Article

Masking Effect of LPSO Structure Phase on Wear Transition in $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ Alloy

Fujun Tao ¹, Hongfei Duan ², Lijun Zhao ¹ and Jian An ^{1,*}

¹ School of Materials Science and Engineering, Jilin University, Changchun 130025, China; taofj19@mails.jlu.edu.cn (F.T.); lijunzhao@jlu.edu.cn (L.Z.)

² Shanxi Academy of Science and Technology Co., Ltd., Jinneng Holding Group Co., Ltd., Taiyuan 037000, China; duanhf_2005@163.com

* Correspondence: anjian@jlu.edu.cn; Tel.: +86-431-58095874; Fax: +86-431-85095876

Abstract: Room- and elevated-temperature wear tests were conducted using a pin-on-disk testing machine to study wear behavior of $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy and role of long-period-stacking-ordered (LPSO) structure phase in mild–severe wear transition (SWT). Variation of wear rate exhibited a three-stage characteristic with load at various test temperatures, i.e., a gradual increasing stage, a slightly higher plateau stage, and a rapid rising stage. The wear mechanisms in the three stages were identified using scanning electron microscope (SEM), from which the first stage was confirmed as mild wear, and the other two stages were verified as severe wear. The interdendritic LPSO structure phase was elongated into strips along the sliding direction with Mg matrix deformation in the subsurface, plate-like LPSO structure phase precipitated at elevated temperatures of 150 and 200 °C. The fiber enhancement effect and precipitation effect of LPSO structure phase resulted in a little difference in wear rate between the first and second stages, i.e., a masking effect on SWT. Microstructure and microhardness were examined in the subsurfaces, from which the mechanism for SWT was confirmed to be dynamic recrystallization (DRX) softening. There is an apparently linear correlation between the critical load for SWT and test temperature, indicating that SWT is governed by a common critical DRX temperature.

Keywords: $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy; long-period stacking ordered phase; wear mechanism; wear transition; dynamic recrystallization

Citation: Tao, F.; Duan, H.; Zhao, L.; An, J. Masking Effect of LPSO Structure Phase on Wear Transition in $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ Alloy. *Metals* **2021**, *11*, 1857. <https://doi.org/10.3390/met11111857>

Academic Editor:
Menachem Bamberger

Received: 18 October 2021
Accepted: 17 November 2021
Published: 18 November 2021

Publisher's Note: MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.

Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

1. Introduction

$\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy is a special type of rare-earth (RE) magnesium alloy not only because it exhibits excellent strength and high thermal stability, but also because it contains an extraordinary long-period-stacking-ordered (LPSO) structure phase. The LPSO phases in Mg–Y–Zn alloys have the same (0001) basal plane as Mg, and their stacking periodicity is lengthened 18- or 14-fold, respectively. These excellent properties are believed to have a close relation to LPSO structure phase. LPSO phase in $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy usually has a long-period 18R modulated structure, and is often referred to as X-Mg₁₂ZnY phase [1]. A type of rapidly solidified powder metallurgy (RS/PM) $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy was fabricated by Kawamura et al. [2] in 2011, possessing super-high yield strength above 600 MPa. Since then, a lot of effectors have been devoted to investigations on the effects of LPSO structure phases on physical, chemical, and mechanical properties of Mg–Y–Zn alloy. Mine et al. [3] reported high thermal stability of LPSO phase up to 500 °C in an extruded $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy, the LPSO phase kept its original morphology at 500 °C during an annealing treatment. The strengthening mechanisms of LPSO phase in $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy are found to include such aspects: refinement of Mg matrix grains, prevention of the growth of {10–12} deformation twin, precipitation hardening and short-fiber reinforcement [4,5]. The most attractive characteristic of LPSO structure phase is the excellent deformation ability. As we know, conventional intermetallic compound phases in magnesium alloys have almost no ability

to deform with metallic matrices. However, LPSO structure phase can deform via the modes such as kink deformation and basal $\langle a \rangle$ slip [6,7], which gives $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy a good combination of strength and ductility.

Even though conventional Mg-based alloys are not regarded as proper alternatives to Al-based alloys in tribological applications owing to their low strength, indifferent ductility and poor thermal stability [8,9], $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy has the potential to make wear components such as bearing, pistons of engine and clutch in automotive industry and low-mediate-load bearing gear in aerospace industry [10,11]. To achieve this goal, the room- and elevated-temperature wear performance of $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy should be studied, in which the mild–severe wear transition (hereafter abbreviated as SWT) and the role of LPSO structure phase are two main concerns. First, Mg-based alloys usually present two different kinds of wear behavior depending on sliding conditions, namely mild wear and severe wear [8,9,12–15]. Mg-based alloys undergo a low wear loss and slight damage to surface in mild wear, but suffered from a high wear loss and a massive surface damage in severe wear. Therefore, mild wear is widely regarded as an allowable working state, while severe wear is thought as a dangerous working state. Investigation of SWT can help to determine the safe working area for $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy. Secondly, during the room- and elevated-temperature wear testing, a series of complicated surface response to wear will occur including oxidation, deformation, microstructure transformation, and property change, etc. It is also an important aspect to study how LPSO structure phase influences microstructural and mechanical properties of surface material as well as wear performance for evaluating the possibility of engineering applications of $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy.

