Effects of Ultrasonic Nanocrystal Surface Modification on Mechanical and Corrosion Behavior of LZ91 Mg–Li Alloy

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The effects of ultrasonic nanocrystal surface modification (UNSM) treatments on the microstructure, mechanical, and corrosion behavior of LZ91 Magnesium–Lithium (Mg–Li) alloy were systematically investigated. The results show that UNSM treatments have greatly positive effects on the mechanical and corrosion behavior improvements. As compared to the bare sample (BS), the near-surface grains were refined and the surface roughness (R_a) of the sample was reduced from 0.514 μm to 0.082 μm ; and the maximum hardness and tensile yield strength remarkable increased by 70% and 65.4% after UNSM treatments, respectively. In addition, the corrosion behavior of LZ91 Mg–Li alloy was also improved after UNSM treatments. The mechanical and corrosion behavior improvements can be attributed to the introduced thick hardening layer with low roughness and smaller grains by UNSM treatments. [doi:10.2320/matertrans.MT-M2019314]

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1. Introduction

Mg alloys as a combination of the lightest metal Mg in the commonly used structural materials and other elements (Li, Al, Zn, rare earth element, and etc.) are widely used. At present, with the increase in the requirements for the lighter weight of machinery and equipment, Mg–Li alloys have been used in aerospace, automotive, electronics, and national defense industries.^{1–3)} All of this owing to their superior properties such as high specific strength and specific stiffness, low density, strong damping and noise reduction ability, good shielding electromagnetic radiation performance, and recyclability. However, due to the low strength and poor corrosion resistance of Mg–Li alloys, it is difficult to apply widely in various fields. Therefore, the improvement of mechanical properties and corrosion resistance performance of Mg–Li alloys has become a problem which needs to be solved urgently. Normally, the mechanical behavior and corrosion resistance of the Mg–Li alloy can be improved by alloying,⁴⁾ post-processing (i.e. hot rolling, extrusion, and heat treatment),^{5,6)} and surface coating.⁷⁾ Simultaneously increase strength and corrosion resistance by alloying, but it is easy to cause elemental pollution and waste. Post-processing can significantly increase the strength of the material by refining the grains, but the plasticity is obviously reduced. The coating on the surface of the material is relatively brittle, and there is a clear interface with the substrate, which is easy to fall off. Recently, the severe surface plastic deformation (S^2PD) technique was also proposed enhancing the performance of the material. The surface treatment of the material realizes the purpose of improving the strength and corrosion resistance performance.^{8–11)} In addition, the S^2PD technique has also widely applied to improve fatigue strength and wear resistance performance of the material.^{12–14)}

The S^2PD technique is to apply energy to a medium (water, pellets or ball tool, etc.) and then act on the surface of metal materials so that the surface of materials subjected to S^2PD and forms a metamorphic layer with a certain depth. It

can change the surface microstructure, introduce residual compressive stress, refine the surface grain, and strengthen the surface of materials, so as to achieve the purpose of improving the overall performance of materials. In general, nanocrystalline materials have higher strength and hardness. With the increase in the hardness and strength of nanometer materials, their plasticity and toughness gradually decrease, the work hardening capacity disappears, and the structural stability worsens.¹⁵⁾ The S^2PD technique could induce a deeper gradient nanostructure surface layer on the surface of materials, and while enhancing the strength of the material, it retains ductility to some extent.¹⁶⁾ The current technologies for obtaining the S^2PD layer on the surface of metallic materials mainly including shot peening (SP),^{17,18)} ultrasonic peening (UP),¹⁹⁾ ultrasonic surface rolling processing (USRP),²⁰⁾ laser shock peening (LSP),²¹⁾ surface mechanical grinding treatment (SMGT),^{22,23)} high pressure waterjet peening,^{24,25)} UNSM, and etc.

