the nanocrystallization at the surface of various metallic materials [12]. During SMAT, a large number of balls vibrated on the surface of material at high frequency [13–15]. Thus, there forms depth-dependent microstructure in the material after SMAT. The nanocrystalline grains achieved by SMAT involve numerous dislocation and sub grain boundaries on the surface, which are far higher than that obtained by Severe Plastic Deformation (SPD) method [16], including equal channel angular pressing (ECAP) [17], high-pressure torsion (HPT) [18], accumulative roll bonding (ARB) [19]. As compared with SPD ways, SMAT was proved to be an effective way to induce sufficient dislocation without strong texture in magnesium alloy.

The present study explored the effect of SMAT on the precipitation behavior of GW103K, in attention to give one new way to promote the precipitation hardening efficiency of Mg alloy.

## 2. Experimental details

The investigation was conducted on four alloys with different process as given in Table 1. As-extruded Mg–10Gd–3Y–0.4Zr (GW103K) alloy with thickness of 1 mm was selected to be the base alloy, RE, due to its intense precipitation strengthening effect [20]. RS and RA was subjected to aging treatment and SMAT, respectively. RSA was subjected to the combination of aging treatment and SMAT. SMAT process was performed at vibrating frequency of 20 kHz using stainless steel balls for 10 min. The detailed procedures have been published in the previous literature [12,21]. Aging treatment was performed in one electric resistance furnace under the protection of the Ar atmosphere at 473 K. Transmission electron microscopy (TEM) observation was performed using a JEM 2100 microscope operated at 200 kV to observe the microstructures. The

**Table 1**  
Processes route on the experimental alloys.

Process route	Process 1	Process 2	Process 3
	Based extrusion	SMAT 10min	Aging treatment 473 K
RE	✓	×	×
RS	✓	✓	×
RA	✓	×	✓
RSA	✓	✓	✓

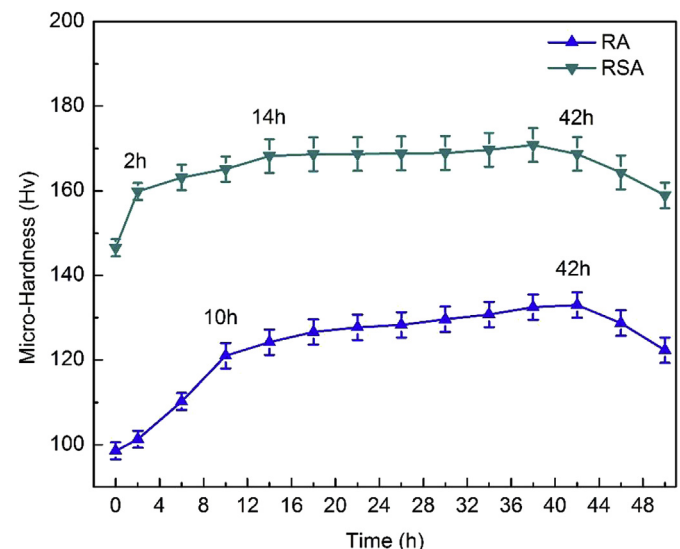
samples for TEM observations were prepared by mechanically polishing following the twin-jet electro polish. Micro hardness was tested on a SHIMADZU HVM-2T with the load of 200 g and the holding time of 15s.

## 3. Result & discussion

### 3.1. Micro hardness

Fig. 1 gives the micro hardness on the surface of RA and RSA. As shown in Fig. 1, the micro hardness of RSA is 146Hv, which is 48Hv higher than RA. The micro hardness of RA increases rapidly to 120Hv after aging for 10 h, and then slowly reaches the peak micro hardness of 127Hv after aging for 42 h. While the micro hardness of the RSA rises up to 159Hv at an express speed after aging only for 2 h, and reaches the peak micro hardness of 168Hv after aging for a shorter duration of 14 h. Such phenomenon indicates that the alloy after SMAT could reach the peak micro hardness with higher value under even shorter duration. It means that SMAT could accelerate the aging precipitation process of GW103K alloy. The dislocation and sub-grain boundary induced by SMAT could serve as the heterogeneous nuclei of precipitate. In addition, the micro hardness of RA rapidly drops to 118Hv with aging duration of 50 h after peak aging. While the micro hardness of RSA exhibits one steady value of 168Hv till the aging duration of 42 h, and then decreases to 158Hv with aging duration of 50 h after peak aging. Generally, the drop of micro hardness after peak aging is attributed to the growth and coarsening of precipitates [22,23]. The dislocation and sub-grain boundary could sever as the barrier of diffusion, which inhibits the growth and coarsening of precipitate [22,24]. Thus, the micro hardness of the alloy by SMAT could keep the consistent value up to 42 h.

Fig. 2(a) shows the cross-sectional micro hardness distribution of RA and RSA with aging for 14 h and 42 h at 473 K. The micro hardness of RE and RS are also given for comparison. The micro hardness of the alloys achieved by SMAT is obviously higher than that of as-extruded alloys due to the change of microstructure. The alloys after SMAT exhibit typical depth-dependent micro hardness, which is attributed to the gradient structure and the dislocation induced by SMAT. The micro hardness of the RS's center and surface has the value of 107Hv and 148Hv, respectively. The surface exhibits



**Fig. 1.** The aging harden curve on the surface of RA and RSA.

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