Therefore, in the present paper, wear tests of $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy were conducted at temperatures of 20–200 °C. At each test temperature, SWT was determined by analysis of wear rate variation with applied load and SEM observation of worn surfaces. The role of LPSO structure phase on wear rate variation was discussed. The linear correlation between SWT and test temperature was interpreted based on microstructure transformation and property change in surface material and friction heating.

2. Experimental Details

A round ingot of $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ (at. %) alloy was prepared by conventional casting technology with protection of Ar gas. The raw materials were pure magnesium, pure zinc, and Mg-20.3 wt.% Y alloy. The casting process was carried out in three steps as follows: the first step was melting in the resistance furnace under protection of Ar gas. When pure Mg was heated to melt at 740 °C in a graphite crucible with 296 mm top diameter, 189 mm bottom diameter, and 356 mm height, pure Zn, and Mg-Y master alloy were added into the melt according to their required amounts. The second step was stirring. Manual stirring was kept for about 3 min using a stainless steel bar of 5 mm diameter to help alloying elements uniformly distribute in the melt. The third step was pouring. The melt was reheated to 760 °C and hold for a period of about 15 min, then was poured into a steel mold. Since the steel mold could accelerate cooling process, it was thus used to avoid grain growth of cast ingots. The steel mold was preheated at 100 °C before pouring. Ingots of 95 mm in diameter and 200 mm in height were yielded.

Phase constituents and microstructure of $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy were analyzed by X-ray diffractometer (XRD, Rigaku Corporation, Tokyo, Japan) using $\text{Cu K}\alpha$ radiation under condition of 40 kV and 30 mA and optical microscope, respectively. Thermal transformations in $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy were examined using a differential thermal analyzer (DTA, PerkinElmer, Waltham, MA, USA). Tensile properties were measured on a material testing system. Hardness was measured on polished surface of $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy using a micro-hardness tester. Both the tensile properties and hardness were obtained from averages of three specimens.

Wear tests were performed at 2.0 m/s with a MG2000 type pin-on-disc machine (Chengxin Test Equipment Manufacturing Company Limited, Zhangjiakou, China). Five test temperatures were used, including 20, 50, 100, 150 and 200 °C. At each temperature,

the first wear test started with 20 N using a pin specimen, after sliding over a distance 377 m, the specimen was removed from wear equipment for measurement of volumetric loss, and then the next ones were carried out with larger load using new pin specimens, usually increasing with a spacing of 20 N or 40 N, until surface melting could be observed by naked eyes. According to the very smooth surface and ripple edge, surface melting can be roughly observed on the worn surface of the pin by naked eyes. In addition, the coefficient is usually very low due to lubricating effect of surface material melting. For accurately measuring the critical load for wear transition, the minimum spacing of 5 N or 10 N was also adopted under certain conditions. Pin specimens, $\varnothing$ 6 mm $\times$ 13 mm, were machined at center part of $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy along the ingot axis to avoid casting defects and microstructure heterogeneity, and their end surfaces were polished to a roughness around 0.5 μm Ra before wear testing. A digital precision micrometer was used to measure the reduction of pin length before and after the wear test. The length reduction of at least three specimens gave the average over the designed sliding distance of 377 m. The average volumetric wear rate W can be calculated using Equation (1).

$$W = \frac{A\Delta L}{S} \quad (1)$$

where A is the end area of pin, ΔL is the length reduction after wear test, S is the sliding distance, i.e., 377 m. The disks, $\varnothing$ 70 mm $\times$ 10 mm, were made of 5 Cr steel. They were hardened to a hardness of 56 HRC by quenching treatment, and their bearing surfaces were ground to a roughness around 0.6 μm Ra. The rotational speed of disk was kept constant at 2.0 m/s during wear testing.

Pin surfaces were examined after wear testing with a scanning electron microscope (SEM, TESCAN, Brno, Czech Republic) equipped with an energy dispersive X-ray spectrometer (EDS, Oxford Instrument Company, Oxford, UK). A few worn specimens were selected to prepare longitude-sections for microstructure observation and hardness measurement. The subsurface microstructure was observed with an optical microscope, and hardness was measured along depth direction on cross section with a microhardness tester under a load of 0.49 N.

3. Results and Discussions

3.1. Phase Constituents, Microstructure, and Tensile Properties of $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ Alloy

The phase constituents in as-cast $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy were identified by XRD analysis. Only two phases were detected in the alloy owing to the detection limit of XRD, i.e., α -Mg solid solution phase and X- Mg_{12}ZnY phase according to JCPDS files (35-0821 (α -Mg) and 36-1273 (Mg_{12}ZnY)), details of which can be found in our previous work [10]. Optical microscopy observation revealed that α -Mg phase took the form of dendrite and X- Mg_{12}ZnY phase was distributed at dendrite boundaries in the shape of intermittent network, as shown in Figure 1.

