Evolution of Microstructure, Texture, and Mechanical Properties of As-Extruded ND/ZK60 Composite during Hot Compression Deformation

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Received: 1 August 2020; Accepted: 2 September 2020; Published: 4 September 2020

Abstract: The effects of temperature, strain rate, and strain on the microstructure, texture, and mechanical properties of as-extruded nanodiamond reinforced ZK60 composite during hot compression was systematically studied. The results revealed that the precipitating MgZn2 and the nanodiamond (ND) particles distributed in the grain interiors hindered the motion of dislocations. The ND particles act as nucleation points and promote the dynamic recrystallization (DRX) of the composites during the hot compression deformation, the flow stress of ND/ZK60 increases with strain rate increases and temperature decreases. {1012} extension twins are nucleated and grown in the coarse grains as the compressive strain increasing. Meanwhile, the fine grains of DRX generate and present first an increasing then a decreasing trend. The result of Schmid factor and kernel average misorientation indicates that high-density dislocation caused by dislocation climbing and cross slip aggregated in composites with increasing strain. Therefore, the work hardening trend of the composite is strengthened.

Keywords: nanodiamond; hot compression deformation; dislocation; work hardening

1. Introduction

Magnesium alloys, which have high specific strength and stiffness, excellent damping property, and good thermal conductivity, are the lightest weight commercially available structural materials [1–4]. Therefore, Mg alloys have been widely used in automobiles, electronics, aerospace, and biomedicine. However, their poor high-temperature strength and corrosion resistance hamper the widespread applications of conventional Mg-based alloys. The addition of nano-particles reinforcement into the matrix is found to be an effective way to improve the overall performance of magnesium alloys, thereby forming the composite microstructures.

Mg matrix composites reinforced with nanoparticle reinforcements, such as MgO, TiB2, TiC, SiC, and Y2O3 nanoparticles, have been extensively studied [5–8]. The ND particles are a promising reinforcement for metal matrix composites due to their high hardness, high thermal conductivity, and high performance compressive strength. There is remarkable reinforced effect on Mg alloy because the movement of dislocations is hindered during plastic deformation. However, the plasticity of the Mg matrix composites was significantly reduced. The added reinforcements restricted the dislocation slip and grain boundaries migration, and the propagation and generation of micro-cracks that primarily occur at the interfaces of particle and matrix. Moreover, stress concentration easily occurs near the reinforcements, resulting in the poor plasticity of Mg composites at room temperature. Li et al. [9]
prepared the ND-reinforced ZK60 matrix composites by the powder metallurgy method, the results indicated that ND could efficiently improve the mechanical properties of ZK60 matrix. There are sparse reports on the hot compression properties of nanodiamond-based Mg matrix composites. In recent years, the hot compression deformation behaviors of Mg alloy have been widely studied [10–12]. Lv et al. [10] found that incomplete dynamic recrystallization appears in cast Mg-2.0% Zn-5.8% Y-0.3% Zr during the hot compression deformation. Zheng et al. [11] investigated that the deformation microstructure evolution and deformation mechanism of high-pressure solidified Mg–Zn–Y–Zr alloy at 250 and 300 °C with a strain rate of 0.001 s$^{-1}$. The deformation constitutive equation of Mg–Gd–Nd–Zr was established, and the hot deformation activation energy of 292.679 kJ/mol is determined by Chen et al. [11]. Zheng et al. [2] studied the effects of nano β-TCP particles on the deformation behavior and texture evolution of Mg–Zn–Zr alloys during hot compression and obtained the optimal hot processing conditions using constitutive equation calculations. Wang et al. [13] reported that as-cast TC4p/AZ91 composites exhibited better ductility than that of SiCp/AZ91 composites. Some non-basal slip planes begin to be activated and the number of slip systems increases, due to critical resolved shear stress (CRSS) significantly decreased at elevated temperature, thus the plasticity and deformation performance are improved [14]. Furthermore, the various slip systems activation strongly contributes to the overall growth of the dynamic recrystallization (DRX) grains, which remarkably refine the grains and improve the strength and plasticity of the materials [14–16]. The hot compression deformation behavior of Mg alloys mainly depends on the deformation temperature and strain rate. Therefore, it is of great importance to study the plastic deformation behavior and processing parameters of Mg composites at elevated temperature. To the best of our knowledge, most works of Mg composites concentrated on the effect of inhomogeneous distribution of nano-reinforcements on the mechanical behavior of Mg matrix composites, the reports on effect of deformation temperature and strain rate on the hot compressive deformation behavior of ND-reinforced Mg matrix composites are almost non-existent.

