



# Improvement of wear and cavitation–erosion by ALD-deposited $\text{LiAl}_x\text{O}_y$ films on an Mg–10Li–0.5Zn alloy

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## ABSTRACT

$\text{LiAl}_x\text{O}_y$  films with thicknesses of 65–200 nm were deposited by the ALD technique on an Mg–10Li–0.5Zn alloy. The ALD-deposited  $\text{LiAl}_x\text{O}_y$  films have an amorphous structure and exhibit properties of low friction coefficient, high hardness/elastic ( $H/E$ ) ratio, strong adhesion, smooth surface roughness and conformable coverage. These excellent properties of ALD-deposited  $\text{LiAl}_x\text{O}_y$  films can significantly improve the wear performance and cavitation–erosion resistance of Mg–10Li–0.5Zn alloy.

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## 1. Introduction

Magnesium alloys are considered as potential candidates for numerous applications, especially in transportation vehicles or lightweight enclosures for 3C (computer, communication and consumer electronic) products owing to their excellent properties, such as low density, high specific strength, high damping capacity and high recycling ability [1–3]. It is known that the Mg–Li alloys with Li content of 5–11 mass% will exhibit a dual-phase structure of  $\alpha$  (HCP) and  $\beta$  (BCC) phases. These dual-phase Mg–Li alloys have excellent formability, as well as their extra-low density [4–8]. Some recent articles reported that the mechanical properties of Mg–Li alloys could be improved effectively by cold working, addition of alloying elements and precipitation hardening [9–14]. However, the Mg alloys still have disadvantages of poor corrosion and wear resistance [15–18]. These poor properties limit the application of Mg–Li alloys and need to be resolved effectively. In a previous paper [15], we introduced a special technique of atomic-layer deposition (ALD) to successfully deposit  $\text{LiAl}_x\text{O}_y$  films on Mg–Li alloys. The corrosion-resistance of Mg–Li alloys could be significantly improved due to the dense and pinhole-free structure as well as the excellent coverage and conformity of the ALD-deposited  $\text{LiAl}_x\text{O}_y$  films. In the present work, we continue to study the effects of these ALD-deposited  $\text{LiAl}_x\text{O}_y$  films on the wear and cavitation erosion of Mg–Li alloys.

## 2. Experimental procedure

Melting of Mg–Li alloys (LZ101 is a representative alloy in this study) was processed in a high frequency electric induction furnace with vacuum capability and inert argon gas. The as-cast LZ101 alloy was analyzed with ICP-AES (Induction Coupled Plasma-Atomic Emission Spectrometry) apparatus, and its chemical compositions are shown in Table 1. The casting rod with a diameter of 200 mm was hot-extruded to plates with 10 mm thickness. These extruded plates were then hot rolled to 3 mm thickness. Specimens for various testing were cut from these hot rolled plates, and then mechanically polished with SiC abrasive paper down to P1200 grit. Afterwards, the  $\text{LiAl}_x\text{O}_y$  films with various thicknesses were deposited on the LZ101 specimens using the ALD technique.  $\text{Al}(\text{CH}_3)_3$  (trimethylaluminum, TMA) and  $\text{H}_2\text{O}$  (water vapor) were the precursors for aluminum and oxygen, respectively [19–22]. Deposition was conducted in a flow reactor at a pressure about 0.1 Torr and a low temperature of 200 °C, with  $\text{N}_2$  as a carrier gas. The ALD process consisted of a number of identical cycles, each of which contained the following sequence:  $\text{H}_2\text{O}$ , 0.1 s  $\rightarrow$   $\text{N}_2$  purge, 5 s  $\rightarrow$  TMA, 0.1 s  $\rightarrow$   $\text{N}_2$  purge, 5 s, and deposited  $\sim 1.3$  Å of  $\text{LiAl}_x\text{O}_y$ . The thicknesses of  $\text{LiAl}_x\text{O}_y$  films were precisely controlled by the number of ALD cycles. As reported in a previous paper [15], the ALD-deposited films on LZ101 substrate were  $\text{LiAl}_x\text{O}_y$  films, instead of  $\text{Al}_2\text{O}_3$  films. This phenomenon is ascribed to that the Li atoms within the Mg–Li substrate will diffuse out easily to react with Al and/or O atoms during the ALD cycles.

