Sliding wear-induced microstructure evolution of nanocrystalline and coarse-grained AZ91D Mg alloy

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Abstract

Wear behavior of nanocrystalline (NC) AZ91D Mg alloy generated by surface mechanical attrition treatment and the corresponding coarse-grained (CG) samples subjected to dry sliding wear at different sliding velocities were investigated. The subsurface microstructure evolution of these two samples was analyzed by cross-sectional transmission electron microscope. The microstructure of NC layer was almost unchanged while grain refinement into nanometer scale was identified for CG sample at high velocity, which contributed to the wear behavior of both NC and CG samples.

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1. Introduction

It has been recognized [1,2] that under the condition of sliding with loading, large plastic strain will be developed, the corresponding subsurface structure will be changed accordingly due to local deformation. The revealing of subsurface structure evolution is quite useful in understanding the friction and wear behavior of crystalline materials. Efforts have been made on the investigation [1–6] of microstructure evolution underneath wear surfaces. Heilmann et al. investigated the development of deformation substructure and described the detailed orientation information on the worn surface of pure copper [3]. Similar experiments on copper were carried out by Hughes et al. [4] under large sliding loads as a function of sliding velocity, where differentiated near surface structures were identified at a sliding velocity of 0.25 mm/s and 25 mm/s, respectively. The experimental evidence illustrated that the sliding velocity affected the recrystallization feature of the subsurface structure.

Nanocrystalline (NC) materials [7–9] are commonly defined as materials with grain size below 100 nm. At such a grain size, the volume fraction of grain boundaries is much larger than that of the conventional coarse-grained (CG) polycrystalline counterparts. Investigations [10–12] have reported that NC materials can exhibit an enhanced wear resistance relative to CG materials under certain friction condition. Owing to the small grain size of NC materials, structure evolution at and below the sliding surface might be quite different from that of CG counterparts, which will consequently contribute to wear behavior of NC material. It would be of interest to explore the wear behavior of NC and CG materials and the microstructure evolution of these two kinds of materials subjected to sliding.

NC surface layer with an elevated hardness on AZ91D Mg alloy has been successfully generated by using surface mechanical attrition treatment (SMAT) [13]. The average grain size of the top surface layer is 30 ± 5 nm with a hardness of about 1.8 GPa. It is the objective of the present work to investigate wear behavior of an NC layer generated by SMAT and the corresponding CG AZ91D Mg alloy, by evaluating the wear rate of NC and CG samples as a function of sliding velocity under dry sliding condition. Microstructure characterization by cross-sectional transmission electron microscope (TEM) was conducted to understand the performance of NC and CG AZ91D Mg alloy.

2. Experimental

Commercial AZ91D Mg alloy with a composition of Al 8.47%, Zn 0.69%, Mn 0.14% (wt.%) was cut into 100 mm × 100 mm × 15 mm blocks. As illustrated in a previous investigation [14], SMAT was performed on SNC-II SMAT apparatus for 20 min at room temperature with a vibrating frequency of 50 Hz under vacuum after the samples were solution treated. GCr15 steel balls were used as the flying balls to strike the surface of samples. The thickness of the
Fig. 1. Wear rates of NC and CG AZ91D Mg alloy as a function of sliding velocity.

dehformed layer can be as thick as 1500 μm and that of the NC layer
is about 100 μm with an average grain size of 30 ± 5 nm [13].

Dry sliding wear experiments were performed on Optimol SRVIII
oscillating friction and wear tester with a ball-on-disc contact con-
figuration at 298 K in air with a relative humidity of 40–50%. Blocks
with a dimension of 8 mm × 8 mm × 3 mm were cut from SMAT
samples with the SMAT surface retained. The roughness is mea-
sured to be 3.7 μm. Some of these blocks were annealed at 686 K
for 24 h for later use as the CG sample for comparison, to obtain
the same surface roughness as the SMAT samples. The counter sliding
part used in wear tests was WC-Co ball with a diameter of 10 mm
and a hardness of HV: 1750. The oscillating stroke is 1–2 mm under
a normal load of 7 N, a frequency of 5–10 Hz, and duration of 30 min.
The volume wear loss was calculated from formula raised by Qu and
Truhan [15], the wear rate was determined by the volume wear loss
divided by total sliding distance.