The UNSM technique, a newly developed cold processing method, which was applied to improve mechanical behavior and corrosion resistance of the materials. UNSM treatments have many advantages compared with other surface deformation strengthening technique. UNSM treatments can change the surface morphology of the material and significantly improve the surface roughness. Besides, the operation of UNSM technique is simple and the whole process is environmentally-friendly and highly controllable. Recently, Hou *et al.*²⁶⁾ utilized UNSM technology to process AZ31B Mg alloy, the results showed that UNSM could significantly improve the mechanical properties of Mg alloys without compromising their corrosion rate. Kim *et al.*²⁷⁾ focus on the effect of the static load in UNSM on the corrosion properties of Alloy 600, they found that an increase in static load under critical static load improved corrosion performance.

At present, only a few kinds of researches have been carried out on the effect of UNSM treatments on Mg alloys, especially dual-phase Mg–Li alloys were rarely investigated. In this study, the mechanical behavior and corrosion resistance of the industrial dual-phase LZ91 Mg–Li alloy before and after UNSM treatments were investigated.

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Fig. 1 Schematic of the UNSM process.

2. Experimental Details

2.1 Sample preparations

The LZ91 Mg–Li alloy sheet with the measured chemical composition of 8.9 mass% Li, 0.98 mass% Zn, and balance Mg was utilized. Samples (130 mm × 130 mm × 2 mm) were cut by electric discharge machining from the rolled plate, and then solution-treated at 573 K for 60 min, followed by water quenching. For mechanical and corrosion behavior testing, the base samples were ground using wet sandpaper from 480 to 2000# and its surface roughness reached approximately 0.15 μm .

The schematic diagram of the UNSM process is illustrated in Fig. 1. The working principle of UNSM is that the ultrasonic generator converts alternating current voltage into ultrasonic frequency oscillation signal, and the ultrasonic transducer converts it into ultrasonic frequency mechanical vibration. The vibration amplitude is amplified by the amplitude transforming rod and then transmitted to the ball tool to generate a certain amount of energy which was applied to process the workpiece. During UNSM treatments, a static pressure of 0.1 MPa was applied to the surface of the sample through the tungsten carbide ball. At the same time, the ball continuously impacted the surface of the sample at a frequency of 28 kHz ultrasonic vibration, which is higher than the used frequency of 20 kHz in most studies.^{8,13,14,26,28–30} In this study, the scanning speed was 1500 mm/min, the interval of two adjacent UNSM treatment tracks was 0.1 mm and the diameter of the ball is 14 mm. Hereafter, the sample with UNSM processing once and twice treatments were labeled as UNSM-1 and UNSM-2, respectively. And the base sample was labeled as BS for comparison.

2.2 Microstructural characterization

To reveal the subsurface microstructure, the samples with/without UNSM treatments were cut along the cross-section, inlaid with epoxy resin, metallographically grinding, and polishing, followed by etching with 2 vol% nitric acid alcohol solution, and then observing the microstructure of the sample along the gradient direction using optical microscopy (OM).

The effect of UNSM treatments on surface roughness was also investigated. The three-dimensional surface topography

measurement system (Bruker's NPFLEX™ 3D Surface Metrology System) was used to measure the change of the surface roughness of the sample before and after UNSM treatments. The measurement area was $472 \times 629 \mu\text{m}^2$. Before and after UNSM treatments, a total of five measurements were made on each sample at different areas and the average value was selected as the standard value.

2.3 Mechanical testing

The distribution of the microhardness value along the UNSM treated surface to the base of the samples was performed using the HV-1000 Vickers hardness tester with a diamond pyramid indenter with an experimental force of 0.5 N. The loading or unloading rates of 300 mN/min, the load was held for 15 s, and the distance between adjacent indentations was 60 μm . Average hardness values of five points at every depth were selected for description.

Tension tests were carried out on a hydraulic servo fatigue tester (MTS 370.02) at room temperature (RT). An extensometer was used during the stretching process with a strain rate of $1 \times 10^{-3} \text{ s}^{-1}$. The tensile samples were subjected to double-sided UNSM treatments. The gauge length, width and thickness of the tensile samples are 10, 4 and 2 mm, respectively. In order to ensure the reliability of the experimental results, the tensile test of the sample before and after UNSM treatments was performed at least 3 times.