Scanning electron microscope (SEM) was employed for observation of the film's surface morphologies. XRD tests were used to identify the microstructure of the deposited films. XRD tests were

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**Table 1**

The ICP-AES measured chemical compositions of the Mg–Li alloy used in this study. (wt.%).

| Alloy | Mg   | Li   | Zn   | Mn   | Si   | Al    |
|-------|------|------|------|------|------|-------|
| LZ101 | Bal. | 9.94 | 0.46 | 0.26 | 0.04 | <0.01 |

carried out on a Rigaku glancing incident angle diffraction using Cu  $K_{\alpha}$  radiation. TEM observations were conducted with a Philips Tecnai-F20 G2 electron microscope operating at 200 kV, including the composition analyses by energy dispersive spectroscopy (EDS) using an electron probe of 10 nm. To clearly examine the cross section of  $\text{LiAl}_x\text{O}_y$  films, the film's surfaces were first coated with Pt and C. Two specimens, with ALD-deposited surfaces facing each other, were glued together. The mounted slice of cross section was ground, dimple polished, and then ion milled using argon ion beam (Gatan 691 Precision Ion Polishing System). The nano-hardness and young's modulus of  $\text{LiAl}_x\text{O}_y$  films were measured by an MTS Nano-Indenter. The wear index of thin films, according to the ASTM D4060-07 method [23], were measured and automatically calculated by using a wear tester, with a rotation speed of 40 rpm at a load of 50 g. The wear index ( $I$ ) of a test specimen was calculated as follows:

$$I = \frac{(A-B) \times 1000}{C} \quad (1)$$

Where

- A weight of test specimen before abrasion, mg,  
 B weight of test specimen after abrasion, mg, and  
 C number of cycles of abrasion recorded.

The abrasion-wear tests were performed on a ball-on-disc tribometer with a constant sliding speed of 40 rpm and a wear load of 1 N in the ambient atmosphere. The cross-cut adhesion test was performed using an ASTM D3359-95 method [24]. A surface roughness analyzer (Mitutoyo Serftest SV-400, made in Japan) was applied to measure the specimen's surface roughness ( $R_a$  value). For each specimen, the average  $R_a$  value was calculated from at least five test readings.

The cavitation-erosion testing was carried out according to ASTM G32-92 [25] in distilled water. Cavitation was induced by longitudinal oscillation at 20 kHz with amplitude of 100  $\mu\text{m}$ . The testing temperature of the medium was 25 °C. The weight loss after erosion was measured by using a precise electronic balance, with weighing accuracy of  $\pm 0.01$  mg.

### 3. Results and discussion

#### 3.1. Microstructure of the LZ101 specimens with ALD-deposited $\text{LiAl}_x\text{O}_y$ films

Fig. 1(a) and (b) show the scanning electron microscopy (SEM) micrographs of the LZ101 alloy without and with an ALD-deposited  $\text{LiAl}_x\text{O}_y$  film of 200 nm, respectively. As shown in Fig. 1(a), the as-ground specimen exhibits many obvious defects, such as scrubbed traces, voids, etc. However, after the deposition of the  $\text{LiAl}_x\text{O}_y$  film, the whole surface of the specimen has been conformably covered, as shown in Fig. 1(b). Namely, even though the bare LZ101 alloys have many serious surface defects, they can be smoothly renovated and entirely protected by the ALD-deposited  $\text{LiAl}_x\text{O}_y$  films. This result indicates that the ALD technique is an effective method to deposit protective films on the Mg–Li alloys. Meanwhile, it is worthy to mention that the thicker  $\text{LiAl}_x\text{O}_y$  films will exhibit smoother surface morphology. Fig. 2 shows the XRD patterns of the LZ101 specimens without and with ALD-deposited  $\text{LiAl}_x\text{O}_y$  films. All the diffraction

