Materials Research Express

PAPER

Investigation of temperature-induced wear transition of an Mg-10Gd-1.4Y-0.4Zr alloy under non-lubricated sliding conditions

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Keywords: wear transition, wear mechanism, non-ferrous metals, wear map, wear modeling

Abstract

The friction and wear behavior of an Mg-10Gd-1.4Y-0.4Zr alloy were investigated in detail within a temperature range of 20 °C–200 °C in order to clarify temperature-induced mild-severe (M-S) wear transition mechanism and verify if contact surface dynamic recrystallization (DRX) temperature criterion can be applicable to elevated temperature M-S wear transition. Coefficient of friction (COF) and wear rate (WR) were plotted against applied load at each test temperature, from which M-S wear transition loads were identified. A wear mechanism transition map was created on test temperature-applied load coordinate system, in which the mild wear region i.e. a safe working region in engineering application was indicated. The M-S wear transition mechanism was proved to be DRX softening by microstructural examination and hardness measurement in subsurfaces. The effects of precipitation and static recrystallization (SRX) occurred at temperatures of 150 °C–200 °C on M-S wear transition were also assessed. According to surface DRX temperature criterion, the transition loads were calculated at temperatures of 20 °C–200 °C, and the results identified applicability of the criterion to wear tests at elevated temperatures.

1. Introduction

Recently, new types of Mg-Gd-Y-Zr alloys have attracted great attention owing to their excellent elevated-temperature mechanical properties and creep resistance [1–5]. Furthermore, they can be significantly strengthened by heat treatments such as solid solution hardening and precipitation hardening [6–8]. Many research works have been made on Mg-Gd-Y-Zr alloys regarding microstructure evolution, grain refinement, precipitation behavior, mechanical properties, flow behavior as well as influence of addition of elements such as Y and Zn [9–16]. Generally, commercial Mg alloys such as AZ91D, AM50A and AM60B are currently limited to structural components working within a very limited temperature range, since unstable β-Mg17Al12 phase in Mg alloys is greatly softened at elevated temperatures above 120 °C. However, several Mg-Gd-Y-Zr alloys have potential as structural materials that can be employed at temperatures above 120 °C, and they are hence expected to find applications in elevated-temperature tribological field, for example, acting as alternatives to wear components that used to be made of aluminum alloys.

Conventional Mg alloys are usually considered not suitable for tribological applications such as bearings, sliding sales and gears. Nevertheless, a great number of studies still have been conducted on a variety of Mg alloys such as AZ, AM, AS and Mg-RE alloys for understanding the effects of experimental parameters (load and/or velocity) on wear performance, wear mechanisms and transitions [17–24]. Among the existing studies, the issue of transition between mild and severe wear in Mg alloys is the most of engineering interest. M-S wear transition in AZ91 alloy was first put forward by Chen and Alpas in 2000 [25]. As stated by them, wear behavior of Mg alloys that is greatly influenced by applied load and sliding speed can be divided into two kinds, namely mild wear and severe wear. Mild wear is acceptable in engineer application, since low wear rate, slight surface damage
and stable wear state are the typical features in this type of wear. Mild wear regime is consequently thought to be a safe operation region, whereas severe wear regime is a dangerous operation region. The two types of wear behavior have already been found on a large number of Mg alloys [21–25]. Since 2000, exploring the physical nature of M-S wear transition has long been a great concern for studying wear behavior of Mg alloys. An et al [24, 26–28] conducted a series of experimental investigations on several types of Mg alloys for clarifying M-S wear transition mechanism, and they finally generalized a criterion for M-S wear transition. They used the critical surface DRX temperature as a basis. The criterion indicates that M-S wear transition happens when frictional heating-induced surface temperature \( T_s \) exceeds the critical DRX temperature \( T_{DRX} \) of surface material, i.e. \( T_s \geq T_{DRX} \). However, it has not been proved whether the criterion applies to M-S wear transition of Mg-Gd-Y-Zr alloys at elevated temperatures yet. The mechanical behavior of metals during their hot and cold working tells us that the critical static recrystallization (SRX) temperature is typically only about 0.4 times the melting temperature of Mg alloys, lower than the DRX temperature. Inevitably, the SRX would involve in microstructure transformation in subsurfaces during sliding at elevated temperatures above 150 °C. In addition, it has not been revealed how the precipitation of intermetallic phase in the process of wear testing at elevated temperatures influences the wear performance of precipitation-hardening Mg-Gd-Y-Zr alloys. Only if these influencing factors are systematically studied, the temperature-induced M-S wear transition can be understood better for Mg alloys.

The objective of this study was to discover M-S wear transition mechanism of an as-cast Mg-10Gd-1.4Y-0.4Zr alloy at test temperatures of 20 °C–200 °C. The reason for choosing Mg-10Gd-1.4Y-0.4Zr alloy was that there was only a little amount of Mg5Gd intermetallic phase formed in the cast alloy. Consequently, the study could present mostly the wear characteristics of matrix of Mg-Gd-Y-Zr alloys, and the temperature-induced microstructural transformation in subsurface was less influenced by primary Mg5Gd phase. The M-S wear transition mechanism was elaborated from two aspects: microstructure transformation and property change in subsurfaces. The influences of SRX and precipitation of intermetallic phase on M-S wear transition were also analyzed, and if the surface DRX temperature criterion could be applicable to elevated-temperature wear transition was experimentally tested.