With the help of differential thermal analysis (DTA), the general developing process of microstructure comprised of α -Mg and X- Mg_{12}ZnY phases can be interpreted. During the continuous heating process in DTA, two endothermic peaks appeared successively, the former had an initial temperature of 533.8 $^{\circ}\text{C}$, while the latter had an initial temperature of 602.8 $^{\circ}\text{C}$ and a final temperature of 662.7 $^{\circ}\text{C}$, details of which can be found in our previous work [10]. Apparently, the former endothermic peak corresponds to the melting of the eutectic (α -Mg + X- Mg_{12}ZnY), while the latter one refers to the melting of primary phase (α -Mg dendrites). During casting process, $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy melt began solidifying into primary α -Mg dendrites at a temperature close to 662.7 $^{\circ}\text{C}$ till a temperature around 602 $^{\circ}\text{C}$, and then the rest of melt transformed into divorced eutectic α -Mg + X- Mg_{12}ZnY till a temperature around 533.8 $^{\circ}\text{C}$. The ultimate strength, yield strength, elongation as well as hardness of $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy are listed in Table 1.

Figure 1. Optical microphotograph of $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy.

Table 1. Mechanical properties of $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy.

Yield Strength (MPa)	Ultimate Strength (MPa)	Elongation (%)	Hardness (HV)
132.5 ± 5.3	165.7 ± 5.9	5.1 ± 1.2	76 ± 2.3

3.2. Wear Rates

Figure 2 shows the wear rates of the alloy at various test temperatures as a function of applied load. It can be seen that the wear rates interweaved together in the range of 20–140 N. For a better demonstration of the difference between them, the variations of wear rates at temperatures of 20–100 °C and 150–200 °C were thus shown in Figure 2a,b, respectively. Apparently, wear rates at various test temperatures totally exhibited an increasing tendency with applied load. In addition, the most prominent influence of applied load on wear rate manifested a three-stage feature. As seen from Figure 2a, for the sliding at 20 °C, wear rate modestly increased with applied load within 20–80 N, then rapidly rose a little but immediately maintained an almost constant value within 100–140 N, finally climbed again, and reached a high-value peak within 160–200 N. The above-mentioned load ranges of 20–80 N, 100–140 N, and 160–200 N corresponded to the first, second, and third stage, respectively. At higher test temperatures such as 150 and 200 °C, wear rate essentially varied in a similar trend with applied load, as shown in Figure 2b. The great decrease of wear rate in the load range of 130–140 N at 150 °C was found to be related to the melting of surface material, which could promote a lubricating effect between pin and disk and consequently reduced the wear rate.

The load ranges corresponding to the three stages of wear rate at various test temperatures are summarized in Table 2. It notes that with temperature rising, the load range corresponding to the first stage decreases within 20–100 °C but increases within 150–200 °C. The wear rate varied within a narrow range in both the first and second stages. The wear rate was at a low level ranging from $5.2 \times 10^{-12} \text{ m}^3\text{m}^{-1}$ to $16.2 \times 10^{-12} \text{ m}^3\text{m}^{-1}$ in the first stage, while it was at a mediate level ranging from $12.1 \times 10^{-12} \text{ m}^3\text{m}^{-1}$ to $23.2 \times 10^{-12} \text{ m}^3\text{m}^{-1}$ in the second stage. Additionally, it is notable that wear rate varies little in the second stage, and its level descends with increasing temperature, as illustrated in Figure 2c. This makes it a little difficult to separate the first stage from second stage at higher temperatures such as 150 °C and 200 °C owing to little difference in wear rate between the two stages. Apparently, according to the low value and gradual increasing characteristic of wear rate, the first stages on the wear rate curves are certainly included in mild wear regime. In addition, clearly, the third stages are easily judged to be included in severe wear regime. However, it is difficult to determine which wear regime the second stages should be included in because two paradoxical phenomena occur in the second

stages. The first one is that after an initial gradual increasing in the first stage, wear rate presents a sudden rising at the beginning of the second stage, which usually corresponds to SWT. If judged from this characteristic, the second stage should be included in severe wear regime. The second is that the feature of a rapid rising but immediately maintaining steadily for wear rate is contrary to a well acknowledged continuous rising trend in severe wear regime. Therefore, the ascription of the second stages should be determined from the viewpoint of surface damage observed by SEM technique, i.e., whether or not severe surface damage was made in the second stage.

Figure 2. Wear rate versus applied load: (a) 20–100 °C, (b) 150 and 200 °C, (c) the second stages.

Table 2. Load ranges corresponding to different stages at different test temperatures.