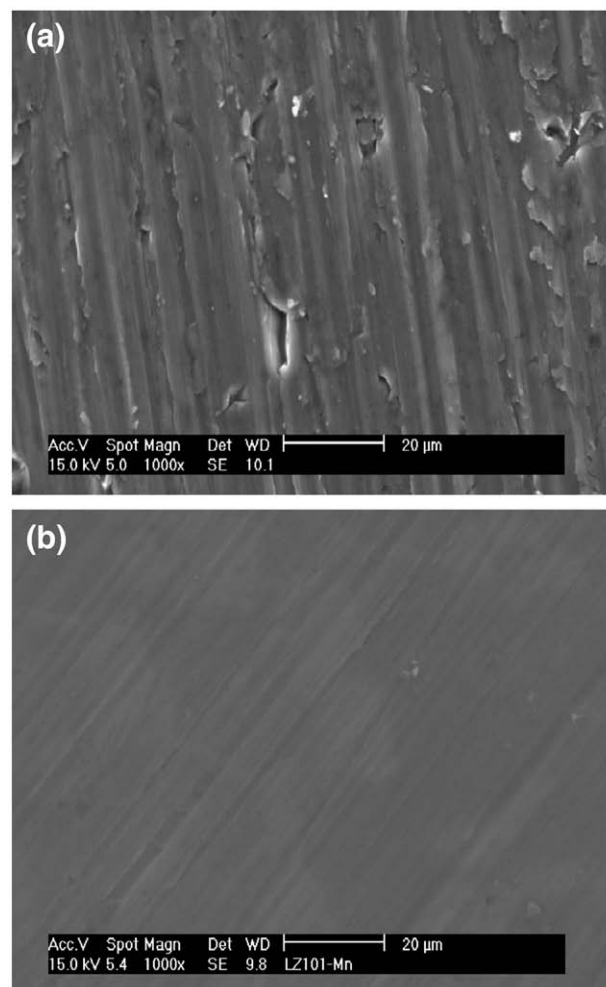


Fig. 1. Surface morphologies of (a) LZ101 substrate and (b) with ALD-deposited  $\text{LiAl}_x\text{O}_y$  film with 200 nm thickness.

spectra in Fig. 2 exhibit a typical dual-phase structure of  $\alpha$  (HCP) and  $\beta$  (BCC) phases of LZ101 alloys. This feature is reasonable because the  $\text{LiAl}_x\text{O}_y$  films are amorphous. The XRD peaks are mainly from the diffraction of LZ101 substrates.

Fig. 3(a) shows the cross-sectional TEM micrograph of the LZ101 specimen with an ALD-deposited  $\text{LiAl}_x\text{O}_y$  film, about 200 nm in

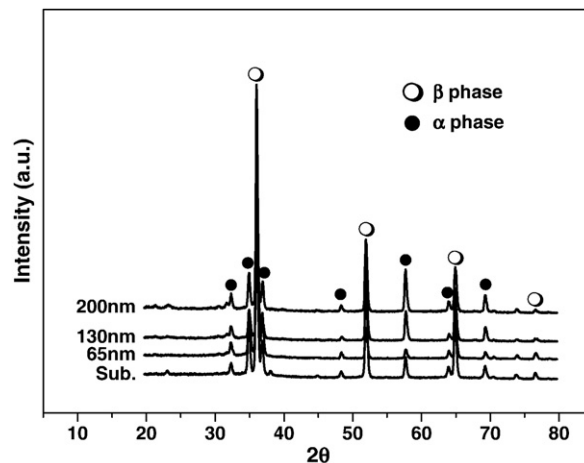


Fig. 2. XRD patterns of the LZ101 specimens without and with ALD-deposited  $\text{LiAl}_x\text{O}_y$  films.

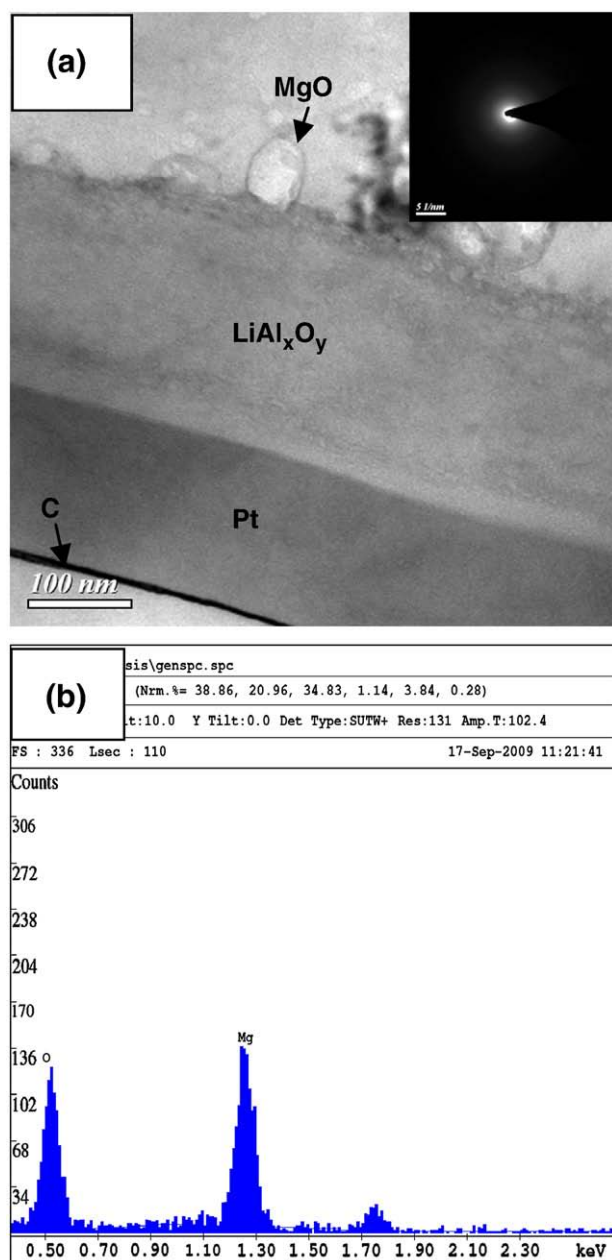


**Fig. 3.** (a) TEM observation of the LZ101 specimen with an ALD-deposited  $\text{LiAl}_x\text{O}_y$  film. The diffraction pattern indicates that the  $\text{LiAl}_x\text{O}_y$  layer has an amorphous structure. (b) TEM-EDS analysis showing the particles around the surface of LZ101 substrate are MgO compound.