2. Experimental details

The studied material was Mg-10Gd-1.4Y-0.4Zr alloy (wt%). It was produced from pure magnesium and master alloys including Mg-17wt%Gd, Mg-15wt%Y and Mg-10wt%Zr alloys in an electric resistance furnace. A mixed gas of CO2–0.05%SF6 was used for protection of the melting process. The melt was firstly poured into a steel die, and then a cylinder ingot was yielded with dimensions of 100 mm diameter and 200 mm height. The chemical composition of the as-cast alloy was obtained by an inductively coupled plasma-atomic emission spectroscopy.

A pin-on-disk type wear apparatus was used for wear tests at various loads and test temperatures under dry sliding condition. Pin specimens (Ø 6 mm × 13 mm) were machined from the cast ingot by wire-electrode cutting. The disks (Ø 70 mm × 20 mm) were made from 50Cr steel, and then quenched and tempered to the hardness of 57HRC. The surface preparation of pins and disks included firstly grinding with SiC paper and finally polishing to 0.4 µm Ra prior to wear tests. The diameter of wear track was 60 mm. The sliding velocity and distance were 0.79 m/s (250 rotations per minute) and 565 m, respectively. The test temperatures were set as 20, 50, 100, 150 and 200 °C, respectively. The temperature range was chosen to cover full or a part of working temperature ranges of clutch piston and engine piston made of aluminum alloys, therefore the experimental results could provide useful reference for these applications. At each test temperature, the applied normal load began with 20 N, and then increased gradually until 300 N or observation of surface melting by naked eyes.

The predetermined test temperature was achieved using a resistance split furnace attached on the wear machine. The furnace guaranteed the desired temperature with an accuracy of ±5 °C using a thermocouple probe. To ensure a stable environment temperature during wear testing, before wear tests commenced, the pin and disk were preserved for 10 min WR was expressed as volume loss per unit sliding distance. Each WR datum was actually an average of three or more wear samples.

After elevated-temperature wear testing, the surface morphologies and chemical compositions of wear samples were analyzed using scanning electron microscope (SEM) attached with an energy dispersive x-ray spectrometer (EDS). The subsurface microstructures were examined with confocal scanning laser microscope. For comparing with subsurface microstructures, the subsurface microhardness was also measured and then plotted against depth beneath the worn surface.
3. Results and discussion

3.1. Material

XRD analysis of Mg-10Gd-1.4Y-0.4Zr alloy indentified the presence of $\alpha$-Mg phase and Mg$_5$Gd phase, as demonstrated in figure 1(a). The optical microstructure is shown in figure 1(b), from which it is noted that the microstructure is composed of the equiaxed $\alpha$-Mg grains with average size of about 31 $\mu$m surrounded by discontinuous network of Mg$_5$Gd phase. Since the amount of Mg$_5$Gd phase in the alloy is quite small, almost all XRD peaks of Mg$_5$Gd phase present a quite low intensity. Apparently, the discontinuous network of Mg$_5$Gd phase was formed in a mode of precipitation during the cooling process of the casting, and this point of view was supported by the subsequent thermal transformation analysis of the studied alloy using a differential thermal analyser (DTA). Two endothermic peaks were detected in DTA thermogram (not shown here), one occurred with a starting temperature of 361.4 $^\circ$C, which corresponded to the dissolution of Mg$_5$Gd precipitates; the other occurred with a starting temperature of 578.4 $^\circ$C, which corresponded to the melting of $\alpha$-Mg phase.

3.2. Friction and wear behavior at temperatures of 20 $^\circ$C–100 $^\circ$C

The variations in COF and WR of Mg-10Gd-1.4Y-0.4Zr alloy with applied load at temperatures of 20 $^\circ$C–100 $^\circ$C were illustrated in figures 2(a) and(b), respectively. As noted from figure 2(a), both test temperature and applied load have a great influence on COF. On the one hand, increasing test temperature decreased COF. For example, in the load range of 20–160 N, COF curve at 20 $^\circ$C was the highest, and then followed by the one at 50 $^\circ$C and finally the one at 100 $^\circ$C. On the other hand, all the three COF curves presented a significant decreasing tendency with the increase in load, and they could be subdivided into two obvious stages variety characteristic by the decreasing rate, i.e. a rapid decreasing stage and a moderate decreasing stage. The first stages ranged from 20 to
130 N at 20 °C, 20 to 100 N at 50 °C and 20 to 80 N at 100 °C, where the COFs rapidly decreased from 0.86 to 0.29 at 20 °C, from 0.66 to 0.32 at 50 °C, and from 0.60 to 0.33 at 100 °C. The second stages lay between 130 and 300 N at 20 °C, 100 and 240 N at 50 °C, 80 and 260 N at 100 °C. At the second stages, the COFs decreased moderately with the increase in load, and the COF curves got more and more flat until formation of a low level of about 0.25. The decreasing of COF with the increase in test temperature was mainly attributed to the decreased yield strength of the studied material by higher environmental temperature. However, the deduction of COF with increasing load may be ascribed to a combination of several factors such as frictional heating, surface contact area and surface morphology relating with wear mechanism. When the applied load was increased, more frictional heating was thus generated, and the deformation ability of surface material was significantly improved. These changes favored improving the contact situation between pin and disk, and consequently increased the real contact area, resulting in a reduction of COF. On the other hand, increasing load could also change the surface morphology in different load ranges. The surface morphology was often associated with the type of wear mechanism. The feature of surface morphology of Mg alloys frequently varied with different wear mechanism transformations such as oxidative wear, delamination, severe plastic deformation (SPD) and surface melting. As previously reported in several AZ alloys, COF typically decreased with mechanism transformation in such a sequence: abrasion, oxidative wear, delamination, SPD (also referred as thermal softening) and surface melting [27, 28].

