Wear behaviour of AZ91D magnesium alloy with a nanocrystalline surface layer

H.Q. Sun \(^a\), Y.-N. Shi \(^a,⁎\), M.-X. Zhang \(^b\)

\(^a\) Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, China
\(^b\) Division of Materials, School of Engineering, University of Queensland, St Lucia, Brisbane, Qld 4072, Australia

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Abstract

The tribological behaviour of nanocrystalline surface layer with an average grain size of 30 ± 5 nm generated by surface mechanical attrition treatment on AZ91D Mg alloy samples has been investigated under dry sliding conditions. Compared with the alloy without SMAT, nano-grained surface layer showed lower friction coefficient. An improved wear resistance of the NC layer was found at the load ranges from 3 N to 9 N due to the grain refinement strengthening effect. Examination of the worn surface and the wear debris indicated that the wear mechanism of NC layer is similar to that of the coarse-grained alloy. Cooperative effects of cutting, plowing and oxidation govern the tribological behaviour of nanocrystalline AZ91D Mg alloys.

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1. Introduction

Mg alloys are attracting high interest in structural components in automotive industry due to their lightness and high specific strength. However, the poor wear resistance extensively limits their practical applications. A number of techniques \(^1–4\) have been developed to improve the tribological behaviour of Mg alloys through surface hardening or strengthening. As nanocrystalline (NC) materials have much higher strength than their coarse-grained (CG) polycrystalline counterparts \(^5–7\), surface nanocrystallization would provide a powerful tool to increase the wear resistance of materials. Surface mechanical attrition treatment (SMAT) developed by Lu et al. \(^8\) represents the most effective technique of surface nanocrystallization. It has been successfully used to generate NC grains in the surface layer of pure metals \(^9,10\) and alloys \(^11,12\) through severe surface plastic deformation. The thickness of the NC layer generated from SMAT varies with materials. Previous result \(^12\) showed that a 100 μm thick NC layer on an as-solution-treated AZ91D Mg alloy could be obtained after 20 min SMAT, which has an average grain size of 30 ± 5 nm. The micro-hardness of the NC on the top surface is 2 times as much as the matrix.

The wear mechanism of Mg alloys is complicated and inconsistent. Chen et al. \(^13\) investigated the sliding map for conventional AZ91D Mg alloy and found two wear regimes, mild wear regime and severe wear regime. The former corresponded to oxidation wear and delamination wear, and the latter was related to severe plastic deformation induced wear and melt wear. Shanthi and co-workers \(^14\) studied the effect of grain size on the wear behaviour of recycled AZ91D Mg alloy with grain size ranging from 0.6 μm to around 30 μm. Under a constant load, no distinguishable difference in wear performance was observed at different grain sizes during dry sliding wear over a wide range of sliding speed. From the examination of the worn surface and the wear debris, abrasive wear and oxidation wear were identified. However, the smallest grain size that Shanthi \(^14\) investigated is 600 nm, which cannot be regarded as NC grains, and their experiments were only carried out at a constant load. Therefore, it would be useful and necessary to investigate the tribological behaviour of nanocrystalline Mg alloy at various loads. In addition, both Chen \(^13\) and Shanthi \(^14\) have found that the abrasive wear and the
plastic deformation induced wear are two major wear mechanisms on AZ91D Mg alloy. Hence, according to Hall–Petch relationship and Archard’s Law [15], it is expected that the abrasive wear behaviour of nanocrystalline Mg alloy would be significantly improved.

The present work aims to investigate the wear behaviour of the NC layers generated by SMAT on an AZ91D Mg alloy, and to identify the possible wear mechanism of the NC materials under dry sliding condition at various loading level through scanning electron microscopy (SEM) analysis on both the worn surface and the wear debris.

2. Experimental

Commercial AZ91D magnesium alloy ingots with a composition of Al 8.47wt.%, Zn 0.69wt.%, Mn 0.14wt.% were cut into 100 × 100 × 12 mm plates. These plates were solution treated at 686 K for 24 h followed by water cooling. After grinding down to 10 mm thick and finalized with grade 1000 silicon carbide sand paper, SMAT was carried out in vacuum in SNC-II surface nanocrystallization treatment machine for 20 min at room temperature. The detailed description of the SMAT process has been presented in previous work [8,9].

The nanocrystalline layer of the plates after SMAT was examined in a JEOL 2010 transmission electron microscopy (TEM) operated at 200 kV. Detailed foil preparation and the experimental process were described in reference [12]. Variation of microhardness with the distance from the surface of SMATed sample was also reported in reference [12].