thickness. The diffraction pattern in Fig. 3(a) also indicates that the  $\text{LiAl}_x\text{O}_y$  layer has an amorphous structure. In Fig. 3(a), some particles can be clearly observed around the surface of LZ101 substrate. The chemical compositions of these particles are semi-quantitatively measured by using TEM-EDS and the results are presented in Fig. 3(b). Fig. 3(b) reveals that the particle contains Mg and O, indicating that these particles are MgO compound. The formation of MgO particles is attributed to the specimen's easy oxidation before and/or during ALD-deposition process.

### 3.2. Wear behavior of the LZ101 specimens with ALD-deposited $\text{LiAl}_x\text{O}_y$ films

It is known that the wear performance of protective coatings mainly depends on the combined effects of nano-hardness, Young's

**Table 2**

Nano-hardness, Young's modulus and  $H/E$  ratio of the ALD-deposited  $\text{LiAl}_x\text{O}_y$  film,  $\text{LiAlO}_2$  [27,28] and  $\text{Al}_2\text{O}_3$  [29] ceramic.

|                              | ALD-deposited $\text{LiAl}_x\text{O}_y$ film | $\text{LiAlO}_2$ | $\text{Al}_2\text{O}_3$ |
|------------------------------|----------------------------------------------|------------------|-------------------------|
| Nano-hardness ( $H$ , GPa)   | 15                                           | 8                | 8–20                    |
| Young's modulus ( $E$ , GPa) | 210                                          | 103–105          | 260–410                 |
| $H/E$                        | 0.07                                         | 0.08             | 0.03–0.05               |

modulus, adhesion and friction coefficient. The nano-hardness ( $H$ ), Young's modulus ( $E$ ) and  $H/E$  ratio of ALD-deposited  $\text{LiAl}_x\text{O}_y$  films are presented in Table 2. These data were obtained by using the nano-indenter with the indentation depth at approximately 20 nm under an applied load of 1 mN. The indentation depth was controlled at about 1/10 of film thickness to avoid the substrate effect [26]. For each specimen, the average values of nano-hardness and Young's modulus were calculated from at least five test readings. The higher hardness implies less plastic yielding at a given contact pressure, and the lower modulus permits the applied load to be distributed over a wider area thus reducing the maximum contact pressure at a given load. As presented in Table 2, the ALD-deposited  $\text{LiAl}_x\text{O}_y$  film exhibits a high  $H/E$  ratio, which is similar to that of bulk  $\text{LiAlO}_2$  [27,28] but is much higher than that of bulk  $\text{Al}_2\text{O}_3$  ceramics [29]. This high  $H/E$  ratio of the ALD-deposited  $\text{LiAl}_x\text{O}_y$  film is expected to have significant improvement on the wear resistance of the LZ101 substrates. Moreover, the ALD-deposited  $\text{LiAl}_x\text{O}_y$  films exhibit an excellent adhesion with the LZ101 substrates. Their cross-cut adhesion strength can reach 5B degree, which indicates that the edges of cross-cuts are still completely smooth and none of the squares of cutting lattice is detached after the adhesion test [24]. This excellent adhesion will also improve the wear performance of LZ101 specimens with deposition of  $\text{LiAl}_x\text{O}_y$  films.

Fig. 4 shows the wear index of the LZ101 specimens without and with ALD-deposited  $\text{LiAl}_x\text{O}_y$  films. As shown in Fig. 4, the LZ101 specimens with ALD-deposited  $\text{LiAl}_x\text{O}_y$  films have lower wear index than the bare one. The thicker the  $\text{LiAl}_x\text{O}_y$  films, the lower the wear index is. Fig. 5 shows the friction coefficient at various travel distances of the LZ101 specimens without and with an ALD-deposited  $\text{LiAl}_x\text{O}_y$  film of 200 nm. In Fig. 5, the specimen with ALD-deposited  $\text{LiAl}_x\text{O}_y$  film has lower friction coefficient than the LZ101 substrate, say  $\sim 0.5$  and  $\sim 0.7$ , respectively. All these results indicate that the ALD-deposited  $\text{LiAl}_x\text{O}_y$  films can indeed improve the wear performance of LZ101 specimens. The significant improvement of wear performance