As seen from figure 2(b), in general, WR curves demonstrated a rising trend with increasing load, and they also presented a stage characteristic according to increasing rate. WR curves could be separated into three variation stages, i.e. a rapid-to-moderate ascending stage, a rapid increasing stage, and a maintaining and reaching peak stage. The first stage was essentially in good agreement with the first stage of COF, namely they existed within the same load range. However, the second and third stages were not in accordance with the second stage of COF curves, the combined load ranges of them corresponded to the load range of second stage of COF. The first stages of WR curves lay between 20 and 130 N at 20 °C, 20 and 100 N at 50 °C, 20 and 80 N at 100 °C, where WRs were all below 30 × 10^{-12} m^3 m^{-1}. The second stages were found within load ranges of 130–180 N at 20 °C, 100–160 N at 50 °C and 80–150 N at 100 °C, where WRs went up considerably in an almost linear fashion with applied load except for a few points, such as 110 N at 50 °C and 100 N at 100 °C. The third stages ranged from 180 to 300 N at 20 °C, 160 to 240 N at 50 °C and 160 to 260 N at 100 °C, where WRs firstly maintained a high level or increase slowly within certain load ranges before a sudden reaching peak. At the first stage, although an abnormal phenomenon that WR was higher at 100 °C than at 100 °C was observed, there was not too much difference in WR for the three curves as the load was above 40 N. However, when wear entered the second and third stages, a great difference arose. The WR was the highest at 20 °C, but the lowest at 100 °C. Apparently, the first stages of the WR curves correspond to the mild wear behavior, judging by the notation of wear behavior of magnesium alloys proposed by Chen and Alpas [24], because the WR increases with load in a low slope, and the WR values are within the similar ranges in mild wear regime for AZ31, AZ51, AZ91, AS31 and Mg97Zn1Y2 alloys [24–28]. Therefore, when wear enters the second stage, it means that Mg-10Gd-1.4Y-0.4Zr alloy enters a severe wear regime. Based on the variations of WRs with load at the three test temperatures, the M-S wear transition loads were identified as 130 N at 20 °C, 100 N at 50 °C and 80 N at 100 °C.

The surface morphologies of the alloy worn at temperatures of 20 °C–100 °C were examined using SEM. EDS analysis gives chemical compositions of worn surfaces in table 1. The morphological characteristics of worn surfaces at 20 and 50 °C were very similar. Therefore, only the morphologies of typical worn surfaces at 50 and 100 °C are presented in figures 3 and 4, respectively. Under the condition of sliding at 50 °C, when subjected to 20 N, the surface was covered with a number of grooves together with small powders of debris detached from oxide layer (figure 3(a)). Furthermore, the oxygen content reached 11.32% on the surface. Hence the predominant wear mechanisms were abrasion and oxidative wear. At 40 N, delamination was apparently a main wear mechanism as the typical feature of delamination was pronounced, i.e. a quite number of surface cracks were formed perpendicularly to the sliding direction (figure 3(b)). Meantime, the oxygen content was 11.62%, but there was no sign of detachment of oxide layer from the surface, suggesting a heavy surface oxidation accompanying the delamination. These two mechanisms maintained until 100 N. Therefore, the wear mechanisms identified at the first stage were abrasion wear; oxidation wear and delamination wear + surface oxidation within 20–100 N. They are also typical wear mechanisms that were found in other Mg alloys during the mild wear behavior. With augmenting load to 110 N, the surface was severely deformed and heavily oxidized with a very high oxygen content of 13.96%. Meanwhile, since the oxide layer became porous and weak, this brought out an extensive small sheet spallation of oxide layer. The spalling of oxide layer was increasingly intense with increasing load until 160 N (figure 3(c)), and surface material was extruded towards the edge of sample (see top right corner of figure 3(c)). The oxygen content reduced to about 7.16% owing to large scale spalling of oxide layer. Influenced by dual factors of SPD and intense spalling of oxide layer (SOL), WR climbed a peak at 160 N. Therefore, the wear mechanisms at the second stage were identified as SPD and SOL within 110–160 N. As wear entered the third stage, i.e. within 180–200 N, the surface appeared smooth owing to SPD, but there was no
apparent sign of SOL even though the oxygen content was increased again to 9.75% (figure 3(d)), indicating that the main wear mechanisms were SPD and surface oxidation. At 220 N, surface melting began to be the main wear mechanism, since evident waved sign was observed on the surface as a result of melt flowing (figure 3(e)), and surface material underwent no massive plastic deformation at the edge of the sample (top right corner of figure 3(e)). Furthermore, at this applied load, the COF decreased to the lowest level of 0.25. It was noted that the surface morphology did not present a rather smooth surface and a typical multilayered structure edge that usually happened to AZ system alloys (AZ31, AZ51 and AZ91) in their surface melting mechanism. The predominant reason may be that heavy surface oxidation results in a high viscosity of surface melt. The oxygen content was 10.25% on the melt surface of the studied alloy, much higher than a usual level of 2%–7% that was detected on the melt surfaces of AZ alloys (AZ31 and AZ51) [27, 28]. Under the condition of sliding at 100 °C, when subjected to 20 N, the surface morphology also displayed an abrasion feature, and the oxygen content reached 13.02%, thus abrasion wear and oxidation wear were the dominant wear mechanisms. With loading to 40–80 N, the surface underwent slight plastic deformation and also demonstrated the feature of delamination (figure 3(f)). Meanwhile, the oxygen content stayed at high level of 9.22%–9.66%. It indicated that delamination and surface oxidation controlled the wear process. Therefore, the wear mechanisms were abrasion wear, oxidative wear, delamination and surface oxidation at the first stage. With loading to 100–160 N in the second stage, the worn surfaces were observed to be similar to those occurred within 110–160 N at 50 °C (figure 3(g)), and the oxygen content correspondingly reduced considerably with increasing load, for example 6.77% at 160 N. Therefore, the wear mechanisms were also SPD and SOL at the second stage. When the load was larger than 160 N, the wear was still dominated by SPD, however, SOL almost disappeared (figure 3(h)), and the oxygen content again rose to 14.27%. This indicates that SPD and heavy surface oxidation were the main wear mechanisms at the third stage. In the absence of oxide layer spalling, the persistence of heavy surface oxidation could be the reason for maintaining a WR plateau within 180–200 N. As the load was above 220 N, surface melting turned to be the wear mechanism.