Test Temperature (°C)	First Stage (N)	Second Stage (N)	Third Stage (N)
20	20–80	100–140	160–200
50	20–70	80–120	140–200
100	20–60	80–100	120–160
150	20–45	50–80	100–140
200	20–35	40–80	100–120

3.3. Wear Mechanisms

Wear mechanisms operating in the above-mentioned three stages were analyzed using SEM. Figure 3 shows the surface morphologies at different loads for sliding at 50 °C. At 20 N, the surface material underwent slight deformation, which resulted in a flat appearance (Figure 3a). Additionally, a few cracks were found to be formed roughly perpendicular to sliding direction, indicating presence of delamination wear mechanism. At 60 N, the deformation extent of surface material became evident as a result of occurrence of a flatter surface, but delamination sign became less significant because of only small amount of delamination scars (Figure 3b). Therefore, wear mechanisms in the first stage were mild plastic deformation and delamination. In the second stage, the most significant morphology feature was the plastic flow, which produced a quite smooth surface without

any signs of cracks or delamination scars. The smooth surfaces at 80 N and 100 N are presented in Figure 3c,d, respectively. Moreover, the surface material was squeezed along sliding direction and formed a protruded edge, as seen from the insert in Figure 3d. Thus, wear mechanisms in the second stage were severe plastic deformation. It is well accepted that severe plastic deformation is a type of severe wear for magnesium alloys since surface material loses its original good ability to resist plastic flow [8–15]. Therefore, according to the wear mechanism operating in this stage, wear enters severe wear regime in the second stage even though the wear rate value does not increase continuously throughout the stage. In the third stage, at 140 N, the surface was still suffered from severe plastic deformation (Figure 3e). With increasing load to 160 N, the surface presented a sign of melting with smooth plane together with a ripple edge (Figure 3f). Such edge morphology was formed as a result of overlying of melt layers driven towards sliding direction by friction force. It also indicates that the frictional heating is large enough to melt the surface material at 160 N. The melting temperature of $Mg_{97}Zn_1Y_2$ alloy was 533.8 °C detected by DTA. Therefore, wear mechanism was severe plastic deformation in the first half of the third stage, and transformed to surface melting in the second half of the third stage.

Figure 3. SEM microphotographs of worn surfaces at 50 °C under different loads: (a) 20 N, (b) 60 N, (c) 80 N, (d) 100 N, (e) 140 N, (f) 160 N.

Morphologies of worn surfaces before and after SWT at 100, 150 and 200 °C were also examined using SEM technique, as illustrated in Figure 4. It is found that in the first stages, the morphological features are mainly delamination scars, cracks and flat surface at 100, 150 and 200 °C, and delamination feature weakens with increasing temperature to 200 °C, but slight plastic deformation feature intensifies, as illustrated in Figure 4a,c,e. Wear mechanisms operating in the first stages were also delamination and mild plastic deformation. Wear in the first stages was thus mild wear. However, in the second stages, those features of mild wear disappeared, but the morphological features are replaced by smooth plane and extruded edge at 100, 150 and 200 °C, as shown in Figure 4b,d,f. The dominant wear mechanism in the second stages was also found to be severe plastic deformation. Wear in the second stages at all test temperatures was therefore found to be severe wear. These observation results identified that although the wear rate in the second stages was not in a mode of continuous rising with increasing load, the wear mechanisms at all test temperatures were severe plastic deformation. Consequently, the wear behavior was actually severe wear. From this we can conclude that the transition between the first and second stage actually is the SWT.

Figure 4. SEM images of worn surfaces at 100 °C (a,b), 150 °C (c,d) and 200 °C (e,f) under different loads: (a) 60 N, (b) 80 N, (c) 20 N, (d) 50 N, (e) 20 N, (f) 40 N.

3.4. Masking Effect of LPSO Structure Phase on SWT

It is interesting to note the phenomenon that wear rate maintains a moderate tendency almost regardless of increasing applied load in the second stages (i.e., the initial stages of severe wear). This phenomenon that happens to nonferrous alloys such as Mg, Al and Ti alloys is usually associated with formation of tribo-layers or mechanically mixed layers (MMLs) on the worn surfaces [16–19]. Formation of continuous and compact tribo-layers or MMLs on the worn surfaces plays a protective role in retarding surface damage and consequently reducing wear rate, because tribo-layers or MMLs can separate metal to metal contact between tribo-pairs, they also have higher hardness than the metallic substrates. However, in the present work, the possibility related to tribo-layers or MMLs can be eliminated as the contents of oxygen element on the worn surfaces at 50 °C were not higher in the second stage than in the first stage, and even reduced to low level of 1.79–3.64%. Table 3 lists EDS analysis results of the worn surfaces in the first and second stages. EDS technique is only a semi-quantitative method to measure contents of elements on the surfaces, and it cannot ensure an enough accuracy, but the measured values are still able to represent the varying trend of composition in the wear process.

Table 3. Contents of major elements on the worn surfaces.