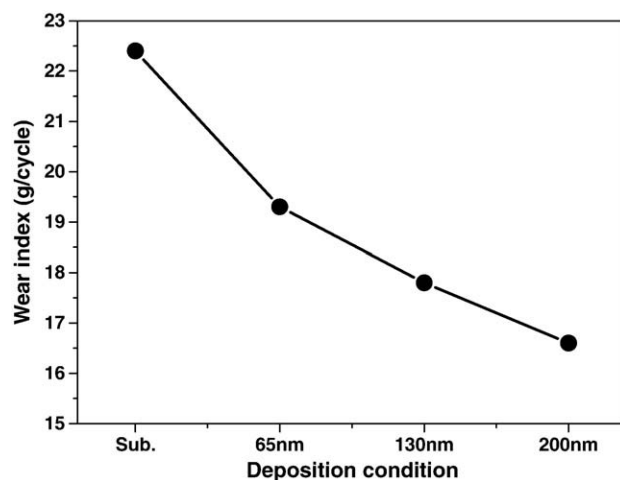


**Fig. 4.** The wear index of the LZ101 specimens without and with ALD-deposited  $\text{LiAl}_x\text{O}_y$  films.

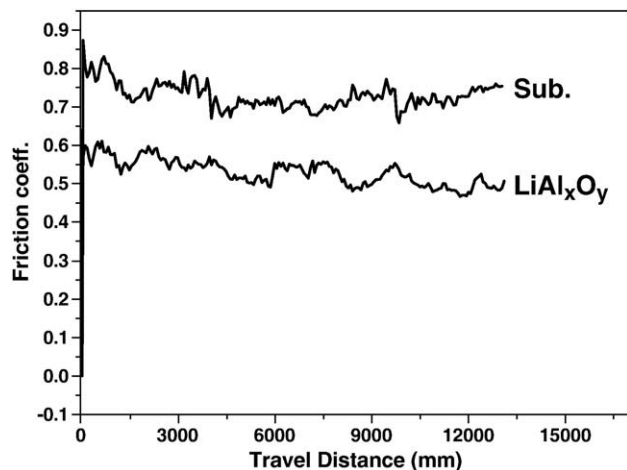


Fig. 5. Friction coefficient at various travel distances of the LZ101 specimens without and with an ALD-deposited LiAl<sub>x</sub>O<sub>y</sub> film of 200 nm.

by ALD-deposited LiAl<sub>x</sub>O<sub>y</sub> films is attributed to their excellent film's properties, such as the high  $H/E$  ratio, strong adhesion and conformable coverage. These results coincide well with the concepts described by Leyand and Matthews [30,31], who noted that a high ratio of  $H/E$  and excellent film's quality could be demonstrative of good wear performance.

### 3.3. Cavitation erosion of the LZ101 specimens with ALD-deposited LiAl<sub>x</sub>O<sub>y</sub> films

It is known that the cavitation erosion is a common cause of failure in machinery such as feed water pumps, ship propellers and hydraulic turbines [32]. The sudden pressure change within the liquid leads to the formation of bubble cavities and their subsequent collapse generates high pressure shock waves and/or micro-jets, which cause localized erosion of the material. It is valuable to understand the effects of ALD-deposited LiAl<sub>x</sub>O<sub>y</sub> films on the erosion resistance of Mg–Li alloys for extending their applications. Fig. 6(a) and (b) shows the cumulative weight loss and erosion rate vs. erosion time, respectively, for the LZ101 specimens without and with ALD-deposited LiAl<sub>x</sub>O<sub>y</sub> films. As shown in Fig. 6(a) and (b), the LZ101 specimens with ALD-deposited LiAl<sub>x</sub>O<sub>y</sub> films have lower cumulative weight loss and erosion rate than the bare specimen. The thicker the LiAl<sub>x</sub>O<sub>y</sub> films, the lower the cumulative weight loss and erosion rate are. This indicates that the ALD-deposited LiAl<sub>x</sub>O<sub>y</sub> films on LZ101 specimens are more resistant to cavitation erosion than the LZ101 substrate. The improvement of cavitation erosion by ALD-deposited LiAl<sub>x</sub>O<sub>y</sub> films is attributed to their excellent film's properties, namely, the high  $H/E$  ratio, strong adhesion and conformable coverage. It is worthy to mention that the specimens' surface roughness ( $R_a$ ) can also affect the resistance to cavitation erosion. For rougher surface, more bubble cavities are nucleated easily and their collapse generates severer erosion damage. In the present study, the ALD-deposited LiAl<sub>x</sub>O<sub>y</sub> films exhibit a smoother surface than the bare one. Their  $R_a$  values are about 15  $\mu\text{m}$ , 10  $\mu\text{m}$ , 7  $\mu\text{m}$ , 4  $\mu\text{m}$  for the bare specimen and with film's thickness of 65 nm, 130 nm, 200 nm, respectively. This phenomenon indicates that the ALD process can repair the surface defects, such as scrubbed traces, voids, etc. and reduce the specimen's surface roughness. The reduction of surface roughness will improve the cavitation erosion of the LZ101 specimens with ALD-deposited LiAl<sub>x</sub>O<sub>y</sub> films.