2.4 Corrosion testing

The corrosion performances of the samples before and after UNSM treatments were studied by electrochemical impedance spectroscopy (EIS), and potentiodynamic-polarization curves in a 3.5 mass% NaCl solution. The electrochemical experiments were carried out on an RST 5000 workstation based on the conventional three-electrode system, in which the working electrode was Mg–Li alloy electrode (10 mm × 10 mm), the auxiliary electrode was a platinum electrode, and the reference electrode was a saturated calomel electrode (SCE). After 20 minutes OCP test, the EIS tests were performed at the OCP in the frequency range of 100000 to 0.01 Hz with a 7 mV amplitude perturbation (peak-to-zero) and the EIS data were obtained. Each sample was soaked in NaCl solution to obtain a stable OCP for 5 minutes, and then, the potentiodynamic polarization curves were obtained by the potentiodynamic polarization scanning method at a scanning rate of 1 mV/s. In order to obtain reliable data, the test was performed for each process condition at least 3 times.

3. Results and Discussions

3.1 Microstructures

The cross-sectional microstructures of LZ91 Mg–Li alloy after UNSM treatments are shown in Fig. 2. The LZ91 Mg–Li alloy represents a typical duplex phase (hcp + bcc) structure, the phase in the bright color is the hcp α phase and that in gray is the bcc β phase (Fig. 2(a)). It is clear that the UNSM treatments induce an obvious gradient microstructure with an SPD layer and the near-surface grain is significantly refined (Fig. 2(b) and (c)). The severe plastic deformation layer of the UNSM-2 sample is obviously thicker than that of

Fig. 2 Optical images of the microstructures observed from cross-sectional of LZ91 Mg-Li alloy (a) BS, (b) UNSM-1, and (c) UNSM-2.

Fig. 3 Three-dimensional (3D) microscope images of (a) BS, (b) UNSM-1, and (c) UNSM-2 samples, and (d) responding surface roughness (R_a) of the samples before and after UNSM treatments.

the UNSM-1 one, which can be attributed to the strong and repeated impacts.

3.2 Variation of surface roughness

The 3D surface topographies and the average roughness R_a before and after UNSM treatments are shown in Fig. 3. From Fig. 3(a), it can be clearly seen that the scratches left on the sample by ground with sandpaper, and the tracks of UNSM treatments could be found from Fig. 3(b) and (c). It can be inferred that when the sample was subjected to the initial UNSM treatments (UNSM-1), the severe plastic deformation of the sample surface can remove the scratches left by sandpaper and improve the surface roughness.

However, when the sample was re-treated (UNSM-2), further surface deformation occurred on the sample surface and the deeper tip traces of UNSM treatments appeared. The average surface roughness R_a of matrix roughness is $0.514 \mu\text{m}$, the roughness R_a of the UNSM-1 and UNSM-2 samples are $0.082 \mu\text{m}$ and $0.145 \mu\text{m}$, respectively (Fig. 3(d)). That is, the UNSM treatments can significantly reduce the roughness (reduced by about 84% and 72% for UNSM-1 and UNSM-2). This is consistent with the previous study²⁹ that processing the quenching and tempering S45C steel by changing the UNSM processing density, and the results showed that the surface roughness of the sample deteriorated as the processing density increased.

Fig. 4 The results of microhardness distribution along the cross-section of samples before and after UNSM treatments.