### Table 1. Quantitative analysis of the main chemical elements on the worn surfaces (wt.).

| Temperature (°C) | Load (N) | Wear regime | O    | Gd  | Y    | Zr  |
|-----------------|----------|-------------|------|-----|------|-----|
| 50              | 20       | Mild        | 11.32| 8.19| 1.34 | 0.56|
|                 | 40       | Mild        | 11.62| 8.36| 1.44 | 0.48|
|                 | 110      | Severe      | 13.96| 7.08| 1.23 | 0.53|
|                 | 160      | Severe      | 7.16 | 8.75| 1.45 | 0.54|
|                 | 220      | Mild        | 10.25| 8.12| 1.35 | 0.46|
| 100             | 20       | Mild        | 13.02| 8.40| 1.30 | 0.42|
|                 | 40       | Mild        | 9.22 | 8.16| 1.39 | 0.43|
|                 | 80       | Severe      | 9.66 | 8.09| 1.37 | 0.45|
|                 | 90       | Severe      | 9.46 | 8.59| 1.48 | 0.44|
|                 | 120      | Severe      | 12.5 | 7.86| 1.33 | 0.46|
|                 | 140      | Severe      | 15.74| 7.58| 1.25 | 0.42|
|                 | 160      | Severe      | 6.77 | 8.17| 1.48 | 0.34|
|                 | 200      | Severe      | 14.27| 7.85| 1.30 | 0.36|
| 150             | 20       | Mild        | 11.11| 8.04| 1.35 | 0.45|
|                 | 60       | Severe      | 4.57 | 8.78| 1.52 | 0.47|
|                 | 90       | Severe      | 8.76 | 8.46| 1.39 | 0.38|
|                 | 120      | Severe      | 14.41| 7.68| 1.36 | 0.37|
|                 | 160      | Severe      | 3.93 | 8.78| 1.53 | 0.47|
|                 | 180      | Severe      | 7.85 | 8.82| 1.39 | 0.48|
| 200             | 20       | Mild        | 0.90 | 8.41| 1.47 | 1.53|
|                 | 50       | Severe      | 1.52 | 9.36| 1.51 | 0.54|
|                 | 80       | Severe      | 1.47 | 9.35| 1.61 | 0.65|
|                 | 100      | Severe      | 0.79 | 9.75| 1.59 | 0.96|
|                 | 140      | Severe      | 1.56 | 9.65| 1.65 | 0.50|
|                 | 160      | Severe      | 1.28 | 9.93| 1.67 | 0.47|
|                 | 180      | Severe      | 1.09 | 9.64| 1.59 | 0.7  |

3.3. Friction and wear behavior at 150 °C and 200 °C

The variations in COF and WR of Mg-10Gd-1.4Y-0.4Zr alloy with applied load at 150 and 200 °C are illustrated in figures 4(a) and (b), respectively. The test temperature exhibited an opposite effect on the COF as described at temperatures of 20 °C–100 °C, namely the COF was higher at 200 °C than at 150 °C in load ranges of 20–30 N and 80–140 N. Both COF curves could also be divided into two stages. They were a rapid decreasing stage and a
Figure 3. SEM micrographs of worn surfaces after sliding at 50 °C (a)–(e) and 100 °C (f)–(h) under different loads: (a) 20 N, (b) 40 N, (c) 160 N, (d) 200 N, (e) 220 N, (f) 80 N, (g) 100 N, (h) 200 N.
As seen from increasing load until a low value of 0.24 at 150 °C, stages were found within 60–120 N at 150 °C and 40–160 N at 200 °C, where COF decreased slightly with increasing load until a low value of 0.24 at 150 °C, whereas the COF decreased with large fluctuations at 200 °C. As seen from figure 4(b), the WRs increased with increasing load at 150 and 200 °C, and the two WR curves also displayed a three-stage feature. The first stages were found within 20–55 N at 150 °C and within 20–35 N at 200 °C, where WRs increased gently with increasing load, and all the WRs were below $23 \times 10^{-12} \text{m}^3\text{m}^{-1}$, suggesting a mild wear. The second stages were found within 60–120 N at 150 °C and within 40–80 N at 200 °C, where WRs went up remarkably with a great slope, indicating a severe wear. Therefore, M-S wear transition loads could be tentatively taken as 55 N at 150 °C and 35 N at 200 °C, respectively. In addition, an interesting phenomenon was observed, namely that WR at 150 °C was almost the same as it was at 200 °C within 20–40 N. The third stages were found within 120–180 N at 150 °C and 100–180 N at 200 °C, where WRs climbed again to their respective peaks, larger than $50 \times 10^{-12} \text{m}^3\text{m}^{-1}$. It was observed by the naked eyes that the worn surfaces were actually molten at 180 N and 160 N respectively when test temperatures were 150 and 200 °C.