Fig. 7(a–d) and (e–h) presents the eroded surface morphologies of the LZ101 specimens without and with ALD-deposited LiAl<sub>x</sub>O<sub>y</sub> films of 200 nm, respectively, after various erosion periods of 0, 5, 35, and

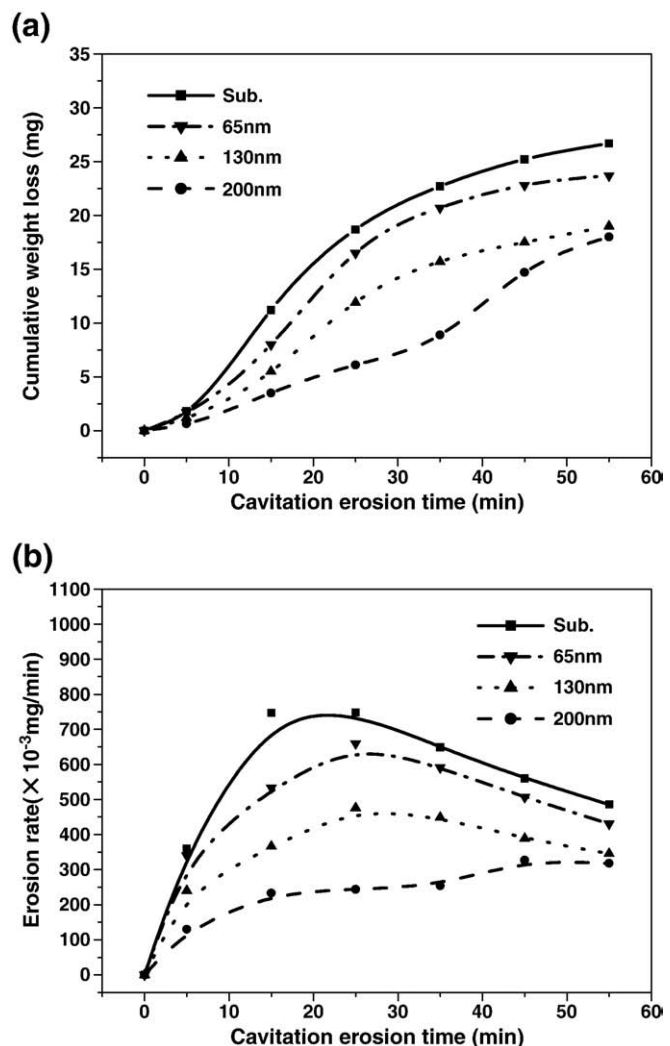


Fig. 6. (a) The cumulative weight loss and (b) erosion rate vs. erosion time for the LZ101 specimens without and with ALD-deposited LiAl<sub>x</sub>O<sub>y</sub> films.

55 min. As shown in Fig. 7(a), the as-ground specimen exhibits many obvious defects, such as scrubbed traces, voids, etc. However, after the deposition of LiAl<sub>x</sub>O<sub>y</sub> film, the whole specimen surface has been conformably covered, as shown in Fig. 7(e). In Fig. 7 (b–d) for the LZ101 bare specimen, an obvious damage trace has been observed after a short period of cavitation erosion and it increases rather rapidly during further cavitation erosion. This feature indicates that the LZ101 bare specimen has a poor resistance to cavitation erosion. For the specimen with ALD-deposited LiAl<sub>x</sub>O<sub>y</sub> film, the erosion damage is not obvious even after a long erosion period of 35 min, as shown in Fig. 7 (f–g). These results are consistent with those shown in Fig. 6(a) and (b). Namely, the deposition of LiAl<sub>x</sub>O<sub>y</sub> films by ALD technique can effectively improve the erosion resistance of LZ101 alloy.

## 4. Conclusions

The improvement of wear and cavitation–erosion by ALD-deposited LiAl<sub>x</sub>O<sub>y</sub> films on LZ101 alloy has been studied. The important conclusions are as follows:

- (1) The LiAl<sub>x</sub>O<sub>y</sub> films with thickness of 65–200 nm have been successfully deposited on LZ101 alloy. The LiAl<sub>x</sub>O<sub>y</sub> films have an amorphous structure. Some particles of MgO compound are observed around the surface of LZ101 substrate.