In this study, ND/ZK60 composites were prepared by using powder metallurgy, and the hot deformation behavior of the composite was obtained using Gleebel-3500 instrument (Dynamic Systems Inc., Austin, TX, USA). The effects of deformation temperature and strain rate on the microstructure and mechanical properties of the composite were studied. The clarification of the role of the temperature and strain rate in deformation behaviors of the composite is significant for understanding the mechanical properties origins of the ND/ZK60 composites materials and designing novel functional composites materials for future industrial applications.

2. Materials and Methods

The chemical composition of ZK60 powder (Sheng Tong Metal Powder Co., Ltd., Tangshan, China) with an average diameter of 55 µm in diameter is shown in Table 1. The ND powders were purchased from XFNANO Materials Tech Co., Ltd. (Nanjing, China) with the diameter of 5-10 nm. The 0.05 wt.% ND/ZK60 composite was fabricated using powder metallurgy (PM). First, the ND particle was dispersed in an alcoholic solution for 20 min by using ultrasound disperser, the ZK60 powder was slowly added into to alcohol solution with mechanical stirring for 40 min. The composite powders were dried in an oven at room temperature for 12 h, and which were milled using a DYXQM-12L planetary ball mill (Tianchuang Powder Technology Co., Ltd., Changsha, China) in the Ar atmosphere for 200 min to fabricate the milled uniformly composite powders. Note that the ball-to-powder weight ratio was 20:1, and the milling speed was 400 rpm. Subsequently, the milled composite powders were sintered with vacuum sintering furnace in the Ar atmosphere, and the sintering temperature and pressure is 600 °C and 10 MPa, respectively. Finally, the sintered billet was extruded using a four-column hydraulic press to gain a rod samples with 10 x 35 mm at 350 °C, and the extrusion ratio and extrusion speed is 20:1 and 0.1 mm/s, respectively.
Table 1. The chemical composition of ZK60 powder.

|          | Zn (wt.%) | Zr (wt.%) | Mg (wt.%) |
|----------|-----------|-----------|-----------|
| ZK60 particle | 5.2033   | 0.2–0.3   | Balance   |

The hot compression specimens were cylinders with diameter of 8 mm and length of 12 mm. The hot compression test was performed on a Gleeble-3500 thermo-mechanical simulator with deformation temperature from 150 °C to 350 °C and strain rate ranges from 0.001 s⁻¹ to 1 s⁻¹. A typical flow stress curve at temperature of 200 °C and strain rate of 0.001 s⁻¹ were obtained, which was loaded up to a certain percentage of strain (10, 20, 30, 40) and removed the load after reaching the desired strain. The tensile and compressive properties of samples were tested by an Instron-5982 testing system (Instron, Norwood, MA, USA), along the extrusion direction (ED) at room temperature. Three tests were conducted for each sample. The microstructure of ND/ZK60 composites and hot compression specimens were observed and analyzed by using the optical microscopy (OM), scanning electron microscopy (SEM, ZEISS-6035, Carl Zeiss Microscopy Ltd., Jena, Germany) equipped with energy dispersive X-ray (EDX, Oxford Instruments, Oxford, UK) and transmission electron microscopy (TEM, JEM-2010 HR, JEOL Ltd., Tokyo, Japan). The specimens were polished and etched with picric-acetic acid. X-ray diffraction (XRD, BRUKER XD8AD VANCE-A25, Bruker, Karlsruhe, Germany) was used to analyze the macro-texture. The evolution of deformation texture was characterized by electron backscatter diffraction (EBSD), with the corresponding data analyzed by the Oxford HKL channel 5 software (Oxford Instruments, Oxford, UK).