The hardness measurements were carried out on a MVK-H300
Vickers hardness tester, with a load of 5 g and a loading time of 10 s
from the matrix to the top surface across the section. The 0.3 mm
thick slices for TEM observation were cut from the cross-sectional
regions of the worn samples of both NC and CG AZ91D Mg alloy fol-
lowed by mechanical grinding to 40–50 μm. These thin foils were
then ion milled at room temperature in a Gatan Precision Ion Pol-
ishing System (PIPS) with a small incident angle till perforation,
the TEM observations were carried out on a JEOL 2010 microscope
operated at 200 kV.

3. Results and discussion

3.1. Wear rate at different sliding velocity

The wear rate (i.e. wear volume loss per unit sliding distance)
of both NC and CG AZ91D Mg alloy with the variation of sliding
velocity at 7 N is presented in Fig. 1. It is noticed that both NC and
CG samples exhibit a reduction trend in wear rate with the incre-
ment of sliding velocity. The wear rate of the SMAT sample is almost
50% smaller than that of the CG sample when the sliding velocity
is 0.002 m/s, but the difference between them gets smaller as the
sliding velocity increases. When the sliding velocity is 0.028 m/s,
the wear rate of the two samples tends to be the same.

Considering that the microstructure evolution due to plastic
deformation from sliding will result in microhardness change in
the subsurface, microhardness detection were carried out along
the depth of the cross-section of both samples after 30 min slid-
ing, as illustrated in Fig. 2. It is presented that the hardness of the
CG alloy increases from 1.0 GPa to 1.8 GPa and that of SMAT sam-
ple remains unchanged at 1.8 GPa. It can be deduced from Fig. 2
that the thickness of the deformed layer of CG AZ91D Mg alloy is
30 μm while that of the SMAT AZ91D Mg alloy is similar to that of
CG. The hardness of the top surface layer of both SMAT sample and
CG sample after sliding are almost the same, 1.8 GPa. This value is
identical to that of the topmost surface of the as-SMAT sample, as
can be identified from the inset of Fig. 2. The microhardness varia-
tion correlates closely with the microstructure change, optical and
TEM examinations of the cross-section of NC and the CG samples
after sliding will further be explored to explain the wear behavior
of the samples.
Fig. 4. TEM BF image showing the formation of nanocrystalline at the topmost surface of NC AZ91D Mg alloy at a sliding velocity of 0.028 m/s, a load of 7 N for 30 min.

Fig. 5. TEM BF image (a) and DF image (b) showing the formation of sub-micro-grains at the worn surface of the CG AZ91D Mg alloy at a sliding velocity of 0.002 m/s, a load of 7 N for 30 min.

Fig. 6. TEM DF image (a) and SAED pattern (b) showing the formation of NC grains at the worn surface of the CG AZ91D Mg alloy at a sliding velocity of 0.028 m/s with a load of 7 N for 30 min. The corresponding histogram of grain size distribution (c) indicating the average grain size is about 40 nm. The inset of (a) is an enlarged picture at higher magnification exhibiting “clear” grain that might be resulted from DRX.
3.2. The subsurface microstructure at different sliding velocity

Fig. 3 is a typical optical micrograph of the cross-section of CG (a) and SMAT (b) AZ91D Mg alloy samples. For SMAT samples, a relatively thick deformation layer is presented, with quite a lot of deformation twins intersecting with each other while CG sample presents a quite thin deformation layer with a thickness of about 30 μm. Deformation twins were also observed within subsurface layer.