SEM surface microphotographs of Mg-10Gd-1.4Y-0.4Zr alloy worn at 150 and 200 °C are demonstrated in figure 5. Under the condition of sliding at 150 °C, at low load of 20 N, the surface was slightly deformed, producing a flatten surface (figure 5(a)). This is attributed to the improved ductility of the material by high test temperature. The non-basal slip system in Mg alloys is activated when the temperature is higher than 120 °C. In addition, a number of cracks were also observed roughly vertical to the sliding direction, while EDS detected oxygen content of 11.3%. Therefore, delamination and surface oxidation were the wear mechanisms. These mechanisms prevailed within 20–55 N at the first stage. With increasing the load to 60–120 N in the second stage, the main wear mechanisms were transformed into SPD and SOL (figure 5(b)), meanwhile oxygen content ranged from 8.76% to 14.41%. This suggests that wear went in severe wear at the second stage. At 160 N, the SPD feature was much more significant, but the sign of oxide layer spalling was absent (figure 5(c)). Meanwhile, the oxygen content decreased considerably to a very low level of 3.93%, indicating that the worn surface can be considered as non-oxidized, and the wear mechanism is therefore a pure SPD. At 180 N, the wear mechanism was transformed into surface melting according to the produced smoothest surface morphology and the waved edge of pin (figure 5(d)). Under the condition of sliding at 200 °C, even at 20 N, the surface was easily deformed, but no delamination feature was observed, and a great amount of fine particles were found on the surface (figure 5(e)). Meanwhile, the oxygen content was rather low, only 0.90%. This suggests that oxide layer cannot exist owing to a large plastic deformation. At the loads of 40–80 N, the surface displayed the features of SPD and extruded edge (figure 5(f)), while the oxygen content ranged from 0.79% to 1.47%. SPD was the wear mechanism. At loads of 100–140 N, another wear mechanism was found besides SPD, it was adhesion that resulted in furrows on the worn surfaces (figure 5(g)). Apparently, adhesion brought out a great rising of COF at loads of 100–140 N. At 160 N, surface melting started to be the wear mechanism, since the waved sign and a little sign of multilayered structure were observed on the centre and edge of the pin (figure 5(h)). The elemental mapping revealed that Zr-rich particles were formed on the surface subjected to 20 N, as shown in figure 6. The particles are supposed to be $\beta'$ phase precipitated at 200 °C [29]. These fine particles can greatly increase the hardness of the material as described in section 3.5, and accordingly give rise to a higher frictional force i.e. COF at 200 °C than at 150 °C within 20–30 N, as well as the almost same WR.
Figure 5. SEM micrographs of worn surfaces after sliding at 150 °C (a)–(d) and 200 °C (e)–(h) under different loads: (a) 20 N, (b) 100 N, (c) 160 N, (d) 180 N, (e) 20 N, (f) 80 N, (g) 120 N, (h) 160 N.
3.4. Wear mechanism transition map

In accordance with the definition given by Chen and Alpas [25], wear performance of Mg-10Gd-1.4Y-0.4Zr alloy can also be classified into two classes at 20 °C–200 °C. They are mild wear and severe wear. Typically, both wear regimes usually consist of several sub-regimes, and each sub-regime corresponds to a primary type of wear mechanism. Figure 7 is the elevated-temperature wear mechanism transition map of Mg-10Gd-1.4Y-0.4Zr alloy, which was drew on test temperature-applied load coordinate system. The map was separated into two regions by boundary line AA’. They were the top severe wear region and the bottom mild wear region. The mild wear region was composed of three subregions, where abrasion, oxidation, delamination, surface oxidation and mild plastic deformation were the main wear mechanisms. The severe wear region was made up of five subregions, where SPD, SOL, surface oxidation, adhesion and surface melting were the predominant wear mechanisms. These subregions were separated from each other by dashed lines BB’, CC’, C’C”, DE’A’, D’E’ and EE’, respectively. The boundary line AA’ was plotted by taking into account a number of factors including turning points of WR curves, appearance of SPD and extruded edge, and so on. The boundary lines of various subregions were mainly determined by SEM and EDS analyses of the variety of wear mechanisms. Key features used to separate various subregions from each other are summarized in table 2.