3.3 Mechanical behavior

The variation of microhardness along the depth profiles of LZ91 samples before and after UNSM treatments are illustrated in Fig. 4. It can be noted that the average surface microhardness value of the UNSM-1 and UNSM-2 samples is 78 HV_{0.05} and 85 HV_{0.05}, respectively. Corresponding to 56% and 70% enhancement, as compared to the BS one (~50 HV_{0.05}). It can be clearly seen that with the increase of distance from the top surface, the value of microhardness gradually decreases and tends to a constant at the depth of about 600 μm. It can be ascribed to the formation of the gradient structure layer caused by UNSM treatments. That is, one would expect an increase in microhardness for smaller grain sizes according to classical Hall-petch relationship.^{30,31} During UNSM treatments, the S²PD induces different grain refinement effect due to the gradient nature of the plastic strain in the depth direction. In addition, the increase of microhardness of the sample after UNSM treatments could be the result of work hardening. Grain refinement means high volume fraction of grain boundary and work hardening lead to the increase of dislocation density, the synergy between them caused the enhancement of microhardness. Compared to UNSM-1, the sample of UNSM-2 has relatively higher and homogeneous microhardness value, which may be related to processing times of UNSM.

The engineering stress-strain curve obtained by stretching the Mg–Li alloy at RT is shown in Fig. 5. During UNSM treatments, the surface of the Mg–Li alloy occurs surface severe plastic deformation, the grain of the surface layer was refined, and the dislocation density and the volume fraction of grain boundary increased. As a result, the tensile yield strength (TYS) and ultimate tensile strength (UTS) of LZ91 Mg–Li alloy are increased. The YYS, the UTS and the elongation of LZ91 Mg–Li alloy before and after UNSM treatments at RT are shown in Table 1. The YYS of the Un-treated samples was ~81 MPa, and the YYS of the UNSM-1 and UNSM-2 samples was ~122 MPa and ~134 MPa, respectively. Compared to the Un-treated sample, the YYS of the sample after UNSM treatments was increased by 50.6% and 65.4%. This shows that UNSM treatment can significantly increase the yield strength of LZ91 Mg–Li alloy. The UTS of the UNSM-2 samples was ~147 MPa

Fig. 5 The engineering stress-strain curves of LZ91 Mg–Li alloy before and after UNSM treatments at RT.

Table 1 The tensile yield strength, the ultimate tensile strength, and the elongation of LZ91 Mg–Li alloy before and after UNSM treatments at RT.

Samples	TYS (MPa)	UTS (MPa)	E _f (%)
BS	81 ± 5	118 ± 4	47 ± 4
UNSM-1	122 ± 4	140 ± 3	43 ± 5
UNSM-2	134 ± 3	147 ± 2	45 ± 3

compared to the BS samples with a UTS of ~118 MPa. The UTS of the samples after UNSM treatments increased by 24.6%, it is clear that UNSM treatments can improve the UTS of Mg–Li alloys. The maximum YYS and UTS of the sample remarkably enhance by ~65.4% and 24.6% without striking loss elongation after UNSM treatments, which can be attributed to the formed thick hardening layer with gradient microstructure. Comparing to strength of the UNSM-1 samples, the strength of UNSM-2 further enhance. This can be mainly ascribed to the thicker S²PD layer of the UNSM-2 sample (Fig. 2). In general, the relationship between strength and ductility of material is when one is rising, the other is falling. As the strength increases, the ductility of the material decreases significantly. The BS sample has an elongation of about 47%, and the UNSM-1 and UNSM-2 samples have elongations of 43% and 45%, respectively. It is evident that the plasticity does not decrease significantly with the apparent increase in the strength of the Mg–Li alloy after UNSM treatments. The tensile stress-strain curves still show a gentle upward trend after UNSM treatments, this shows that the material has a certain work hardening effect during the stretching process at RT. It could be concluded that LZ91 Mg–Li alloy can retain the ductility and work hardening capacity to some extent while improving the strength by UNSM treatments.

3.4 Corrosion behavior

Figure 6 shows the potential-time curves of LZ91 Mg–Li alloy electrodes before and after UNSM treatments at RT. It can be seen that the electrodes exhibit similar electrochemical behavior. After 1200 s immersion, the average OCP of BS, UNSM-1, and UNSM-2 electrode was –1.646 V, –1.646 V, and –1.641 V, respectively. It could be found that each

Fig. 6 Galvanostatic potential-time curves of LZ91 Mg-Li alloy electrode before and after UNSM treatments in 3.5 mass% NaCl solution at RT.

electrode has a relatively close average OCP. In addition, it was no obvious abrupt change after the OCP was stabilized, which indicated that there is no apparent interface between the S²PD layer and the core matrix.