3. Results

3.1. Microstructural Observation of the ND/ZK60 Composite

Figure 1a shows the microstructures of as-sintered 0.05 wt.% ND/ZK60 composite. Most ZK60 particles retain the original spherical shape in the samples, and their size is about 55 µm. The particles are not entirely welded in the sintering because of the MgO on the surface of the ZK60 particles hindered their combination. The average grain size of composites is ~15 µm. Compared with the as-sintered composite, the grains of as-extruded composite are fine, as shown in Figure 1b, the average grain size is ~5 µm. The spherical particles of ZK60 become thin strips during the hot extrusion process, some small white particles distribute at the particle boundaries, grain boundaries and grain interiors. White particles agglomerated at particle boundaries, but uniformly distributed in the grain interiors and at the grain boundaries, as shown in the magnified image Figure 1c. The EDS spectrum acquired from points d, e, and f are shown in Figure 1c–f, respectively. The EDS spectrum of point e indicates that the white particles in grain interiors and at grain boundaries are MgZn₂ phases, which can hinder the motion of dislocations and the growth of grains, thereby improved the mechanical properties of the composites. A small amount of O element was detected in white particles at particle boundaries from Figure 1f, which indicates the existence of MgO at the particle boundaries in the as-extruded composites.

Figure 2 shows the distribution of ND particles and the interface bonding status in the as-extruded 0.05 wt.% ND/ZK60 composite. According to our previous work [16], Figure 2a shows that ND particles uniformly distribute in grain interiors, however, a small amount of ND particles appear and agglomerate at the grain boundaries. Figure 2b shows a typical high-resolution image of the interface bonding between the ND reinforcement particle and the ZK60 matrix. There is only mechanical embedment or physical bonding without interface reaction taking place between Mg and ND.
3.2. The Flow Stress and Microstructure of Composites at Different Deformation Temperature and Strain Rate

The effect of strain rate and temperature on mechanical properties of ND/ZK60 composites were analyzed by Gleeble-3500 thermo-mechanical simulator. The true strain–stress curves of 0.05 wt.% ND/ZK60 composite compressed are depicted in Figure 3. Temperature has a significant effect on the mechanical properties of composites during hot deformation. When the strain rate is 0.001 s\(^{-1}\), the flow
stress remarkably decreases as with elevating temperature. The CRSS required to activate the basal slip and the non-basal slip decreases and the DRX occurs earlier when the temperature is increased, which leads to lower work hardening, thus, the flow stress is reduced [17–20]. A typical flow stress curve of ND/ZK60 was obtained at 200 °C. It was found that the flow stress of ND/ZK60 increases to a peak in the initial stage of deformation and then decreases to a steady state with increasing of strain, which indicates that the DRX occurred during plastic process [17]. The flow stress of ND/ZK60 slight increases or remains in a balance state when the temperature is over 300 °C, which indicates that work hardening balances with work softening at high temperatures. When the temperature is constant, the flow stress of ND/ZK60 increases obviously with strain rate increases. The flow stress of ND/ZK60 decreases slightly with increasing of strain at the strain rate of 0.001 s⁻¹ and 0.01 s⁻¹, which means the work softening is more obvious than work hardening at lower strain rates. However, the flow stress increases with increasing strain during the whole deformation process at the strain rate of 1 s⁻¹, which indicates that the deformation process is dominated by the work hardening at the high strain rate. Therefore, the flow stress of ND/ZK60 increases with strain rate increases and temperature decreases.

![Figure 3](image_url)

**Figure 3.** The true stress–strain curves of the 0.05 wt.% ND/ZK60 composite deformed at different strain rates and deformation temperatures. (a) The true stress–strain curves of composite deformed under different temperatures at strain rate of 0.001 s⁻¹; (b) The true stress–strain curves of composite deformed under different strain rate at 200 °C.