Dry sliding wear tests were performed on an Optimol SRVIII oscillating friction and wear tester at room temperature (25°C) in air with a relative humidity of 40–50%. The machine was equipped with a ball-on-disc contact configuration. In the present work, 8 × 8 × 3 mm blocks were cut from the SMATed samples and WC-Co balls with a hardness of HV-1750 were used as the counter friction pair. Testing details are as follows: 2 mm oscillating stroke with a frequency of 5Hz, and applied load ranging from 3 to 15 N were used; wear depth and wear volume were determined after 30 min sliding. For comparison, some SMATed blocks were annealed at 686K for 24h in argon to obtain CG samples with the same surface roughness (Ra, about 3.7 μm) as the NC samples. The tribological behaviours of these CG samples were explored under the same conditions.

The profiles of the worn surfaces were measured using a surface profile-meter and the wear depth and volume were calculated by the method proposed by Qu et al. [16]. The morphologies of the worn surfaces and debris at different testing conditions were examined in a Quanta 600 MK2 scanning electron microscope (SEM) operated at 20 kV. Energy dispersive spectrum (EDS) was used to determine the content of oxidation of the worn surfaces and the debris. Prior to SEM observations, the collected debris and the worn surfaces were ultrasonically cleaned in acetone for about 10min.

3. Results and discussion

Typical NC microstructure in the topmost surface layer of the SMATed AZ91D Mg alloy was shown in Fig. 1. The average grain size within this layer is 30 ± 5 nm [12]. Fig. 2 shows the microhardness variation with the distance from the SMATed top surface to the substrate [12]. The hardness on the top surface is 1.8 GPa, which is almost two times the value of the substrate. The thickness of the whole deformed layer is 1500 μm, which includes a NC layer of about 100 μm thick layer as indicated previously [12].
3.1. Friction coefficient

Fig. 3 shows the variation of friction coefficient as a function of sliding time at 13 N applied load. It is illustrated that the friction coefficient rises slightly with the increase of sliding time for both the NC and the CG samples. However, when the testing time is less than 6 min, both the CG sample (a) and the NC sample (b) have similar friction coefficient. As the time increases, the friction coefficient of NC sample drops slightly compared with the CG one, the average value of which tends to be a constant.

Previous work on pure copper [17] shows that the increasing in friction coefficient with the sliding time is due to work-hardening or to accumulating of debris. But, for AZ91D Mg alloys, as Mathis [18] pointed out in his work, there is no work-hardening observed during friction and wear process for both CG and fine-grained samples. Hence, the reduction of friction coefficient of NC as shown in Fig. 3 can be attributed to the refined grains. To further investigate the effect of NC surface layer generated from SMAT, friction coefficient was determined at different applied load at the sliding time of 30 min. As shown in Fig. 4, although the friction coefficients of both the NC and the CG magnesium alloys are quite similar with the value of the NC layer slightly lower than that of CG when the loads are less than 7 N, the difference gets evidenced at the applied loads over 7 N. When the applied loads are higher than 9 N, the friction coefficient of the two samples remains almost unchanged. It seems that surface nanocrystallization leads to the reduction of the friction coefficient. Lim and Ashby [19] indicated that the friction coefficient depended on the real contact area, the contact state and the lubricant role of debris. For magnesium alloy, the higher applied load leads to an enlargement of contact area, therefore reduces the friction coefficient of the both samples. Compared with the conventional CG grains, NC samples, which normally have higher hardness, will result in lower friction coefficient, because harder surface normally involves smaller contact area at the same applied load.

3.2. Wear resistance

Wear resistance can be expressed by either wear depth or wear volume or both. Fig. 5 shows the variation of wear depth of the NC and CG AZ91D Mg alloy with the applied load at a fixed sliding time (30 min). When the applied load is less than 10 N, the wear depth of the NC layer is below 100 μm and is always smaller than that of the CG sample. When the load is over 10 N, the wear depths of both the NC and the CG samples tend to be the same. The experimental results in Fig. 5 signify that the NC AZ91D Mg alloy has higher wear resistance than that of the conventional alloy with coarse grains. Once the wear depth is beyond 100 μm, the NC layer was worn off, so that the wear behaviours of both the samples with and without SMAT tend to be the same. The inset in Fig. 5 is a typical cross-section profile of the worn scars of the NC (a) and the CG (b) AZ91D Mg alloy at the applied load of 7 N. The cross-section profiles
present an arc figuration. The profile shows 90 μm wear depth for the NC sample and 120 μm for the CG sample. This also implies a better load-bearing ability of the NC Mg alloys than the CG alloys. The profile in Fig. 5 is different from the reported results of copper [17], no material accumulation was found along the edges of the scar in the present NC and CG AZ91D, which might be related to the poorer plastic deformation ability of Mg alloys.