Temperature (°C)	Stage	Load (N)	O	Y	Zn	Fe
50	First	60	6.22	6.44	2.55	0.13
		80	4.00	6.46	2.56	0.09
		100	5.17	6.68	2.59	0.15
100	First	60	3.28	6.45	2.66	0.10
		80	2.42	6.86	2.77	0.18
		100	3.64	6.97	2.48	0.04
150	First	40	3.12	6.32	2.58	0.11
		50	2.48	7.18	2.74	0.12
		80	3.52	8.38	2.52	0.05
200	First	20	3.72	6.33	2.23	0.06
		30	2.89	6.38	2.47	0.08
	Second	40	1.79	7.26	2.71	0.11
		60	3.81	6.78	2.66	0.17

A distinguished feature of chemical composition was found by comparing the worn surfaces in the first and second stages, i.e., the content of Y element is higher in the second stage. In the first stage, the content of Y element ranged from 6.33% to 6.45%, whereas in the second stage, it ranged from 6.46% to 8.38%. At higher temperatures of 150 and 200 °C in the second stage, the contents of Y element reach as high as 8.38% and 7.26%, respectively. Since part of Y element is dissolved in the α -Mg solid solution phase, the other existed in the X-Mg₁₂ZnY phase. In addition, higher content of Y element accompanied higher content of Zn element in the second stage, for example, at 150 and 200 °C, when the contents of Y element were 7.18% and 7.26%, the corresponding contents of Zn element were 2.74% and 2.71%, respectively. Mostly, Zn element had a content range of 2.23–2.58% in the first stage. Both increase in contents of Y and Zn elements suggest that that amount of X-Mg₁₂ZnY phase increases on the surface i.e., enriches during sliding in the second stage.

To further evaluate the role of LPSO structure phase in the three stages of wear rate, a thin layer of about 7–10 μ m thickness was removed off from worn surfaces by polishing, and then the surfaces were etched and observed under optical microscope. Figure 5 shows the microstructures nearby worn surface for sliding at 150 °C under 40, 80, and 120 N. At 40 N in the first stage, the LPSO structure phase underwent a little extent of deformation accompanied with Mg matrix along sliding direction (Figure 5a). However, at 80 N in the second stage, the LPSO structure phase was elongated significantly along sliding direction, resulting in formation of many strips of LPSO structure phase (Figure 5b). Meanwhile, the Mg matrix was observed to be dynamically recrystallized at locations between strips of LPSO structure phase. DRX realization enhanced the deformation ability of Mg matrix,

while strips of LPSO structure phase acted as fiber-enforcement in Mg matrix. The fiber enhancement effect of LPSO structure phase could be the reason for a little higher plateau with an almost constant wear rate in the second stage, namely played a masking effect on SWT. At 120 N in the third stage, a great number of slip bands were observed in LPSO structure phase as indicated by white arrows, meanwhile DRX grains grew bigger than they were at 80 N (Figure 5c). The occurrence of widespread slip deformation in LPSO structure phase implies the loss of fiber enhancement effect in the third stage, while the growth of dynamic recrystallization (DRX) grains suggests the decrease of deformation resistance in Mg matrix. These two factors could cause a significant rising of wear rate in the third stage.

Figure 5. Optical microstructures in the near surface region for sliding at 150 °C under different loads: (a) 40 N, (b) 80 N, (c) 120 N.

3.5. Microstructures and Hardness in Subsurfaces

The first-second wear transition i.e., SWT was dominated by severe plastic deformation. Since Mg alloy has a close-packed hexagonal structure, which decides a small number of sliding systems and limited ductility. However, it is found that the surface appears quite smooth due to severe plastic deformation, but no cracks are caused whether the test temperature is low or high. Therefore, the microstructure and property (e.g., hardness) in the subsurfaces were examined to explore the reason for severe plastic deformation.

Figure 6 shows the subsurface microstructures at 100 °C and 200 °C before and after SWT. For the sliding at 100 °C, at 60 N, the grains in the depth range from surface to about 200 μm (i.e., plastic deformation zone (PDZ)) were significantly affected by friction force. The grains were stretched towards surface, presenting a bended conical shape, while LPSO structure phase deformed with α -Mg grains by bending or kinking (Figure 6a). Meanwhile, it could be observed that a great number of deformation twins occurred in α -Mg grains, as shown in the magnification image of Figure 6b. As wear entered severe wear, at 80 N, the friction-affected region (FAR) reached about 242.3 μm depth underneath the surface (Figure 6c). As observed in Figure 6d, a special zone with about 40 μm thickness occurred

underneath surface in FAR, and its bottom part was followed by a PDZ. In the special zone, the grains were apparently refined and most of deformation twins disappeared, which could be as a result of a DRX microstructure activated by large frictional heating and great plastic deformation. For the sliding at 200 °C, at 20 N, the PDZ was about 150 µm thick (Figure 6e). A great number of plates of LPSO phase were found in the plastically deformed grains, as shown in Figure 6f. This could be because higher test temperature favors precipitation of LPSO phase since α -Mg dendrites in as-cast Mg₉₇Zn₁Y₂ alloy were supersaturated slightly during the fast cooling rate in the steel model. However, at 40 N in the severe wear, FAR reached about 280 µm depth (Figure 6g). A refined microstructure zone with about 30 µm thickness also appeared in the top part of FAR, and it was followed by a PDZ, as shown in Figure 6h. The precipitation of plate-like LPSO can exert a positive effect on deformation resistance of α -Mg, which contributes to a certain extent to the lowest level of wear rate in the second stage at 200 °C, and makes contribution to the masking effect on severe wear transition.

