Thermal Stability of Nanocrystalline AZ31 Magnesium Alloy Fabricated by Surface Mechanical Attrition Treatment

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Abstract A nanocrystalline layer (NL) was fabricated on the surface of AZ31 magnesium (Mg) alloy sheet by surface mechanical attrition treatment (SMAT). The microstructure of the Mg alloy was characterized by optical microscopy, X-ray diffraction and microhardness test. The results showed that both the microstructure and microhardness of AZ31 Mg alloy sheet after SMAT revealed a gradient distribution along depth from surface to center. The thermal stability of the NL was investigated through characterizing the microstructure evolution during the post-isothermal annealing treatment within the temperature range from 150 to 250 °C. The NL exhibits a certain degree of thermal stability below 150 °C, while it disappears quickly when annealing at the temperature range of 200–250 °C. The grain growth kinetics of the nanocrystalline of AZ31 Mg alloy induced by SMAT was investigated. The activation energy of nanocrystalline AZ31 Mg alloy was obtained with a value of 92.8 kJ/mol.

KEY WORDS: Magnesium alloy; Thermal stability; Surface mechanical attrition treatment; Nanocrystalline structure; Gradient structure

1 Introduction

Nanocrystalline materials have been paid a continuous attention over the past decades due to their excellent mechanical and other physical properties [1].

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Nanocrystalline structure exhibits high strength owing to the high density of grain boundary and superplasticity at high temperature due to the slip of grain boundary [2]. Diffusivities of nanocrystalline structures are higher than those of the conventional coarse-grained counterparts as well [3]. In the past decades, many SPD methods have been reported to fabricate nanocrystalline, such as rolling, equal channel angular pressing (ECAP) [4], high-pressure torsion (HPT) [5] and high-energy shot peening (HESP) [6]. However, these methods could fail to balance high strength and ductility of materials [7]. Fortunately, recent researches have demonstrated that the gradient structure of the grain size usually renders outstanding balance of high strength and ductility in nanostructured materials induced by non-homogeneous deformation processes with large strain gradients [8, 9]. Recently, a number of processing methods based on strain-induced grain refinement have been applied to obtain the nanocrystalline materials, such as ultrasonic nanocrystal surface modification (UNSM) [10, 11], surface

mechanical grinding treatment (SMGT) [12] and surface mechanical attrition treatment (SMAT) [13].

Among these currently available techniques, surface mechanical attrition treatment (SMAT) is a novel method to induce nanocrystalline surface layer and gradient structure in materials [14]. In recent years, SMAT has attracted growing interests and been applied to various materials, such as pure Fe [15], Mg alloy [16] and stainless steels [17]. Wei *et al.* [18] further investigated the combination of SMAT with other surface techniques, such as rolling, which indicated that it would be expected to strengthen material surface, avoid service failure and prolong service time.

In general, the mechanical properties of nanocrystalline materials induced by SMAT can be improved obviously due to the formation of nanocrystalline structure in surface layer, such as hardness and strength [19–21]. However, such nanocrystalline structure in surface layer is usually in the non-equilibrium state and liable to grow during heating. Therefore, it is important to investigate the thermal stability of nanocrystalline structure in the surface layer in order to control the grain-size-dependent properties for engineering applications. Some researchers have focused on the thermal stability of nanocrystalline structure in various alloys or pure metals [22–27], which have a large volume fraction of the grain boundaries. Roland *et al.* [28] reported that the microstructure of 316 stainless steels by SMAT with an average grain size of 20 nm was stable up to 600 °C. Bonetti *et al.* [29] found nanocrystalline microstructure on the surface of iron by SMAT with the grain size of 10 nm was thermally stable up to 297 °C. The latest paper of Liu *et al.* [30] indicated that the nanocrystalline surface layer of pure Al produced by SMAT exhibited high thermal stability, which could be retained up to 275 °C. It is reported that the excellent properties induced by SMAT depend on the thermal stabilities of the nanocrystalline layers [31, 32]. However, there is rare literature investigating the thermal stability of nanostructure in Mg alloy by SMAT. Although the thermal stability of the nanostructure in Mg alloys produced by SPD methods has been studied [33], as a mechanical deformation method, SMAT is quite different from SPD in Mg alloy system because of the bad deformability at ambient temperature [34]. SMAT method is operated at room temperature and extremely high strain rates of 10^4 – 10^5 s^{−1} [35, 36], while SPD methods are always implemented at high temperatures and low strain rates less than 10 s^{−1}. The difference is probably attributed to the result that the nanocrystalline structure of Mg alloys produced by SMAT at room temperature exhibits large potential engineering application as well as its high specific strength and light weight.