Generally, ND particles and precipitates can hinder dislocation motion during hot deformation, thus, a large number of dislocations generate and accumulate around ND particles, precipitates, and grain boundaries in composites, which lead to obvious work hardening effect of the materials. The dislocations near the grain boundaries, ND particles, and precipitates have a sufficient time to release at a low strain rate, and the dynamic recrystallization phenomenon has sufficient time to occur, which results in the work softening of composites. While the composite does not have sufficient time to undergo dynamic recrystallization at a high strain rate. The dislocations generated near the grain boundaries, ND particles, and precipitates do not have sufficient time to release, which eventually contributes to the work hardening of composites. The work hardening and work softening are two primary competing mechanisms in hot deformation [18,19].

Figure 4 demonstrates microstructures of 0.05 wt.% ND/ZK60 composite compressed at different deformation temperatures and strain rates. The fine recrystallized grains appear and uniformly distribute in the composites at 200 °C. The grains of composite become coarsely when the temperature is over 300 °C. It is found that the ND particles act as nucleation points and promote the DRX of the composite during the high-temperature deformation process. When the temperature is below 250 °C, the DRX could develop sufficiently with the lower strain rate, which may also lead to the decrease of flow stress. There are fewer and fewer fine recrystallized grains observed in the composites with the
strain rate gradually decreasing from 1 s\(^{-1}\) to 0.001 s\(^{-1}\), and the grains grows into coarse equiaxed grains at the strain rate of 0.001 s\(^{-1}\).

![Figure 4](image)

**Figure 4.** Optical microscope images of 0.05 wt.% ND/ZK60 composite compressed at different deformation temperatures and strain rates.

3.3. Flow Stress Behaviour with Different Strain at the Temperature of 200 °C and a Strain Rate of 1 s\(^{-1}\)

It is worth mentioning that a typical flow stress curve is obtained at temperature of 200 °C and strain rate of 0.001 s\(^{-1}\), as show in Figure 3a. The stress attains a peak value and then decreases at a steady state slowly. However, the stress rises until the compressive process is suspended at temperature of 200 °C and strain rate of 1 s\(^{-1}\), as shown in Figure 3b. The curve is similar to what Lu [21] mentioned for the Mg–Ga–Y alloys, the stresses instantly increased to peak stress, and then fractured at a strain of about 0.3. Therefore, the true stress–strain curves of 0.05 wt.% ND/ZK60 composite with different strains at the deformation temperature of 200 °C and strain rate of 1 s\(^{-1}\) were obtained and shown in Figure 5.

![Figure 5](image)

**Figure 5.** True stress–strain curves of the as-extruded 0.05 wt.% ND/ZK60 composite with different strain at the deformation temperature of 200 °C and a strain rate of 1 s\(^{-1}\).
3.4. Microstructure and Texture with Different Strain at the Temperature of 200 °C and a Strain Rate of 1 s⁻¹

The evolutions of macro-textures (XRD) of the as-extruded 0.05 wt.% ND/ZK60 composite with different strains at the temperature of 200 °C and strain rate of 1 s⁻¹ were obtained. According to the pole figure in Figure 6a, as-extruded composite had a typical (0001) basal fiber texture with a texture intensity of 4.7. The c-axes of most grains were perpendicular to the ED. The pole figure of the composite with 10% strain is shown in Figure 6b, the texture intensity was weakened compared with the as-extruded composite. Grains of composite begin to rotate, the amount of the c-axis of grains parallel to the ED were increased, and the maximum texture intensity was accordingly weakened. When the engineering strain further increases to 20%, a part of (0001) pole figures were distributed along the ED (Figure 6c). It was found that many DRXed grains appeared and no twins existed in the composite. However, the texture intensity was enhanced compared to that of the engineering strain of 10%. The reason was attributed to the fact that the c-axis of most grains rotate being perpendicular to the ED from being parallel to the ED under the strain of 20%. Although the texture component with the strain of 30% is the same as that of strain of 20%, while the texture intensity was weakened slightly. According to Figure 6d, a part of pole density points of (0001) pole figure were distributed along the ED, and another part pole density points of (1010) pole figure became cluttered compared with that of the strain of 20%, thereby the texture intensity was weakened. When the engineering strain continued to increase to 40%, the volume fraction of DRXed grains decreased, which indicated that DRXed grains merged with each other because of the expansion of grain boundaries at higher strain [22]. Meanwhile, the pole density points of the c-axis of grains perpendicular to the ED completely disappeared, which means the c-axis of grains turned to the direction paralleled to the ED, thereby the intensity of texture enhanced to 6.09 (shown in Figure 6e).