Figure 6. Cont.

Figure 6. Optical images of cross-sectional microstructures at 100 °C (a–d) and 200 °C (e–h) under different loads: (a) and (b) 60 N, (c) and (d) 80 N, (e) and (f) 20 N, (g) and (h) 40 N.

The microhardness variations with depth from surface at 100 °C and 200 °C are shown in Figure 7. For the sliding at 100 °C (Figure 7a), at 60 N in mild wear, with increasing depth, the microhardness descended continuously until the hardness of about 78 HV i.e., the original hardness of $Mg_{97}Zn_1Y_2$ alloy. The depth range for hardness descending was about 200 μm , approximately equal to the PDZ thickness in Figure 6a. This suggests that material is work-hardened in the plastic deformation zone due to plastic deformation, and work-hardening effect weakens with increasing depth. Nevertheless, at 80 N in severe wear, the hardness variation was quite different in the depth range from surface to about 40 μm , in which a low hardness slope appeared, and beyond that the hardness rose again and decreased continuously until the hardness of about 78 HV. The width of low hardness slope is about 40 μm , approximately equal to the refined microstructure zone thickness shown in Figure 6d. For the sliding at 200 °C (Figure 7b), the hardness at 20 N in mild wear also presented a continuous descending trend with increasing depth, while the hardness at 40 N in severe wear also exhibited a low hardness slope and a sequent decreasing trend with increasing depth. It is well known that DRX realization can eliminate work-hardening effect, resulting in a significant decrease in the hardness and improvement of ductility for deformed metallic materials. The low hardness slope in mild wear indicates the occurrence of softening near the surface, which confirms DRX realization in the refined microstructure zone. Therefore, the process for SWT is deduced to be as follows: on the one hand, with increasing applied load, the material undergoes larger plastic deformation; meanwhile much larger amount of friction heating is also produced, and contact temperature rises continuously. On the other hand, with increasing load to a critical value, the friction heat promotes surface reaching DRX temperature, deformed surface layer will be dynamically recrystallized and softened, therefore, wear begins to enter severe wear.

Figure 7. Microhardness versus depth from surface at 100 °C (a) and 200 °C (b).

3.6. Relationship between Critical Applied Load for SWT and Test Temperature

The critical load for SWT is plotted against test temperature (Figure 8). There is an apparent linear correlation between them. The critical load decreases linearly with increasing test temperature. After linearly fitting, the intercept of horizontal axis is $T_c = 335.6\text{ }^{\circ}\text{C}$, the slope is -0.25 . The critical applied load can be expressed as Equation (2).

$$F = 0.25(T_c - T) \quad (2)$$

Figure 8. Critical load versus test temperature at mild–severe wear transition and surface melting states.

As mentioned above, the real origin of SWT is the surface softening effect due to DRX realization, and it has been reported that the plastic deformation strain nearby surface is usually substantial enough for deformation requirement of DRX realization [9,11]. Thus, the surface temperature is the only one key factor for DRX realization. Once upon the surface temperature reaches the critical DRX temperature, DRX will take place in the plastically deformed surface layer. Lim and Ashby [20] pointed out that most of the work against friction was turned into heat during two surfaces sliding together. The amount of frictional heat increased almost linearly with applied load if under the sliding conditions with an approximately constant coefficient of friction, as expressed by Equation (3) [20].

$$q = \frac{\mu F v}{A_n} \quad (3)$$

where q is the heat generated per unit area per second, μ is the coefficient of friction, F is the normal load, v is the sliding speed and A_n is the nominal area of pin. In the present study, the coefficients of friction at different critical loads are nearly a constant around 0.32. Thus, q has an almost linear relationship with F . For a pin-on-disk tribo-system, the bulk surface temperature rising ΔT over the environmental temperature can be expressed by Equation (4) [20].

$$\Delta T = \frac{\alpha q l_b}{K_m} \quad (4)$$

where α is the fraction of frictional heat diffusing into the pin, K_m is the thermal conductivity and l_b is mean diffusion distance for pin. Because the wear rates at different critical loads are close to each other, consequently l_b varies little. K_m of $\text{Mg}_{97}\text{Zn}_1\text{Y}_2$ alloy varies little within 50–200 $^{\circ}\text{C}$, between 57 and 64 $\text{Wm}^{-1}\text{K}^{-1}$ [21]. α , l_b , and K_m can be approximately taken to be constants. Therefore, ΔT is almost linearly dependent on q , and approximately presents a linear relationship with F .

Apparently, with increasing test temperature, the difference between critical DRX temperature and test temperature i.e., ΔT ($\Delta T = T_{\text{DRX}} - T$) decreases, and the applied load required for compensating the temperature difference ΔT also decreases. There is an extreme case that when test temperature is increased to the critical DRX temperature, the critical load for DRX realization goes to zero. Therefore, it is reasonable to assume

that T_c (335.6 °C) is the critical DRX temperature for SWT within 20–200 °C under 2.0 m/s sliding speed.