In the present work, the gradient nanocrystalline structure was obtained in AZ31 Mg alloy by SMAT. The thermal stability of the nanocrystalline structure of AZ31 Mg alloy

was further investigated using a series of annealing treatment and characterization of microstructure and microhardness.

2 Materials and Methods

The experimental materials were commercial rolled AZ31 Mg alloy sheets with the thickness of 1.7 mm. The sheets were subjected to SMAT at vibrating frequency of 20 kHz using 304 stainless steel balls with diameter of 3 mm at room temperature for 3 min. The detailed procedures of the SMAT process can be found in the previous literature [37, 38]. After SMAT, the specimens were cut with the size of 10 mm × 12 mm from the sheet. The annealing treatment was carried out under the temperature of 150, 200 and 250 °C for different durations of 10 min, 30 min, 1 and 2 h. The cross-sectional microstructures were analyzed by using optical microscope (OM, Olympus BX-51). The Vicker microhardness (HV) was determined by a SHIMADZU HMV-2T with the load of 200 g and the holding time of 15 s.

In order to analyze the phase composition and measure the grain sizes of the top surface layer, X-ray diffraction (XRD) was carried out on a Philips X'pert X-ray diffractometer using CuK_α radiation. The samples were prepared carefully by mechanical polishing to 20 μm depth from the surface. The average grain size of the nanocrystalline within the surface layer was calculated by the analysis of XRD peak according to the Williamson–Hall method [39]:

$$\beta \cos \theta = \frac{K\lambda}{d} + \eta \sin \theta \quad (1)$$

where β is the peak width at half the maximum intensity, θ is the position of peak maximum (the Bragg angle), K is the Scherrer constant (0.9), $\eta = 2\varepsilon$ is the strain and λ is the X-ray wavelength ($\lambda = 0.154056$ nm).

3 Results and Discussion

3.1 Microstructure Evolution with SMAT

Figure 1 shows the XRD patterns of the AZ31 Mg alloy before and after SMAT. It is observed that both kinds of samples consist of a large volume fraction of α -Mg phases and a small amount of Mg₁₇Al₁₂ phases in the surface layers. The strongest peak (002) can also be found in both AZ31 Mg alloy samples before and after SMAT, which indicates the feature of the basal texture due to the rolling process. The insert in Fig. 1 gives the magnification of (002) peak in order to check the detailed information of the surface layer. As compared to the as-received sample, an increase in intensity and peak broadening of (002) peak can be observed in the surface layer of the sample induced by

Fig. 1 XRD patterns of the AZ31 Mg alloy sheet surface layers before and after SMAT. The inset is the Mg (002) peak

SMAT, which could be attributed to the nanocrystalline grains and the lattice strain. According to the Williamson–Hall method with Eq. (1), the grain size on the surface of AZ31 Mg alloy subjected to SMAT for 3 min was measured to be about 41.1 nm.

Figure 2 shows an overview of the cross-sectional microstructure of the AZ31 Mg alloy sheet before and after SMAT. For the as-received sample, a typical deformed microstructure of AZ31 Mg alloy can be found with refined grains around the coarse grains, as shown in

Fig. 2a. Chang *et al.* [40] have introduced that the microstructure of as-rolled AZ31 Mg alloy was not homogeneous and shear banding was evident. The average grain size was measured with the value of approximately 7.6 μm , which is similar to the fine grain size of AZ31 Mg alloy by rolling process reported by Zhang *et al.* [41]. After SMAT, the microstructure of the AZ31 Mg alloy exhibits a gradient structure in the surface layer, as shown in Fig. 2b. Moreover, the surface of the sample exhibits the nanocrystalline structure, while the center still reveals the fine grain inherited from the rolling process. The gradient structure layer (GL) has the thickness of about 160 μm . Similar depth-dependent gradient microstructure induced by SMAT process was also reported by Lu *et al.* [37]. Variations of the grain size and the microhardness of AZ31 Mg alloy sheet by SMAT along depth from the center to the surface of both sides are given in Fig. 3. A gradient structure from the surface with nanoscale to the center with microscale as well as the microhardness gradient was obviously induced by SMAT, which is in good agreement with the variation of the grain size according to the Hall–Petch relationship as reported by previous work [42]. The AZ31 Mg alloy by SMAT has a lower recrystallization temperature than the as-received one. Dynamic recrystallization may occur during the SMAT process, leading to grain refinement and high microhardness in the surface layer.

