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### Nuclear Instruments and Methods in Physics Research B

journal homepage: www.elsevier.com/locate/nimb



# Producing nano-grained and Al-enriched surface microstructure on AZ91 magnesium alloy by high current pulsed electron beam treatment



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#### ARTICLE INFO

Article history:
Received 16 February 2016
Received in revised form 13 March 2016
Accepted 18 March 2016
Available online 23 March 2016

Keywords:
High current pulsed electron beam
AZ91 magnesium alloy
Surface treatment
Microstructure
Microhardness
Corrosion resistance

#### ABSTRACT

Surface treatment of AZ91 magnesium alloy was carried out by high current pulsed electron beam (HCPEB) with accelerating voltage 27 kV and energy density 3 J/cm². The surface microstructure and phase composition were characterized by using optical microscope (OM), X-ray diffraction (XRD), and scanning electron microscope (SEM) equipped with energy dispersive spectrometer (EDS). The surface microhardness and corrosion resistance were measured. Under HCPEB treatments, the preferential evaporation of Mg element occurred intensively on irradiated surface and the initial large  $Mg_{17}Al_{12}$  phases were dissolved. The nano-grained and Al-enriched surface modified layer was ultimately formed of depth  $\sim$ 8 µm. According to the testing results, the surface microhardness increased from 63 to 141 HK after 30 pulses of HCPEB treatment, while the best improvement of corrosion resistance was obtained by 15 pulses of HCPEB treatment with a cathodic current density decreased by two orders of magnitude as compared with the initial AZ91 sample.

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#### 1. Introduction

In recent years, high current pulsed electron beam (HCPEB) technique has been widely investigated as an efficient method for surface modification of materials [1–6]. With the extremely high energy deposition onto target surface within a short pulse of microsecond level, the processes including rapid heating ( $\sim 10^8~{\rm Ks}^{-1}$ ) to melting or evaporation, thermal stress impact ( $\sim {\rm GPa}$ ) and self-quenching ( $\sim 10^7~{\rm Ks}^{-1}$ ) effects occur drastically, which give rise to the formation of non-equilibrium surface microstructure accompanied by modified properties. It was found that the ultra-refined surface microstructure of grain size less than 100 nm could be produced by HCPEB treatment on carbon steels, titanium alloys and even WC/Co hard alloys while the modified surface exhibited noticeable improvement in mechanical and electro-chemical corrosion performance [7–10].

It is swell recognized that magnesium alloys can provide promising advantages as applying in automobile, electronics, aerospace and biomaterial industries for their low density, high specific strength, electromagnetic shielding, damping and recyclable properties. However, the inherent tender surface and reactivity of Mg element constitute the main limitations for their wide applications [11–15]. Up to now, a variety of surface engineering methods have

been essayed, such as chemical conversion coating, micro-arc oxidation, electroplating, shot peening, surface mechanical attrition treatment (SMAT), laser cladding and ion implantation etc., and the enhancement of surface microhardness or corrosion resistance had been achieved under certain circumstances [16–24]. Considering the further applications of magnesium alloys, new and effective surface treatment techniques are still urgently desired especially for high-efficiency, simple operability and comprehensive performance.

In this work, the HCPEB treatment was conducted on a commonly-used AZ91 magnesium alloy. The surface microstructure and phase composition were characterized with efforts to investigate the HCPEB modification regularities and explore an effective method to improve the surface microhardness and corrosion resistance of magnesium alloys simultaneously.

#### 2. Experimental procedure

The samples of size  $\Phi12 \times 6$  mm were cut directly from a cast ingot of AZ91 magnesium alloy. The chemical composition (wt.%) was 8.76 Al, 0.69 Zn, 0.27 Mn, Mg balance. All the samples were grounded, polished and ultrasonically cleaned in acetone. The surface treatment was conducted on a HCPEB equipment of type HOPE-I with working parameters as following: vacuum pressure  $7 \times 10^{-3}$  Pa, accelerating voltage 27 kV, energy density 3 J/cm², pulse duration 2.5  $\mu$ s and pulse number 3, 8, 15 and 30.