Unlike NC samples, dry sliding wear lead to significant microstructure change within the subsurface of CG samples. Fig. 5 is a typical TEM micrograph of the subsurface close to the worn surface of CG AZ91D alloy after sliding at velocity of 0.002 m/s. Subgrains with grain (or cell) size in sub-micrometer scale and few NC grains can be observed. However, for the sample sliding at 0.028 m/s, TEM micrograph taken from the top layer (Fig. 6(a)) shows a complete NC structure. The corresponding selected area diffraction pattern in Fig. 6(b) indicates the formation of randomly oriented grains. The histogram of grain size distribution measured from more than five TEM micrographs is shown in Fig. 6(c). It is illustrated that the grain size of most of the grains is a few tens of nanometers and the average grain size is about 40 nm, which is quite close to the grain size of the NC structure generated by SMAT in the same alloy [15]. Fig. 7(a) is a TEM micrograph at depth of about 10 μm below the worn surface, which shows the sub-micrometer grains (or cells). At further depth of about 20 μm below the surface (Fig. 7(b)), micro-bands, and dislocation arrays together with the intersection of deformation twins can be observed. The microstructure evolution during dry sliding wear at a velocity of 0.028 m/s exhibits a similar process to that observed in plastic strain-induced grain refinement by using SMAT. The microstructure evolution involves twinning, intersection of twinning, dislocation slip and pile-up and dynamic recrystallization (DRX).

Compared with what we have presented in a previous investigation [13] on grain refinement process during SMAT, great similarity can be evidenced between the two processes. The only difference might exist in the thickness of the deformed layer, with the one from wear is much thinner than that from SMAT, which might result from the difference in strain and strain rate in these two processes. It is quite clear that for CG AZ91D Mg alloy, grain size of the top surface worn at higher sliding velocity (0.028 m/s) can be much smaller than that at lower sliding velocity (0.002 m/s).

As stated above the grain refinement into nanometer scale subjected to sliding only occurred in the original CG AZ91D Mg alloys. For the NC structure generated by using SMAT, no further grain refinement could be identified. Previous result [13] has suggested that grain refinement into nanometer scale of Mg alloys resulted from severe plastic deformation involves the formation of dislocation arrays and cross-slips, which require dislocation multiplication. According to the Frank–Read mechanism [16], the minimum shear stress, \( \tau_{CR} \), required to activate the Frank–Read source is inversely proportional to the distance \( L \) between the two pinning points that pin down a dislocation segment: \( \tau_{CR} = \frac{Gb}{L} \), where \( G \) is the shear modulus, \( b \) is the Burgers vector. Normally, the maximum \( L \) is the grain size. If the grains are small enough, the shear stress required to activate the Frank–Read source will be extremely high [17]. Thus, dislocation multiplication becomes very difficult. It would be extremely hard to further divide the NC grains into a much smaller grain size.

It has been recognized that sliding wear could result in the formation of NC structure [18]. In addition, the present results show that the sliding wear-induced grain refinement into nanometer scale takes place at higher sliding velocity, which might be associated with the fact that different sliding rate leads to different strain rate. Rigney and co-workers [19] investigated the plastic response of a material subjected to unlubricated sliding and found that spacial extent of the deformed zone under sliding is determined by the imposed sliding velocity and other parameters. Changing the sliding velocity, there are mainly two parameters will change accordingly, strain rate and local temperature. With the increasing of sliding velocity, the strain rate will increase [19], as has been indicated by Wang et al. [20], strain rate plays a critical role in plastic strain-induced grain refinement process, so the higher strain rate at sliding speed of 0.028 m/s will contribute much to the much smaller grains in CG AZ91D. Further, as Ashby and co-worker [21] reported that under the same applied load, the local temperature depends directly on the relative velocity of the sliding profile. In addition, previous result [13] has also suggested that severe plastic deformation-induced nanocrystallization in Mg alloys involves
DRX. It is not difficult to understand that at higher sliding speed, the local temperature rise from frictional heat will be higher than that from the lower velocity, which may contribute to the formation of NC grains by DRX. Higher local temperature resulted from higher sliding velocity could promote the DRX process. Hence, higher strain rate and temperature at higher sliding velocity might be responsible for the grain refinement into nanometer scale in Mg alloys during dry sliding wear.

4. Conclusions

SMAT AZ91D Mg alloy with an NC layer exhibits a lower wear rate than the CG sample at sliding velocities below 0.028 m/s but a similar wear rate at velocity of 0.028 m/s. TEM observations of the subsurface structure evolution revealed that at velocity of 0.028 m/s, the NC layer remains almost unchanged during sliding while the grain size of the subsurface of the CG sample get into nanometer scale due to the plastic deformation and the increment of local temperature developed during sliding.

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