Fig. 2 Cross-sectional microstructures of the AZ31 magnesium alloy samples: **a** before SMAT, **b** after SMAT

Fig. 3 Variations of the grain size and the microhardness along depth from the center of AZ31 Mg alloy after SMAT

3.2 Thermal Stability

3.2.1 Microstructure

The cross-sectional microstructures of AZ31 Mg alloy by SMAT under heat treatment are given in Fig. 4. It can be found that the NL is quite heat sensitive. After annealing for 10 min at the temperature of 150 °C, the thickness of the deformed layer decreased to the value of about 100 μm. However, as the annealing temperature increased to 200 °C with duration of 10 min, the deformed NL on the topmost surface almost disappeared and even completely disappeared after the temperature increased to 250 °C. After annealing for above 30 min, the NL had a thickness about 20 μm with the annealing temperature of only 150 °C. But when the annealing temperature was above 150 °C, the refined grain within the surface layer grew rapidly to micrometer scale from the nanometer scale. It could be attributed to the recrystallization and the growth of nanocrystalline grains. It reveals that the NL has a certain degree of thermal stability below 150 °C. As shown in Fig. 4, it can be clearly seen that with the increase in annealing time, the surface grains gradually grow up in the form of the recrystallization process of the NL. The growth rate of the recrystallized grain is slow under the temperature 150 °C for 10 min. The increase in the annealing temperature and time leads to high fraction of the recrystallized grains. Yu *et al.* [43] also presented similar discontinuous change in Al produced by equal channel angular extrusion (ECAE) in the range of 275–300 °C. The more detailed variation of the thickness of gradient layers with the annealing time at different temperatures is illustrated in Fig. 5.

3.2.2 Microhardness

The microhardness of AZ31 Mg alloy induced by SMAT after annealing treatment is shown in Fig. 6. As shown in

Fig. 6a, the microhardness gradually decreases from the topmost surface to the center. The microhardness of the surface layer is about 109 HV, which is 39 HV higher than that of the matrix. After annealing for 10 min at 150 °C, the microhardness of the surface layer sharply decreases down to 87 HV, while that in the matrix has little change with an average value of 70 HV. When the annealing time exceeds 10 min, the microhardness of the surface layer rapidly falls to the similar value of about 70 HV with the matrix. As described in Fig. 6b and c, under the condition of annealing temperature of 200 and 250 °C, the microhardness of the surface layer decreases shortly to an average value of 60 HV, which is even lower than that of the as-rolled AZ31 Mg alloy [41]. The microhardness gradient almost disappeared when the annealing duration is above 30 min at the temperature of 150 °C. Additionally, it soon disappeared when the annealing temperature exceeds 150 °C. The microhardness gradient was found to be in common with that of NL's thickness resulting from nanocrystalline in the surface layer as analyzed in section of 3.2.1 above.

3.2.3 Grain Size

Figure 7 gives variations of the grain size with different annealing temperatures and durations in the topmost surface of the SMAT samples. As shown in Fig. 7, the grain size of AZ31 alloy increases gradually with the extension of the annealing time. The original average grain size of the nanocrystalline layer before annealing has the value of about 41.1 nm. With the annealing time of 120 min, the grain sizes grew into 3.2 μm, 6.2 μm and 8.8 μm under the annealing temperature of 150, 200 and 250 °C, respectively. Both the annealing temperature and the duration play significantly role in the grain growth rate. The microstructure could be stable within 10 min under 150 °C. When the annealing temperatures increased to 200 and 250 °C, the grain grew very fast within 10 min. When the annealing duration exceeds 10 min, the grain growth becomes slow and hardly changes. Similar phenomenon in grain growth of pure Al in the range of 155–355 °C was reported by Liu *et al.* [30].