Figure 6. SEM micrograph and elemental mappings of worn surface at 20 N after sliding at 200 °C: (a) SEM micrograph, (b) Gd, (c) Y, (d) Zr.
3.5. Microstructures and hardness in subsurfaces

Figure 8 shows the cross-sectional microstructures of pins subjected to various applied loads after sliding at 100 \(^\circ\)C. When wear was mild, for example, at 20 N, beneath the surface was an about 10 \(\mu\)m thick dark-etched zone, which was actually a mechanically mixed layer (MML) composed of magnesium oxide and iron oxide (figure 8(a)). Next to it was an about 70 \(\mu\)m thick plastic deformation zone where the alloy was plastically deformed by the friction force and normal load. Consequently, \(\alpha\)-Mg grains adjacent to the surface were deformed along the sliding direction, and a large number of twins were produced at the bottom of deformation zone. At 60 N, an extended deformation zone was formed beneath about 20 \(\mu\)m thick MML. The deformation zone was enlarged to about 85 \(\mu\)m thickness. Deformation twins became so small that they could not be well recognized at top location, whereas large twins were still seen at bottom location at high magnification (figure 8(b)). When wear was severe, for instance, at 100 N, only part of surface was covered by MML, beneath that was an about 180 \(\mu\)m thick friction-affected zone (FAZ) (figure 8(c)). At high magnification, FAZ consisted of two sub-zones, i.e. beneath the surface were an about 10 \(\mu\)m thick distinct sub-zone and an about 170 \(\mu\)m thick plastic deformation sub-zone (figure 8(d)). The top sub-zone could be composed of DRX microstructure, because twins were absent and a certain amount of fine grains appeared to be formed besides elongated \(\alpha\)-Mg grains in the sub-zone, while twins were clearly observed in the plastic deformation sub-zone. At 140 N, wear test produced a larger FAZ with 240 \(\mu\)m thickness. It was also composed of two sub-zones: DRX and plastic deformation sub-zones. They were about 40 \(\mu\)m thick and 200 \(\mu\)m thick, respectively. At high magnification, it was found that in the DRX sub-zone, the original elongated coarse \(\alpha\)-Mg grains apparently experienced a microstructure transformation, which brought out fine grains (figure 8(e)). The DRX grains were very fine and formed at locations of original twins in \(\alpha\)-Mg grains. At 200 N, surface melting happened, while an about 180 \(\mu\)m thick FAZ was produced. The FAZ was composed of three sub-zones. From top to bottom the sub-zones were in order, solidified sub-zone, DRX sub-zone, deformation sub-zone. As seen from the photograph (figure 8(f)), there exists fine solidified grains and residue elongated \(\alpha\)-Mg grains in the solidified sub-zone due to the partly melting process. In the DRX sub-zone, a lot of newly formed fine equiaxed DRX grains in micrometer scale together with elongated \(\alpha\)-Mg grains were observed. In the plastic deformation sub-zone, a number of twins were clearly found to bend towards the sliding direction.

It is well known that DRX microstructure transformation gives rise to DRX-induced softening for metallic materials. Therefore, the microhardness change in subsurface was examined on several selected specimens that were worn in mild and severe wear respectively. Figure 9(a) shows the subsurface microhardness distributions for specimens subjected to various applied loads of 20–70 N at 100 \(^\circ\)C in mild wear. Because MMLs were formed on the worn surfaces at 100 \(^\circ\)C, the hardness comparison should be made by excluding the effect of MML. It can be seen that beneath the MML, i.e. deeper than 10–20 \(\mu\)m, as the load varied from 20 to 70 N, an increasingly intense strain-hardening effect showed in FAZs. The hardness demonstrated a monotonically decreasing relationship with the depth from surface until 80 HV, namely the original hardness of the studied alloy. The hardness was even higher than 100 HV at depth of 20–30 \(\mu\)m under 60 and 70 N. This is obviously attributed to the influence of deformation-induced strain hardening. However, as seen from figure 9(b), the subsurface...
| Sub-regime                                      | Dominant wear mechanism | Oxidation degree | Surface morphology                                                                 | Surface damage |
|-----------------------------------------------|-------------------------|------------------|------------------------------------------------------------------------------------|----------------|
| Abrasion + Oxidation (Ab. + Ox.)              | Abrasion                | High             | Grooves and fine oxide powders                                                     | Mild           |
| Delamination + surface oxidation (De. + S.O.) | Delamination            | High             | Cracks and no evident oxide layer detachment                                        | Mild           |
| Slight plastic deformation (S.P.D.)           | Plastic deformation     | Low              | Slightly flatten surface                                                            | Mild           |
| Severe plastic deformation + spallation of oxide layer (S.P.D. + S.O.L.) | Severe plastic deformation | High         | Severely deformed surface and oxide layer detachment                                | Severe         |
| Severe plastic deformation (S.P.D.)           | Severe plastic deformation | Low              | Severely deformed surface and extruded edge                                        | Severe         |
| Severe plastic deformation + adhesion (S.P.D. + Ad.) | Severe plastic deformation | Low              | Severe Plastic deformation and adhesive furrows                                     | Severe         |
| Surface melting (S.M.)                        | Surface melting         | High or low      | Waved surface and/or multilayered structure edge                                   | Severe         |
hardness distributions for the pins under 100 and 140 N in severe wear were greatly different from those in mild wear. For purpose of comparison, the hardness distribution at 70 N was also included in figure 9(b). It was noted that the hardness values of specimens at 100 and 140 N were apparently lower at depth of 0–50 μm as compared with the values at 70 N. In addition, a plateau and a V-shaped valley were formed at depth of 20 and 30 μm on hardness curves under 100 and 140 N, respectively. They are typical subsurface softening features. The subsurface softening phenomenon in severe wear is greatly attributed to the DRX realization, on consideration that a DRX microstructure transformation happened after initial plastic deformation. Moreover, the plateau and V-shaped regions were found having almost the same widths as the DRX sub-zones observed in figures 8(d) and (e). Based on above results of analysis, DRX softening in subsurface was confirmed as the dominant mechanism of M-S wear transition during wear testing at 50 °C–100 °C.