![Figure 6](image-url)  
**Figure 6.** Macro-textures of as-extruded 0.05 wt.% ND/ZK60 composite under different strains at the deformation temperature of 200 °C and strain rate of 1 s⁻¹: (a) 0, (b) 10%, (c) 20%, (d) 30%, and (e) 40%.

Figure 7 is EBSD maps and misorientation distribution maps of the 0.05 wt.% ND/ZK60 composites with different strains at the temperature of 200 °C and strain rate of 1 s⁻¹. Narrow grains and a lot of equiaxed grains were found in the initial extruded composite from Figure 7a, twins did not generate during extrusion. Additionally, the proportion of high angle grain boundaries (HAGBs) is extremely lower than that of low angle grain boundaries (LAGBs), as shown in Figure 7b. [1012] extension twins appear in the composite materials when a specific pressure applied along the ED of the composites at high temperatures [23,24]. [1012] extension twins are nucleated and grown in the coarse grains as the compressive strain increases. Meanwhile, the fine grains of DRX generate and present first an increasing then a decreasing trend during the hot compressive deformation, as shown in Figure 7c–i.
The number fraction of DRXed grains in initial state (0.38) is much less than DRXed grains in deformation state, for example, the number fraction of DRXed grains is 9.56 with the strain of 20%. Robson et al. [25] studied particles in matrix effect on recrystallization, which found that DRXed grains
prior precipitates occur along the grain boundaries because of the strain induced boundary migration (SIBM). ND particles and precipitates can hinder the dislocation motion during hot deformation, which makes dislocations can pile up quickly. However, the work softening such as DRX was restricted in high strain rate. In addition, the accumulated dislocations were difficult to expand in time, which led to the work hardening rate higher than work softening rate. Kernel average misorientation (KAM) plays an important role in studying the deformation mechanism, it can determine the degree of local misorientation induced by dislocation slip during hot compression process [26]. Figure 8 displays the KAM maps and local misorientation angle distribution maps of as-extruded 0.05 wt.% ND/ZK60 composite with different strain at the temperature of 200 °C and strain rate of 1 s⁻¹. The average KAM value (0.425) is lowest because of the accumulated dislocations which are mainly annihilated by DRX during hot extrusion (in Figure 8a,b). From Figure 8c–h, the green color gradually deepened with the increasing of engineering strain, which indicating that more and more dislocations were accumulated. The average KAM value significantly increased and reached a peak (1.080) at the engineering strain of 30%, then, it decreased to 0.953 at the engineering strain of 40%. It is worth noticing that many fine grains generated by DRX distribute around coarse grains as the engineering strains are 20% and 30%. Simultaneously, the local misorientations are highly concentrated near the grain boundaries at the early stage of deformation. However, most local misorientations appear inside coarse grain with the increasing of engineering strain. The dislocation density reached to saturation in the fine grains and new dislocations generated in coarse grain, which promotes work hardening and results in a higher strength.