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The surface microstructure was observed using Olympus BX51 optical microscope (OM) and Zeiss Supra55 scanning electron microscope (SEM) equipped with energy dispersive spectrometer (EDS). The phase composition was examined using Bruker D8 X-ray diffraction (XRD) with CuKα radiation and step size of 0.02°. The surface microhardness (HK) was measured using DMH-2LS tester at a load of 10 g. The corrosion resistance was measured using CS350 electrochemical workstation with Pt counter electrode and saturated calomel electrode (SCE) in 3.5 wt.% NaCl solution.

#### 3. Results and discussion

#### 3.1. Surface morphology

As shown in Fig. 1a, the initial microstructure of AZ91 sample was composed of primary  $\alpha\textsc{-Mg}$  dendrites of average size  $\sim\!200~\mu m$  and large  $\beta\textsc{-Mg}_{17}\textsc{Al}_{12}$  phases dispersed mainly at grain boundaries. After 3 pulses of HCPEB treatment, the surface became fluctuant and a lot of craters were observed (Fig. 1b). The formation mechanism of surface craters under HCPEB treatment was detailed in the previous studies, in the present case, the large Mg $_{17}\textsc{Al}_{12}$  particles would provide the favorable positions of eruption center [25,26]. Besides, numerous small ablation pits emerged on the modified surface, as denoted in Fig. 1b. After 8 pulses of HCPEB treatment, the modified surface became flatter and the large cra-

ters were healed substantially (Fig. 1c). While the small and dense ablation pits were still kept, that was closely related to the intensive evaporation occurring on irradiated surface. From the highresolution SEM image, a very refined microstructure was formed with a mosaic contrast difference. For the sample treated with 15 HCPEB pulses, the modified surface became very flat (Fig. 1d). By HR-SEM image, the regular nano-grained microstructure of average size ~70 nm and network-like arrangement was observed on the modified surface. When the more HCPEB pulses applied, microcracks would appear and tend to be severe, as shown in Fig. 1e. The formation of surface microcracks can be attributed to the tensile stress produced in surface layer during the rapid solidification and quenching processes of HCPEB treatment. Besides, it is suggested that the modified surface layer became more and more brittle due to the related phase composition transformations as described in the followings.

#### 3.2. Cross-sectional morphology

Fig. 2 gives the cross-sectional SEM observation results. After 3 pulses of HCPEB treatment, the surface modified layer was not homogenous where the top part of  $Mg_{17}Al_{12}$  phase was melted and dissolved into the surround matrix (Fig. 2a). At the position corresponding to initial  $\alpha$ -Mg grain, according to the EDS line scanning result, the content of Al element was noticeably higher in the

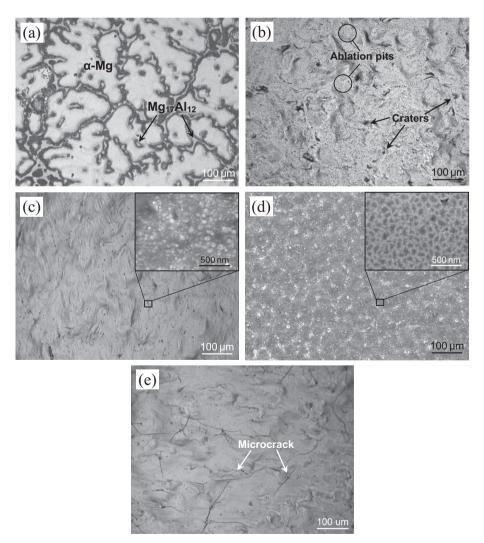


Fig. 1. Surface OM images of initial AZ91 sample (a) and after HCPEB treatment with 3, 8, 15 and 30 pulses (b-e), and the high-resolution SEM images were given as insets.

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