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Fatigue property improvements of ZK60 magnesium alloy: Effects of thin film metallic glass

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ABSTRACT

The application of thin film metallic glass (TFMG) has been shown to improve the fatigue characteristics of ZK60 magnesium alloys. This paper reports on the deposition of $Zr_{51}Cu_{32}Al_{10}Ni_7$ (*Z*-TFMG) and $W_{70}Ni_{20}B_{10}$ (W-TFMG) on ZK60 Mg alloy to a thickness of 200 nm and its effect on fatigue characteristics. In four-point-bending fatigue studies at room temperature, the fatigue limits of the bare, W-, and Z-TFMG-coated Mg alloys were 225, 270, and 280 MPa, respectively. These values indicate that the TFMG coatings improved fatigue resistance by as much as 24% through 10^7 cycles. The hardness of TFMGs, their excellent adhesion to the substrate, and the resulting reduction in surface roughness are believed to account for the enhanced fatigue characteristics.

1. Introduction

The low density, high specific strength, and machinability of wrought magnesium (Mg) alloys has led to its widespread use in the fabrication of lightweight structural components in the automotive and aircraft industries [1–5]. However, the fatigue characteristics of these alloys remains a notable shortcoming, which has prompted the development of surface treatment procedures, such as shot peening and the application of hard protective coatings, to inhibit or delay the formation of fatigue cracks initiated at the surface [6,7].

A new class of protective coatings, referred to as thin film metallic glasses (TFMGs), have been shown to improve the fatigue characteristics of metal alloys. These amorphous materials (lacking grain boundaries) feature high strength and good corrosion resistance [8]. TFMGs have been used to improve the fatigue performance of 316L stainless steel [9,10], Ti-6Al-4V [11], 7075-T6 Al [12,13], and Ni-based alloys [14]. Among the numerous TFMG systems that have been developed, our previous works [8–11] familiarized us with the good ductility and excellent glass-forming ability of Zr-based TFMG. Furthermore, this system has been widely shown to improve the fatigue performance of 316L stainless steel and Ti-6Al-4V. Moreover, Madge et al. [15] reported that W-based metallic glass possesses hardness values approaching the level of those seen in tribological materials such as TiN. Thus, in this study, we sputter deposited Zr-based TFMG ($Zr_{51}Cu_{32}Al_{10}Ni_7$, *Z*-TFMG) and W-based TFMG ($W_{70}Ni_{20}B_{10}$, W-TFMG) on the surface of extruded ZK60 Mg alloy. We then evaluated the effects of the coatings in four-point-bending-fatigue tests under ambient conditions.

2. Experimental details

As-extruded commercial ZK60 magnesium alloy (a nominal composition of Mg–6Zn–0.5Zr, wt.%) was cut into specimens (30 mm × 3 mm × 3 mm) and mechanically polished using a series of abrasive papers up to 4000-grade. Quaternary Zr-based ($Zr_{55}Cu_{30}Al_{10}Ni_5$) and ternary W-based ($W_{50}Ni_{25}B_{25}$) targets were used to deposit *Z*-TFMG ($Zr_{51}Cu_{32}Al_{10}Ni_7$) and W-TFMG ($W_{70}Ni_{20}B_{10}$) on Mg alloy substrates. Both of these film compositions were studied using electron probe X-ray microanalysis (EPMA, JXA-8800 M). Prior to the deposition of the TFMG, the Mg alloy was sputter etched using argon ions to remove the native surface oxide layer. RF magnetron sputtering at a base pressure of 1.6×10^{-4} Pa and working pressure of 0.4 Pa at a sputtering power of 100 W was used to deposit Z-TFMG and W-TFMG to a thickness of 200 nm. Substrate bias of −100 V was introduced during the deposition of the first 20 nm (thickness), followed by −50 V for the remaining 180 nm in order to promote adhesion of the film. We also sputter-deposited a layer of nickel to a thickness of approximately 10-nm using the method described above in order to improve the adhesion of the W-TFMG to the substrate.