Another approach to evaluate the contact surface temperatures at different transition loads is to refer to the surface melting temperature during sliding. Since $Mg_{97}Zn_1Y_2$ alloy begins to melt at 533.8 °C as a result of the eutectics (α -Mg + X- $Mg_{12}ZnY$) melting, the contact surface should also begin to melt at a constant temperature around 533.8 °C during sliding at different test temperatures. The applied load corresponding to commence of surface melting is also plotted against test temperature in Figure 8 for reference, and as expected that an almost linear relationship is found between them. After linearly fitting, the intercept of fitted line on horizontal axis is calculated to be 523.3 °C, roughly consistent with 533.8 °C i.e., the onset temperature of melting of $Mg_{97}Zn_1Y_2$ alloy measured by DTA. Since the surface temperatures at severe wear transition and surface melting states are dependent on frictional heat as described by Equation (3), at each test temperature, the ratio of temperature rising at SWT and surface melting states should follow the approximate relation expressed by Equation (5) if α , l_b , and K_m can be approximately taken to be constants.

$$\frac{\Delta T_1}{\Delta T_2} \approx \frac{F_1 \mu_1}{F_2 \mu_2} \quad (5)$$

where ΔT_1 and ΔT_2 are temperature risings from test temperature to 335.6 °C and 523.3 °C, respectively. F_1 and F_2 are the critical transition loads for severe wear and surface melting, respectively. μ_1 and μ_2 are the coefficients of friction at the two types of critical states, respectively. A comparison was made between the temperature rising ratio and friction force ratio using Equation (5). The calculated results are given in Table 4. It shows that the temperature rising ratio between SWT and surface melting states ranges from 0.42 to 0.63, and the friction force ratio at two states also ranges from 0.42 to 0.63. Furthermore, at each test temperature, the two ratios roughly agree well with each other. These results imply that the critical surface temperature for SWT is reasonably thought as around 335.6 °C.

Table 4. Ratios of temperature increase and frictional force at mild–severe wear transition state and surface melting state.

Temperature (°C)	ΔT_1	ΔT_2	$\Delta T_1/\Delta T_2$	F_1	F_2	μ_1	μ_2	$F_1 \mu_1 / F_2 \mu_2$
20	315.6	503.3	0.63	80	170	0.31	0.24	0.61
50	285.6	473.3	0.60	70	160	0.32	0.23	0.61
100	235.6	423.3	0.56	60	140	0.29	0.23	0.54
150	185.6	373.3	0.50	45	125	0.34	0.23	0.53
200	135.6	323.3	0.42	35	110	0.33	0.25	0.42

4. Conclusions

1. Wear rate presents a three-stage of increasing tendency with applied load at each test temperature. Wear rate increased gradually in the first stage, then moved up to a slightly higher plateau and kept an almost constant state in the second stage, and finally increased quickly to the highest level in the third stage.
2. Wear in the first stage manifested itself mild wear by delamination and mild plastic deformation, while wear in the second and third stage proved it severe wear by severe plastic deformation and surface melting.
3. A plateau state of wear rate rather than a rapid increasing state in the second stage was attributed to the fiber enhancement effect and precipitation effect of plate-like LPSO structure phase.
4. In mild wear, subsurface was plastically deformed and strain hardened, while in severe wear, microstructure transformation from the plastically deformed to DRXed happened in the near surface region, and the induced softening resulted in SWT.
5. The critical applied load for SWT decreases linearly with test temperature, which suggests that SWT is dependent on a common critical DRX temperature. The critical DRX was determined to be 335.6 °C by linear-fitting method.

Author Contributions: J.A. conceived and designed the experiments; F.T., H.D. and L.Z. performed the wear tests; F.T. performed SEM analysis; F.T. and H.D. analyzed the data; J.A. wrote the paper. All authors have read and agreed to the published version of the manuscript.

Funding: This research is fund by National Natural Science Foundation of China (Grant No.51775226).

Institutional Review Board Statement: Exclude this statement.

Informed Consent Statement: Exclude this statement.

Data Availability Statement: Exclude this statement.

Acknowledgments: The authors wish to express their gratitude for the support from National Natural Science Foundation of China.