![Figure 8. Kernel average misorientation maps and local misorientation angle distribution maps of as-extruded 0.05 wt.% ND/ZK60 composite with varied strains at the deformation temperature of 200 °C and the strain rate of 1 s⁻¹: (a,b) 0, (c,d) 10%, (e,f) 20%, (g,h) 30%, and (i,j) 40%, (k) line chart of average KAM value.](image)

The Schmid factor (SF) is an essential factor in the characteristic of microstructure and mechanical properties during hot deformation [27]. The characteristic of twin variant and slip systems depend on the SF of different slip systems in deformation process [28]. The slip systems and twin variants with the highest SF value have a higher possibility to activate [29]. Figure 9 shows the SF distribution maps of the 0.05 wt.% ND/ZK60 composite compressed under different strains at the deformation temperature of 200 °C and strain rate of 1 s⁻¹. The average SF of basal <a> and pyramidal <c +
a> slips (0.35) are higher than that of prismatic <a> slip (0.21) at initial stage. The average SF of pyramidal <c + a> slip slightly increased (0.38) when the engineering strain of composites reached up to 40%. However, the average SF of basal <a> slip gradually decreased to 0.24, the average SF of prismatic <a> slip significantly increased to 0.40 at the engineering strain of 40%. The average SF of prismatic slip was the highest and the basal slip’s was the lowest after hot compression deformation, which means the slip mode trended from the basal slip to the prismatic and pyramidal slips during the whole hot compression deformation. It is beneficial to form high-density dislocation accumulation caused by dislocation climbing and cross slip. Therefore, the work hardening trend of the composite is strengthened.

Figure 9. Schmid factor (SF) distribution maps of as-extruded 0.05 wt.% ND/ZK60 composite under varied strains at the deformation temperature of 200 °C and strain rate of 1 s⁻¹: (a) 0, (b) 10%, (c) 20%, (d) 30%, and (e) 40%, (f) the average Schmid factor with different engineering strain.

In order to further investigate the deformation mechanism, the microstructure of the as-extruded 0.05 wt.% ND/ZK60 composite under the engineering strains of 0, 10%, 30%, and 40% was analyzed by TEM. Figure 10 revealed that dislocations were generated near the grain boundary. Moreover, a bit of stress concentration appeared after hot extrusion, indicating that the composite exerted an annular compressive stress perpendicular to the basal plane during hot extrusion, as shown in Figure 10a. Thus, the movement of dislocations were hindered by grain boundary, resulting in accumulation of dislocations and formation of stress concentration zone. When the engineering strain reached to 10%, more dislocations were accelerated and formed a wall (marked in red dotted frame). The stress concentration can increase the stress to CRSS for the activation of non-basal slip. Meanwhile, the DRX occurs and exhibits obvious strain rate sensitivity at 200 °C [30], which means the DRX process was impeded at the strain rate of 1 s⁻¹. Hence, more and more dislocations entangled which cannot be eliminated easily with increasing strain, as shown in Figure 10c,d, which result in the contribution of work hardening, and further enhancing the strength of the composite.
In this study, ND/ZK60 composite were prepared by powder metallurgy and hot extrusion processes. The effects of deformation temperature, strain rate, as well as strain on the microstructure, texture evolution and mechanical properties of the as-extruded ND/ZK60 composite during high-temperature compression were examined, and the following conclusions were obtained:

1. The precipitated MgZn$_2$ phase and the ND particles hindered the motion of dislocations, thereby improving the mechanical properties of the composite, and the MgO particles on the surface of the ZK60 particles impeded the welding between ZK60 particles.

2. The flow stress of composites increases gradually with the decreasing temperature and increasing strain rate during hot compression. When the deformation temperature and strain rate are 200 °C and 1 s$^{-1}$, many fine DRXed grains generated and surrounded coarse grains with the increasing of engineering strain, which promotes work hardening, resulting in higher strength.

3. The slip mode trended from the basal slip to the prismatic and pyramidal slips during whole hot compression deformation at 200 °C and 1 s$^{-1}$, which means the crystallographic orientation of initial grains transformed, resulting in the elevation of texture intensity.

Author Contributions: Conceptualization, J.W.; methodology, S.L.; investigation, H.M.; writing—original draft preparation, S.L.; writing—review and editing, H.M. and J.W. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by of Qing Hai Provincial Natural Science Foundation (Grant No. 2018-ZJ-949Q).

Conflicts of Interest: The authors declare no conflict of interest.

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