Fatigue testing was performed using a MTS370 hydraulic system with samples loaded under various stress levels at the R value (P_{min}/P_{max}, where P_{min} and P_{max} are the applied minimum and maximum loads, respectively) of 0.1 in load-control mode using a 10 Hz sinusoidal waveform. The length of the support and loading spans were 20 mm

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and 10 mm, respectively. The bottom surface of the samples, which was subjected to continuous tension during fatigue testing, was coated with Z-TFMG or W-TFMG/Ni. In accordance with ASTM (American Society for Testing and Materials) D6272-10 standards, Eq. (1) was used to calculate the four-point bending stress, as follows:

$$\sigma = 3P\,(l - d)/2wt^2 \tag{1}$$

where P is the applied load, l and d are the support and loading span lengths, and w and t are the width and thickness of the specimens, respectively.

Crystallographic analysis was performed using an X-ray diffractometer (XRD, Bruker D8 Discover SSS) with Cu Kα radiation (λ = 1.5406 Å), accelerating voltage of 40 kV, and emission current of 200 mA. The sample was scanned continuously between 20° and 80° (2θ) at a glancing angle of 0.5° with a step size of 0.05° and step time of 1 s. Thermal characteristics were obtained using a differential scanning calorimeter (DSC, Netzsch 404 F3) at a heating rate of 40 K/min under Ar to verify the amorphous characteristics, such as the glass-transition (*Tg*) and crystallization (*Tx*) temperatures. We examined the surface roughness of bare and coated substrates using an atomic force microscope (AFM, Bruker Icon) in contact-mode with a scanning area of 30 μm × 30 μm. A nanoindenter (Hysitrons TI 950 TriboIndenter) was used with a Berkovich 142.3° diamond probe to measure the hardness of the 500-nm-thick coatings under a maximum applied load of 1.2 mN on Si substrate. To avoid the substrate effect, the nanoindentation depth was <50 nm, which was within one-tenth of the film thickness. A scratch test to evaluate the adhesion of the W-TFMG and Z-TFMG coatings was performed using a scratch tester (J & L Tech. Co.) with a 200 μm-radius diamond tip. The initial load was 0.1 N and the final load was 25 N at a speed of 0.01 mm/s. A dual-beam focused ion beam (FIB, FEI Quanta 3D FEG)-equipped scanning-electron-microscope (SEM) was used at an accelerating voltage of 20 kV for fractographic analysis and in the preparation of samples for the transmission electron microscopy (TEM, FEI Tecnai G2) observation, operated at 200 KeV.

3. Results and discussion

3.1. Characterizations of substrate and TFMGs

Fig. 1(a) presents XRD diffraction patterns obtained from the ZK60 substrate and TFMGs. The XRD Bragg peaks of the substrate in this figure are indicative of a solid solution of Mg (JCPD#35-0821) as the major phase with minor phases of $MgZn_2$ (JCPD#34-0457). W- and Z-TFMGs both present broad humps at approximately 30°–50° (2θ), which is typical of the amorphous structure of TFMGs. The DSC measurements in Fig. 1(b) reveal an exothermic peak of a crystalline phase indicating that the TFMGs underwent glass transition. The *Tg* and *Tx* values of W-TFMG were 947.8 and 1019.5 °C, respectively. The *Tg* and *Tx* values of Z-TFMG were 547 and 590.5 °C, respectively. Thus, the XRD and DSC results both confirm the amorphous nature of the TFMGs in this study.