Two types of microstructure transformation would happen to the pin subsurface during sliding at elevated temperatures of 150 and 200 °C. They were precipitation of intermetallic phases and SRX. The studied material was actually composed of supersaturated α-Mg phase owing to a fast cooling rate in steel die during casting process. It was reported that solid solutionized and as-cast Mg-Gd–Y-Zr alloys could precipitated intermetallic phases at temperatures of 100 °C–250 °C, in which a series of phase transformations were identified, such as

Figure 8. Subsurface microstructures of test pins after sliding at 100 °C under different loads: (a) 20 N, (b) 60 N, (c) 100 N, (d) 100 N, (e) 140 N, (f) 200 N.
supersaturated solid solution (s.s.s.s. → β′ → β → β(Mg, Gd)) [30]. It is well known that the plastically deformed Mg alloys would experience SRX microstructure transformation if they are heat treated at temperatures above 150 °C [31, 32]. The subsurfaces of Mg alloys typically suffered from a large plastic strain even under a low load of 20 N during a short period of wear testing [27]. Therefore, the transformation of deformed microstructure-SRX microstructure might occur at a certain depth in subsurface during sliding at elevated temperatures above 150 °C. An additional experiment was therefore conducted to verify SRX realization.

On the basis of understanding of precipitation and SRX in subsurface of the studied alloy, the subsurface microstructure evolution during wear testing at 200 °C was therefore examined. The microstructural evolution at 200 °C under various applied loads was shown in figure 10. When wear was mild, loading 20 N produced an about 150 μm thick FAZ (figure 10(a)). At high magnification, the FAZ was composed of three sub-zones. From top to bottom they were, in order, a plastic deformation sub-zone, a distinct sub-zone, and again a plastic deformation sub-zone. They were about 10 μm, 35 μm and 110 μm thick, respectively. The distinct sub-zone consisted of newly formed fine grains in micrometer scale between elongated coarse Mg grains. This type of fine grains in middle sub-zone was different from the DRX grains formed during wear test, because they were much larger the friction-induced DRX grains and mainly occurred within a certain depth range under the worn surface. These fine grains could be SRX grains since the test temperature 200 °C was higher than the SRX temperature of 150 °C for the studied alloy. The twins in the top and bottom sub-zones of plastic deformation can be observed clearly in figure 10(b). Increasing to the load of 35 N resulted in a wider FAZ of about 220 μm thickness. The FAZ also can be divided into three sub-zones similarly (figure 10(c)). The thickness of the three sub-zones was about 20 μm, 30 μm, 170 μm, respectively. When wear was severe, for instance, loading 70 N created an about 240 μm thick FAZ (figure 10(d)). The FAZ was composed of three sub-zones. The sub-zones were DRX, SRX and plastic deformation sub-zones. They were about 20 μm, 30 μm and 190 μm thick, respectively.

The corresponding hardness distributions in subsurfaces are demonstrated in figure 11. In mild wear, with loading from 20 to 35 N, the strain-hardening effect gave rise to the increase of hardness correspondingly at the depth of 0–20 μm, and then both hardness curves presented a V-shaped decreasing-rising characteristic within certain depth ranges, namely 20–50 μm at 20 N and 20–90 μm at 35 N, and eventually reduced to about 82HV, i.e. the unaffected substrate hardness. The V-shaped hardness variation was in fact related with softening originating from SRX microstructure. In severe wear, at 70 N, it was apparent that the hardness became much less at depth of 0–20 μm when compared with the hardness at 20 and 35 N, and a wide hardness valley appeared at depth of 20–100 μm. These two depth ranges corresponded to the DRX sub-zone and SRX sub-zone, respectively. This implies that the two types of hardness variations are ascribed to DRX softening and SRX softening, respectively. Therefore, it can be deduced from the hardness distributions that M-S wear transition is still dominated by DRX softening of surface material. SRX has a small influence on softening of surface layer material. The precipitation of the studied alloy during sliding at different test temperatures was first found in the hardness distributions in subsurfaces. The unaffected substrates far from wear surface presented a slight increasing trend with rising test temperature. Their average hardness values were about 76 HV at 20 °C, 80 HV at 100 °C, and 84 HV at 200 °C, respectively. The precipitation effect was also confirmed by subsequent aging treatment of the as-cast alloy at temperatures of 50 °C–210 °C for 25 min (equal to the total wear testing period). The hardness of the aged alloy showed a gradual increasing trend with treatment temperature similar to that in unaffected substrates. The precipitation hardening of the studied alloy during wear testing exhibited a significant
influence on the variation of wear rate with applied load at temperatures of 150 and 200 °C. After M-S wear transition, the increasing of wear rate was not as pronounced as that at temperatures of 20 °C–100 °C, that is, the wear rate increased with applied load in a lower slope at 150 and 200 °C.