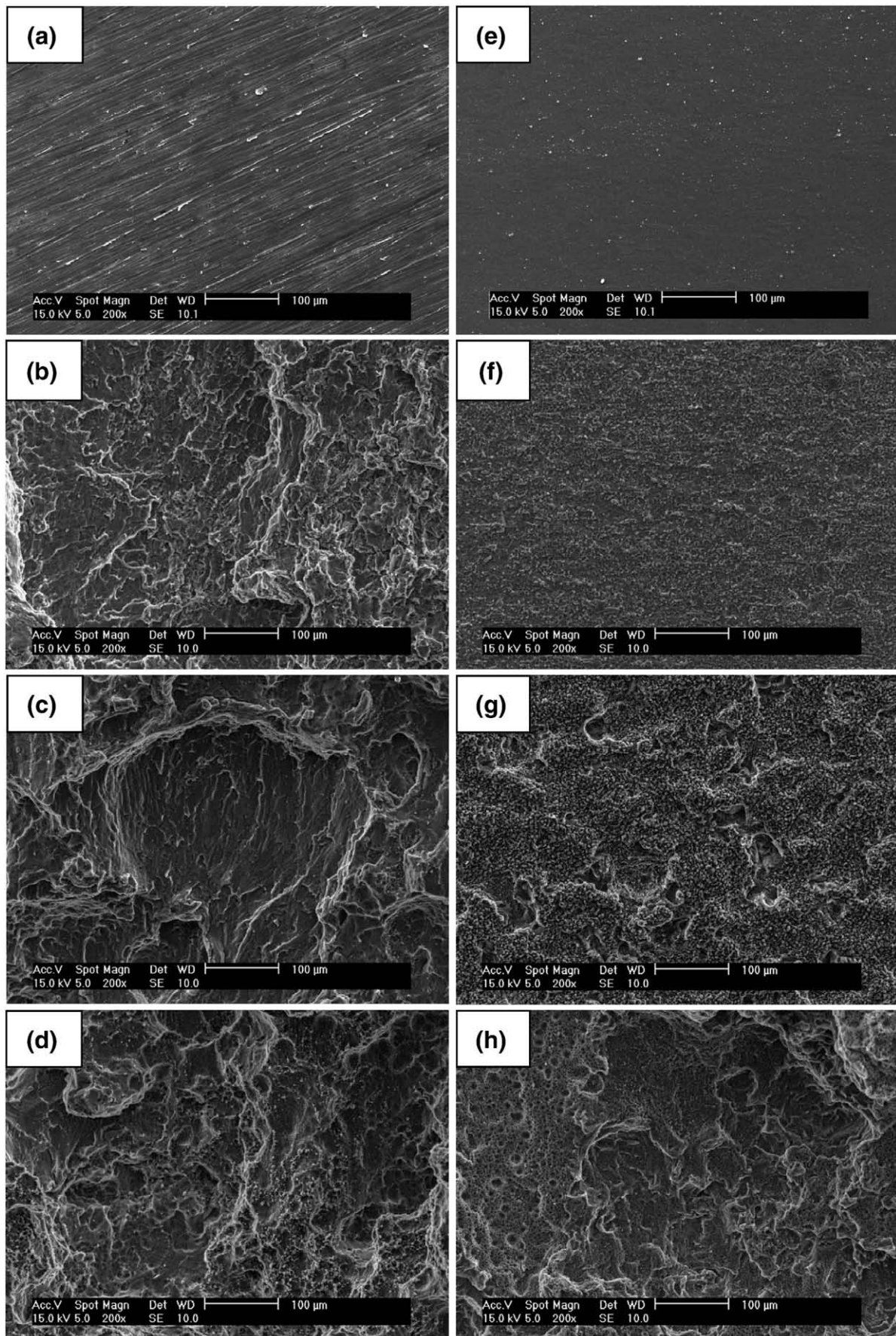


**Fig. 7.** The eroded surface morphologies of the LZ101 specimens after various erosion periods of 0, 5, 35, and 55 min. (a–d) bare specimen, (e–h) specimen with an ALD-deposited  $\text{LiAl}_x\text{O}_y$  film of 200 nm.

- (2) The ALD-deposited  $\text{LiAl}_x\text{O}_y$  films can significantly improve the wear performance of LZ101 specimens. The improvement of wear performance is attributed to the excellent film's properties, such as the low friction coefficient, high  $H/E$  ratio, strong adhesion and conformable coverage.
- (3) The LZ101 bare specimen has a poor resistance to cavitation erosion. The deposition of  $\text{LiAl}_x\text{O}_y$  films by ALD technique can effectively improve the erosion resistance of LZ101 alloy. The high  $H/E$  ratio, strong adhesion and smooth surface roughness of ALD-deposited  $\text{LiAl}_x\text{O}_y$  films contribute to this improvement of erosion resistance.