The corrosion performance of the LZ91 Mg-Li alloy electrodes before and after UNSM treatments, which is studied by EIS and potentiodynamic-polarization curves, is shown in Fig. 7. The Nyquist plots of the samples, which are obtained from the EIS, are exhibited in Fig. 7(a). The EIS data were fitted by the ZSimpWin 3.10 software using the given equivalent circuit $R_s(QR_{ct}(LR_1))$ (Inset in Fig. 7(a)) and the fitting parameters are summarized in Table 2. In the equivalent circuit, R_s and R_{ct} represent the solution

Table 2 The EIS parameters of LZ91 Mg-Li alloy extracted from the fitted equivalent circuit.

Parameter	BS	UNSM-1	UNSM-2
R_s ($\Omega \text{ cm}^2$)	5.118	4.705	4.009
CPE ($\text{S cm}^{-2} \text{ s}^n$)	8.07×10^{-5}	3.95×10^{-5}	3.72×10^{-5}
n	0.8419	0.8392	0.8837
R_{ct} ($\Omega \text{ cm}^2$)	120.0	307.3	246.1
L (H cm^2)	272.3	620.8	697.7
R_1 ($\Omega \text{ cm}^2$)	32.8	102.8	104.0
R_p ($\Omega \text{ cm}^2$)	152.8	410.1	350.1

resistance and charge-transfer resistance. The resistance, R_1 , and inductance, L , are used to represent the inductive behavior. The constant phase element (CPE, designated as Q) is used instead of the ideal double-layer capacitance (Cdl) to account for the non-ideal behavior of the double-layer due to the surface inhomogeneity, roughness, and adsorption effects.³²⁾ The polarization resistance, R_p , is calculated as $R_p = R_1 + R_{ct}$. Figure 7(b), (c) show the Bode plot of EIS of the LZ91 Mg-Li alloy. It is clear that the Mg-Li alloy electrode before and after UNSM treatments exhibit similar electrochemical impedance behavior, and all the samples are composed of a large-capacity anti-arc in the high-frequency and intermediate-frequency regions and an inductive arc in

Fig. 7 Electrochemical impedance spectra (a), (b), (c) and potentiodynamic polarization curves (d) of LZ91 Mg-Li alloy electrode before and after UNSM treatments in 3.5 mass% NaCl solution at RT.

the low-frequency region. The capacitive reactance arc corresponds to a larger phase angle peak in the high-frequency and intermediate-frequency regions of the Bode plot. In the low-frequency region, the Mg–Li alloy exhibits a large diameter inductive arc corresponding to the phase angle peak of the lower frequency region in the low frequency region of the Bode plot. In the low-frequency region, there is a large diameter inductive arc of Mg–Li alloy electrode, which corresponds to the phase angle peak of lower convex in the low-frequency region of the Bode plot. In contrast, the UNSM-1 sample have larger capacitive reactance diameters than the BS and UNSM-2 samples. Combined with the Bode plot, the impedance modulus values of the UNSM-1 samples are larger than BS and UNSM-2 over the entire frequency range. This result indicate that the BS sample has a small charge transfer resistance and strong activity, which is beneficial to the activation and dissolution of the electrode at the corrosion potential, which is basically consistent with the results in Table 2. The polarization resistance, R_p , of UNSM-1 and UNSM-2 is 410.1 and 350.1 $\Omega \text{ cm}^2$, respectively. Increased by 168% and 129%, as compared to the BS one (152.8 $\Omega \text{ cm}^2$), indicating the enhanced corrosion resistance performance with the UNSM treatments.