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

References

1. Luo, Z.P.; Zhang, S.Q. High-resolution electron microscopy on the X-Mg₁₂ZnY phase in a high strength Mg-Zn-Zr-Y magnesium alloy. *J. Mater. Sci. Lett.* **2000**, *19*, 813–815. [\[CrossRef\]](#)
2. Kawamura, Y.; Hayashi, K.; Inoue, A.; Masumoto, T. Rapidly solidified powder metallurgy Mg₉₇Zn₁Y₂ alloys with excellent tensile yield strength above 600 Mpa. *Mater. Trans.* **2001**, *42*, 1171–1176. [\[CrossRef\]](#)
3. Mine, Y.; Yoshimura, H.; Matsuda, M.; Takashima, K.; Kawamura, Y. Microfracture behaviour of extruded Mg-Zn-Y alloys containing long-period stacking ordered structure at room and elevated temperatures. *Mater. Sci. Eng. A* **2013**, *570*, 63–69. [\[CrossRef\]](#)
4. Matsuda, M.; Li, S.; Kawamura, Y.; Ikuhara, Y.; Nishida, M. Interaction between long period stacking order phase and deformation twin in rapidly solidified Mg₉₇Zn₁Y₂ alloy. *Mater. Sci. Eng. A* **2004**, *386*, 447–452. [\[CrossRef\]](#)
5. Hagihara, K.; Kinoshita, A.; Fukusumi, Y.; Yamasaki, M.; Kawamura, Y. High-temperature compressive deformation behavior of Mg₉₇Zn₁Y₂ extruded alloy containing a long-period stacking ordered (LPSO) phase. *Mater. Sci. Eng. A* **2013**, *560*, 71–79. [\[CrossRef\]](#)
6. Gao, H.; Ikeda, K.; Morikawa, T.; Higashida, K.; Nakashima, H. Analysis of kink boundaries in deformed synchronized long-period stacking ordered magnesium alloys. *Mater. Lett.* **2015**, *146*, 30–33. [\[CrossRef\]](#)
7. Kim, J.; Sandlobes, S.; Raabe, D. On the room temperature deformation mechanisms of a Mg-Y-Zn alloy with long-period-stacking-ordered structures. *Acta Mater.* **2015**, *82*, 414–423. [\[CrossRef\]](#)
8. Chen, H.; Alpas, A.T. Sliding wear map for the magnesium alloy Mg-9Al-0.9Zn (AZ91). *Wear* **2000**, *246*, 106–116. [\[CrossRef\]](#)
9. Liang, C.; Lv, X.X.; An, J. Correlation between friction-induced microstructural evolution, strain hardening in subsurface and tribological properties of AZ31 magnesium alloy. *Wear* **2014**, *312*, 29–39. [\[CrossRef\]](#)
10. An, J.; Xuan, X.H.; Zhao, J.; Sun, W.; Liang, C. Dry sliding wear behavior and subsurface microstructure evolution of Mg₉₇Zn₁Y₂ alloy in a wide sliding speed range. *J. Mater. Perform. Eng.* **2016**, *25*, 5363–5373. [\[CrossRef\]](#)
11. An, J.; Tian, Y.X.; Feng, C.Q. Correlation between test temperature, applied load and wear transition of Mg₉₇Zn₁Y₂ alloy. *J. Mag. Alloys* **2021**, *9*, 592–603. [\[CrossRef\]](#)
12. Prasad, A.; Jain, J.; Gosvami, N.N. Effect of minor La addition on wear behavior of Mg-10Dy alloy. *Wear* **2021**, *486–487*, 204121. [\[CrossRef\]](#)
13. Taltavull, C.; Rodrigo, P.; Torres, B.; Lopez, A.J.; Rams, J. Dry sliding wear behavior of AM50B magnesium alloy. *Mater. Des.* **2014**, *56*, 549–556. [\[CrossRef\]](#)
14. Poddar, P.; Das, A.; Sahoo, K.L. Dry sliding wear characteristics of gravity die-cast magnesium alloys. *Metal. Mater. Trans. A* **2014**, *37*, 2270–2283. [\[CrossRef\]](#)
15. Niu, X.D.; An, D.Q.; Han, X.; Sun, W.; Su, T.F.; An, J.; Li, R.G. Effects of loading and sliding speed on the dry sliding wear behavior of Mg-3Al-0.4Si magnesium alloy. *Tribol. Trans.* **2017**, *60*, 238–248. [\[CrossRef\]](#)
16. Li, L.; Feng, J.; Liang, C.; An, J. Dry sliding wear behavior and mild-severe wear transition of Mg₉₇Zn₁Y₂ alloy at elevated temperatures. *Materials* **2018**, *11*, 1735. [\[CrossRef\]](#)
17. Zhang, J.; Alpas, A.T. Transition between mild and severe wear in aluminum alloys. *Acta Mater.* **1997**, *45*, 513–518. [\[CrossRef\]](#)
18. Li, X.X.; Zhang, Q.Y.; Zhou, Y.; Liu, J.Q.; Chen, K.M.; Wang, S.Q. Mild and severe wear of titanium alloys. *Tribol. Lett.* **2016**, *61*, 14. [\[CrossRef\]](#)
19. Farokhzadeh, K.; Edrisy, A. Transition between mild and severe wear in titanium alloys. *Tribol. Int.* **2016**, *94*, 96–111. [\[CrossRef\]](#)
20. Lim, S.C.; Ashby, M.F. Wear-mechanism maps. *Acta Metall.* **1987**, *35*, 1–24. [\[CrossRef\]](#)
21. Yamasaki, M.; Kawamura, Y. Thermal diffusivity and thermal conductivity of Mg-Zn-rare earth element alloys with long-period stacking ordered phase. *Scripta Mater.* **2009**, *60*, 264–267. [\[CrossRef\]](#)