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## References

- [1] F. Zarandi, G. Seale, R. Verma, E. Essadiqi, S. Yue, *Mater. Sci. Eng. A* 496 (2008) 159.
- [2] Y. Kojima, *Mater. Sci. Forum* 350 (2000) 3.
- [3] B.L. Mordike, T. Ebert, *Mater. Sci. Eng. A* 302 (2001) 37.
- [4] P. Metenier, G. Gonzalez-Doncel, *Mater. Sci. Eng. A* 125 (1990) 195.
- [5] Y.W. Kim, D.H. Kim, H.I. Lee, C.P. Hong, *Scripta Mater.* 38 (1998) 923.
- [6] G.S. Song, M.V. Kral, *Mater. Charact.* 54 (2005) 279.
- [7] H. Haferkamp, R. Boehm, U. Holzkamp, C. Jaschik, V. Kaese, M. Niemeyer, *Mater. Trans.* 42 (2001) 1160.
- [8] K. Matuzawa, T. Koshihara, Y. Kojima, *J. Jpn. Inst. Light Met.* 39 (1989) 45.
- [9] J.Y. Wang, W.P. Hong, P.C. Hsu, C.C. Hsu, S. Lee, *Mater. Sci. Forum* 419–422 (2003) 165.
- [10] A. Yamamoto, T. Ashida, Y. Kouta, K.B. Kim, S. Fukumoto, H. Tsubakino, *Mater. Trans.* 44 (2003) 619.
- [11] C.H. Chiu, H.Y. Wu, J.Y. Wang, S. Lee, *J. Alloys Compd.* 460 (2008) 246.
- [12] H. Takuda, S. Kikuchi, T. Tsukada, K. Kubota, N. Hatta, *J. Mater. Sci.* 37 (2002) 51.
- [13] C.C. Hsu, J.Y. Wang, S. Lee, *Mater. Trans.* 49 (2008) 2728.
- [14] P.C. Wang, H.C. Lin, K.M. Lin, M.T. Yeh, C.Y. Lin, *Mater. Trans.* 50 (2009) 2259.
- [15] P.C. Wang, Y.T. Shih, H.C. Lin, M.J. Chen, K.M. Lin, *Proceeding of International Thin Films Conference*, 2009, Taipei, D027.
- [16] H. Altun, S. Sen, *Mater. Charact.* 58 (2007) 917.
- [17] H. Hoche, H. Scheerer, D. Probst, E. Broszeit, C. Berger, *Surf. Coat. Technol.* 174–175 (2003) 1018.
- [18] H. Hoche, C. Blawert, E. Broszeit, C. Berger, *Surf. Coat. Technol.* 193 (2005) 223.
- [19] S.M. George, A.W. Ott, J.W. Klaus, *J. Phys. Chem.* 100 (1996) 13121.
- [20] G.S. Higashi, C.G. Fleming, *Appl. Phys. Lett.* 55 (1989) 1963.
- [21] A.C. Dillon, A.W. Ott, J.D. Way, S.M. George, *Surf. Sci.* 322 (1995) 230.
- [22] A.W. Ott, J.W. Klaus, J.M. Johnson, S.M. George, *Thin Solid Films* 292 (1997) 135.
- [23] ASTM D4060-07, *Standard Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser*, ASTM, Philadelphia, PA, 2007.
- [24] ASTM D3359-97, *Standard Test Method for Measuring Adhesion by Tape Test*, ASTM, Philadelphia, PA, 2003.
- [25] ASTM Committee, *ASTM Standard G32-92, Standard Test Method for Cavitation Erosion Using Vibratory Apparatus*, ASTM, West Conshohocken, PA, USA, 1992.
- [26] R. Saha, W.D. Nix, *Acta Mater.* 50 (2002) 23.
- [27] Hadis Morkoç, *Handbook of Nitride Semiconductors and Devices*, Materials Properties, Physics and Growth, vol. 1, 2008, p. 358.
- [28] S.Q. Wu, Z.F. Hou, Z.Z. Zhu, *Comput. Mater. Sci.* 46 (2009) 221.
- [29] P. Auerkari, *Mechanical and physical properties of engineering alumina ceramics*, Espoo 1996, Technical Research Centre of Finland, VTT Tiedotteita – Meddelanden – research Notes 1792. 26pp.
- [30] Leyland, A. Matthews, *Surf. Coat. Technol.* 177–178 (2004) 317.
- [31] A. Leyland, A. Matthews, *Wear* 246 (2000) 1.
- [32] B.S. Mann, V. Arya, *Wear* 253 (2002) 650.