3.2.4 Grain Growth Kinetics

In order to investigate the thermal stability of nanostructured material, the kinetics of nanocrystalline grain growth of AZ31 Mg alloy was analyzed. The activation energy for the grain growth can be calculated by Arrhenius Eq. (2) [44]:

$$k = k_0 \exp\left(\frac{-E_g}{RT}\right) \quad (2)$$

Fig. 4 Comparison of the surface layer microstructure of AZ31 alloys under heat treatment: **a** 150 °C/10 min, **b** 150 °C/30 min, **c** 200 °C/10 min, **d** 200 °C/30 min; **e** 250 °C/10 min, **f** 250 °C/30 min

where k_0 is a rate constant which is independent of the absolute temperature of annealing T . E_g is the activation energy for the grain growth, and R is the molar gas constant. In order to find the grain growth constant k at each annealing temperature, the grain growth exponent n was obtained by using a nonlinear general curve fitting the isothermal growth data mathematically from Eq. (3) [45–47]:

$$D^n - D_0^n = kt \quad (3)$$

where D is the grain size at different time t , D_0 is the initial grain size and k is the rate constant which can be related to Eq. (2). According to the results of the microhardness and the grain size, the kinetics of the grain growth was analyzed by the isothermal growth data. The values of the grain growth exponent n were

Fig. 5 Thickness of the gradient layers with the increase in annealing time at different temperatures

derived by using Eq. (2), and their average value is 2. The grain growth exponent n was fit from the fact that the previous studies demonstrated that the value of

n ranges from 2 to 8 for different magnesium alloys [45, 48, 49].

As shown in Fig. 8, the activation energy E_g of the nanocrystalline AZ31 Mg alloy induced by SMAT has a value of 92.8 kJ/mol, which is similar to the value 92 kJ/mol of activation energies for the grain boundary diffusion in as-cast pure Mg [45, 48] and much smaller than the activation energy for self-diffusion of magnesium, which is about 135 kJ/mol [48, 50]. The value is close to the value of grain boundary than that of self-diffusion, indicating that the grain growth mechanism mainly relies on grain boundary diffusion. It is generally accepted that the nanocrystalline material has a large fraction of the grain boundaries (GBs) [51, 52]. As compared to as-cast pure Mg, a majority of GBs in nanocrystalline materials leads to grain boundary migration. It has been reported that SMAT can induce an extremely high free energy state and the dislocation density in the near-surface region of the material [28, 53]. Grain boundary migration is atom diffusion in essence. Moreover, during the processing of SMAT, the interdiffusion of elements could occur between the surface of the samples and small steel balls due to the high strain rate in the chamber.

Fig. 6 Variations of the microhardness along depth from the center of AZ31 Mg alloy by SMAT under annealing at different temperatures: **a** 150 °C, **b** 200 °C, **c** 250 °C

Fig. 7 Variations of the grain size with annealing temperature and time in the top treated surface of AZ31 Mg alloy sheet samples induced by SMAT

Fig. 8 Plot of $\ln k$ (rate constant) versus $1000/T$ of nanocrystalline AZ31 induced by SMAT

Wen *et al.* [54] found that some impurities are introduced into the nanocrystalline surface layer on 2024 Al alloy, which was attributed to the element transfer of the steel balls during the SMAT process. Owing to the impurities, the effect of solute or impurities drag is considered to be a predominant factor influencing the grain boundary migration. In this case, interdiffusion in SMAT process is expected to play a certain role in grain growth of the nanocrystalline material. These results indicate a thermal stability of AZ31 Mg alloy.

4 Conclusions

The AZ31 Mg alloy with gradient structure is produced by SMAT process, in which the variation of microhardness along the cross section shows a “U” type characteristics

and the variation of the grain size from the surface to the center type shows an inverted “U” type. The average grain size at the topmost surface of the alloy after SMAT has the value of about 41.1 nm. The grain refinement by SMAT can result in much higher microhardness on the surface than that of as-received one. The nanocrystalline layer induced by SMAT has a low grain growth rate under annealing temperature of 150 °C. The activation energy was calculated to be 92.8 kJ/mol by analyzing the grain growth kinetics of AZ31 Mg alloy with SMAT. Such nanocrystalline surface layer of AZ31 Mg alloy fabricated by SMAT exhibits a high thermal stability retained up to 150 °C.

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