3.6. Prediction of transition load
The surface DRX temperature criterion was originally put forward for prediction of M-S wear transition for Mg alloys under room-temperature wear testing [27]. Since M-S wear transition of Mg-10Gd-1.4Y-0.4Zr alloy at 20 °C–200 °C was still governed by DRX transformation in subsurface, this criterion can be also expected to apply to elevated-temperature wear situations. When the sliding wear test was conducted at 0.79 m/s and 20 °C,
the average strain rate was measured at depth of 10–20 \( \mu \text{m} \) in subsurface. It ranged from 9.80 \( \times \) 10^{-3} to 8.75 \( \times \) 10^{-2} \( \text{s}^{-1} \). In order to obtain the critical DRX temperature under condition of 0.79 m/s, hot compressive deformation tests were performed on Mg-10Gd-1.4Y-0.4Zr alloy at strain rate of 1.0 \( \times \) 10^{-2} \( \text{s}^{-1} \) within a temperature range of 260 °C–340 °C. The critical DRX temperature was identified as 290 °C by analysis of microstructure evolution. It was found that the microstructure did not change significantly at 280 °C, whereas a necklace of fine DRX grains grew mostly in the interior of Mg grains at 290 °C, as shown in Figure 12.

The surface temperature \( T_s \) is formulated by temperature \( T \), load \( F \), sliding velocity \( v \), COF \( \mu \), thermal conductivity of pin \( K_{mp} \), and so on, using the equation \( (1) \) given by Lim and Ashby [33]

\[
T_s = T + \frac{\alpha \mu F v l_b}{A_n K_{mp}}
\]

where \( l_b \) is the mean diffusion distance, \( A_n \) the nominal contact area, and \( \alpha \) the fraction of the heat conducted into the pin. It has been reported that a steady-state temperature distribution was typically achieved between pin and disk in mild wear for Mg alloys and Al alloys [25, 34]. Even in a condition of critical M-S wear transition, \( \alpha \) can be approximately regarded as a constant. Considering that at the critical M-S wear transition, the volumetric losses at different test temperatures are much smaller as compared the volumes of specimens, \( l_b \) and \( A_n \) can also be considered as constants. Since the difference in the thermal conductivity values of AZ and Mg-Gd/Y alloys varies little within 50 °C–200 °C, for example, from 92 to 105 Wm^{-1}K^{-1} for AZ31 alloy, 38 to 47 Wm^{-1}K^{-1} for Mg-10Gd-2Zn (Mg_{97}Zn_{2}Gd_{1}) alloy and 63 to 72 Wm^{-1}K^{-1} for Mg-6Y-5Zn (Mg_{96}Zn_{2}Y_{2}) alloy [35, 36]. The thermal conductivity \( K_{mp} \) can also be approximately taken as a constant in the present study. Accordingly, equation \( (1) \) can be simplified to equation \( (2) \) by inserting an approximate constant \( K_{DRX} \).

\[
F = \frac{(T_s - T)}{K_{DRX} \mu v}
\]

\[
K_{DRX} = \frac{\alpha l_b}{A_n K_{mp}}
\]

It was noted that the COF at transition load was affected by test temperature. It varied little within temperature range of 20 °C–100 °C, about 0.30–0.32, but went up to 0.39 and 0.40 as test temperatures were 150 and 200 °C, respectively. Considering the less oxidized surface at 150 °C, the value of \( K_{DRX} \) was ascertained as 9.14 using the following data of Mg-10Gd-1.4Y-0.4Zr alloy: \( T_{DRX} 290 °C, F_T 50 \text{N and } \mu 0.39 \). The transition loads were therefore calculated by inserting their respective test temperature and COF. The variations of measured and calculated transition loads with test temperature are illustrated in Figure 13. The calculated and measured transition loads in general conform very closely to each other except for the values at 100 °C, where a big deviation is 4.5 N, about 4.5% of the measured transition load. These calculated results prove that the surface DRX temperature criterion can also assess the elevated-temperature M-S wear transition.

### 4. Conclusions

1. The studied alloy exhibited mild and severe wear within the tested temperature range. M-S wear transition load decreased with increases in test temperature, and an approximate linear relationship was found between them.
2. The subsurface microstructures experienced transformations such as precipitation and SRX at temperatures of 150 °C–200 °C, but these transformations did not influence the elevated-temperature M-S wear transition mechanism.

3. Under the action of normal load, frictional force and test temperature, it was the resulting DRX softening in surface material that triggered M-S wear transition.

4. The elevated-temperature induced M-S wear transition of Mg-10Gd-1.4Y-0.4Zr alloy could be evaluated by the contact surface DRX temperature criterion within temperature range of 20 °C–200 °C.

Acknowledgments

This work is supported by the National Natural Science Foundation of China (NSFC) under Grant No.51775226.

Author Contributions

An Jian conceived and designed the experiments; Wang Yuanbo and Liu Zhihui performed the elevated-temperature wear measurements and performed SEM analysis; Sun Yuan and An Jian analyzed the data; Sun Yuan and An Jian wrote the paper.

Conflicts of Interest

The authors declare no conflict of interest.

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