The polarization curve measured by the potentiodynamic polarization can reflect the discharge behavior of the Mg–Li alloy electrode over a wide voltage range. The potentiodynamic polarization curves of LZ91 Mg–Li alloy electrodes before and after UNSM treatments in 3.5 mass% NaCl solution at RT are shown in Fig. 7(d). The calculated electrochemical corrosion data, corrosion current-density (i_{corr}), corrosion potential (E_{corr}), anode Tafel slope (b_a), and cathode Tafel slope (b_c), determined from the potentiodynamic-polarization curves are summarized in Table 3. The E_{corr} of the Mg–Li alloy electrode treated by UNSM is slightly positive than that of the BS one, indicating that UNSM treatments can slightly positively shift the corrosion potential of the LZ91 Mg–Li alloy electrode and reduce the corrosion driving force. In addition, the BS electrode has a greater current density than the samples after UNSM treatments during cathodic polarization. Which indicates that the BS electrode has a faster activation dissolution rate at the corrosion potential, and UNSM treatments can reduce the activation dissolution rate of LZ91 Mg–Li alloy.

Generally, the corrosion resistance behavior of the material is directly related to the surface grain size³³⁾ and surface roughness.³⁴⁾ The smaller grains and smoother surface help to improve the corrosion resistance of the material. Due to the grain refinement of the surface layer, the volume fraction of

grain boundary increases, and it may provide more diffusion paths than the coarse grain to form a denser passive film on the surface of the material.^{35,36)} In general, a smoother surface offers better corrosion resistance and vice versa.³⁷⁾ When the surface roughness deteriorates, making it easy to absorb the corrosive medium, which is advantageous for the generation and growth of the metastable etch point, thereby suppressing alloy passivation.³⁴⁾ That is, the improved corrosion resistance of the LZ91 Mg–Li alloy can be ascribed to the obvious surface grain refinement effect and the lower surface roughness after the UNSM treatments. Compared with UNSM-1, the smaller improvement in corrosion resistance of UNSM-2 samples may be attributed to deterioration of surface roughness.

4. Conclusions

The effects of UNSM treatments on and the microstructure, mechanical, and corrosion behavior of LZ91 Mg–Li alloy were systematically investigated. According to our observation, the following conclusions can be drawn:

- (1) An obvious surface severe plastic deformation layer of LZ91 Mg–Li alloy was induced by UNSM treatments. The near-surface grains were refined, and the surface roughness (R_a) was significantly lowered after UNSM treatments (reduced by $\sim 84\%$ and 72% for UNSM-1 and UNSM-2).
- (2) The microhardness of the LZ91 Mg–Li alloy was significantly enhanced after UNSM treatments and the hardening layer is $\sim 600 \mu\text{m}$. As compared to the base alloy, the maximum hardness of UNSM-1 and UNSM-2 samples increased by 56% and 70% , respectively.
- (3) The maximum TYS and UTS of the sample are remarkably enhanced by $\sim 65.4\%$ and 24.6% without striking loss elongation after UNSM treatments, which can be attributed to the formed thick hardening layer.
- (4) The corrosion performance of the LZ91 Mg–Li alloy was also improved after UNSM treatments, which can be attributed to the smaller grains and smoother surface induced by UNSM treatments.

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Table 3 Corrosion potentials (E_{corr}), corrosion current densities (i_{corr}), anode Tafel slope (b_a), and cathode Tafel slope (b_c) of LZ91 Mg–Li alloy electrodes before and after UNSM treatments.

Samples	E_{corr} (vs SCE)/V	$i_{\text{corr}}/(\mu\text{A} \cdot \text{cm}^{-2})$	b_a (V/dec)	b_c (V/dec)
BS	-1.571 ± 0.011	166.5 ± 6.1	0.253 ± 0.006	0.057 ± 0.009
UNSM-1	-1.556 ± 0.002	70.8 ± 6.3	0.258 ± 0.005	0.089 ± 0.004
UNSM-2	-1.559 ± 0.005	109.3 ± 11.5	0.240 ± 0.005	0.067 ± 0.002

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