3.2. Mechanical properties

In four-point-bending tests, the substrate was shown to have a yield strength of 393.7 MPa. Thus, bare and coated samples were subsequently tested at a maximum alternating stress of 320 MPa, corresponding to ~80% of the yield strength, the results of which are indicated by the S-N curve [maximum alternating stress amplitude (S) versus number of cycles (N) to failure] in Fig. 2. The arrows indicate where run out was performed up to 10^7 cycles without failure. These results clearly demonstrate that the TFMG-coated samples outperformed the bare samples in resistance to fatigue. The fatigue-endurance limits were as follows: bare substrate (225 MPa), coated with W-TFMG (270 MPa) and coated with Z-TFMG (280 MPa). This shows that the W-TFMG coating improved fatigue resistance by 20%, whereas Z-TFMG improved resistance by 24%. Under stress of 280 MPa, the fatigue of the substrates life (in cycles) were as follows: bare substrate (3.9×10^4), W-TFMG (1.6×10^5), and Z-TFMG ($>1 \times 10^7$). Thus, the W-TFMG coating extended the fatigue life by ~4 times, whereas Z-TFMG extended the fatigue life by >250 times. Under stress of 320 MPa (~80% of the yield stress

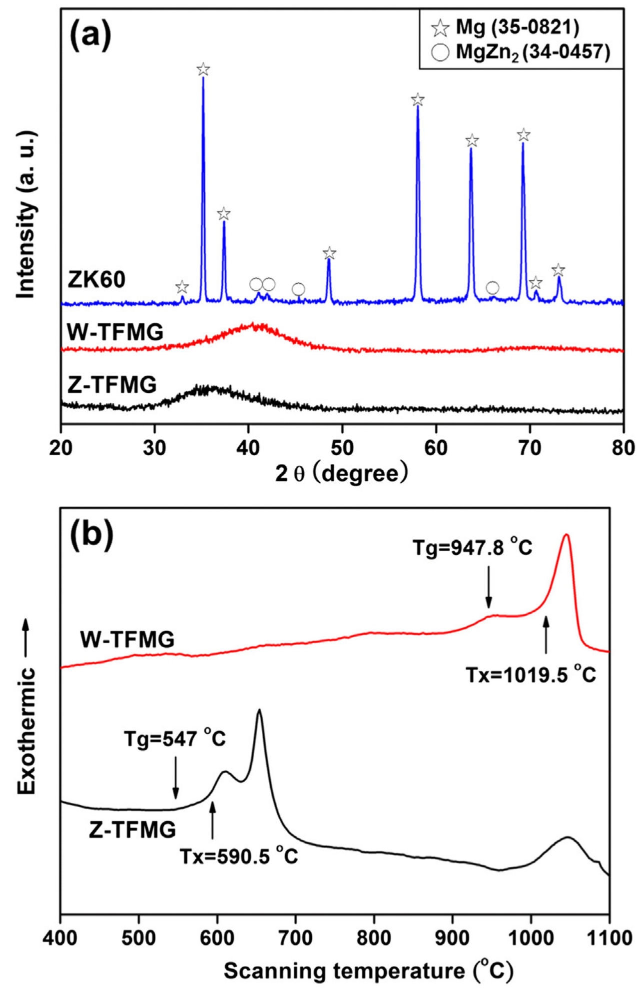

Fig. 1. (a) XRD diffraction patterns of TFMGs and ZK60 Mg alloy substrate; and (b) DSC curves of TFMGs.

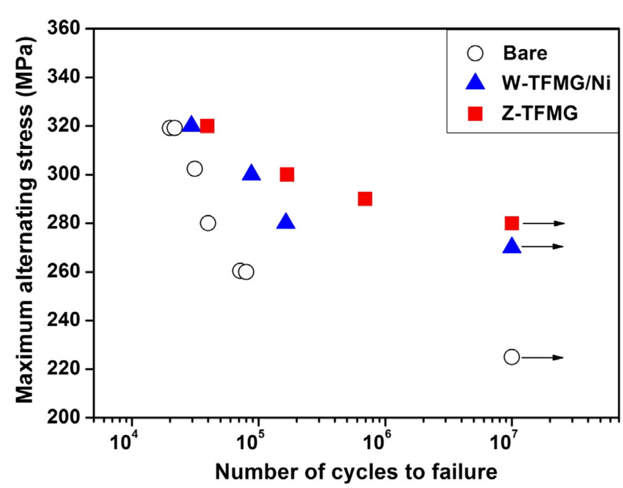

Fig. 2. S-N curves of coated and bare samples obtained from four-point-bending fatigue tests.