Fig. 6 shows the variation of wear volume with the applied load at sliding time of 30 min. The variation tendency is similar to that of the wear depth. The NC layer exhibits a lower wear volume than the CG sample at load range of 3 N to 9 N, but a similar wear loss to that of the CG at higher loads above 9 N. The NC layer generated by SMAT shows an enhanced wear resistance in comparison to the CG sample when the applied load ranges from 3 N to 9 N.

3.3. Wear mechanism

In order to understand the wear mechanism of both the NC and the CG AZ91D Mg alloys, the worn surfaces of the two samples were characterized by using SEM. Fig. 7 is the typical worn surface morphologies of samples after sliding wear test for 30 min at applied load of 7 N of both the NC and the CG samples. No significant metallographic difference can be observed between the NC and the CG samples. Numerous ridges and grooves that are parallel to the sliding direction with some randomly distributed particles can be found. Traces of abrasive particles pointed by the white arrows are another feature of the worn surfaces. According to Shanthi and Mondal’s results [14,20], the above metallographic characteristic of the worn surface represents typical abrasive wear.

During wear process, hard asperities on the WC-Co ball counter-face, or hard particles between the WC-Co ball and the block, plow or cut into the metal block, which causes wear through the removal of small fragments and consequently forms the scratch on the block surface.

Examination of the wear debris is another important technique to understand the wear mechanism. Fig. 8 presents three types of SEM morphology of the wear debris from both the NC and the CG AZ91D Mg alloy. Fig. 8(a) is a typical ribbon-shaped particle taken from the NC sample, which has the characteristics of machining chips produced by a Vee-point tool. It is smooth on one side and serrated on the opposite side. This indicates abrasive mechanism of cutting [21]. It is also noticed that the serrated side of the particle is composed of some parallel fringes marked by the white arrow with an average spacing of about 2 μm. Campbell et al. [22] considered it as the indication of the formation of shear bands. Similar particles were also observed in the CG sample. Fig. 8(b) is typical lathy-shaped debris that can be found in both the NC and the CG samples, even though the present micrograph was taken from the CG sample. This also implies the abrasive mechanism of plowing. In this case, plowing induces fracture and the separation of debris from the matrix. In addition, Fig. 8(c) shows typical agglomeration debris collected from both the NC and the CG AZ91D Mg alloys. Because remarkable oxygen content has been detected by EDS as shown in Fig. 9(a), it is believed that the wear process also involves oxidation in the two samples. But considering no oxygen were detected from the worn surface by EDS, as shown in Fig. 9(b), the oxidation wear may not be the dominant wear mechanism in the current case.
3.4. The effect of grain refinement

Although both the NC and the CG AZ91D Mg alloys exhibit similar wear mechanism, the present experimental results indicate that the NC surface has lower friction coefficient and smaller wear volume, which implies higher wear resistance, than the CG surface. Because no material loss caused by delamination wear and melt wear, as Chen et al. [13] reported, was found in the present work, and both the cutting and plowing abrasive wears are closely associated with the ability to resist plastic deformation of the material, it is considered that the improvement of the wear resistance of the NC surface layer is resulted from the surface hardening by grain refinement. According to the well known Hall–Petch relationship, the refinement in grain size will resulted in the strengthening or hardening of the materials, thereby in accordance with Archard’s law [15]:

\[ \frac{V}{L} = \frac{KW}{H} \]

where \( V \) is the volume of wear; \( L \) the sliding distance; \( W \) the normal load; \( H \) the hardness of the softer one in the two contacting materials (AZ91D in the present work); and \( K \) is the wear coefficient, which is valid to both the adhesive and abrasive wears, the higher the hardness, the lower the wear volume would be. As shown in Fig. 2 [12], the hardness of the NC layer generated by SMAT is two times as much as that of the CG substrate, thus, smaller wear volume was obtained on the nanocrystalline surface of the AZ91D Mg alloy. Although the oxidation wear is also involved in the wear process, which may diminish the positive effect of grain refinement on wear resistance because oxidation wear can be strengthened by the small grain size due to the steadily increase of the grain boundary fraction and the consequent increase of chemical activity, owing to the less effect of oxidation wear, surface nanocrystallization leads to an increase in wear resistance of the AZ91D Mg alloy.

4. Conclusions

(1) The friction coefficient of the NC surface layer of AZ91D Mg alloy generated by SMAT is slightly lower than that of the conventional coarse-grained alloy under dry sliding condition.

(2) The surface NC layer generated by SMAT exhibits an enhanced wear resistance under dry sliding wear condition at the applied range from 3N to 9N compared with the conventional AZ91D Mg alloy with coarse grains.

(3) Cutting, plowing abrasive wear and oxidation wear are identified in both NC and CG Mg alloys. The first two may dominate the wear process.

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