Original

Schlueter, K.; Reverey, J.; Hort, N.; Zamponi, C.; Quandt, E.:

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Materials Science Forum, Light Metals Technology V (2011)
Trans Tech Publications

DOI: 10.4028/www.scientific.net/MSF.690.286

Materials Science Forum Vol. 690 (2011) pp 286-289

doi:10.4028/www.scientific.net/MSF.690.286

Mechanical behaviour and corrosion performance of thin film magnesium WE alloys

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Keywords: magnesium alloys, magnetron sputtering, thin films, tensile tests, polarization measurements

Abstract. Mg based thin films are of increasing interest due to the potential in varying the corrosion properties in comparison to bulk alloys of the same nominal composition.
In this work the mechanical behaviour and the corrosion performance of sputtered thin films consisting of magnesium alloys with the compositions Mg4Y3Gd and Mg4Y3Nd were investigated by tensile tests and electrochemical corrosion tests, respectively.
The tensile tests showed that the sputtering parameters have an enormous influence on the mechanical properties of the thin films. By variation of the Ar sputtering pressure and the DC sputtering power it was possible to fabricate films with widely varying mechanical behaviour reaching from brittle to very ductile films which could be strained to more than 30%. The determined dependency between mechanical properties and deposition conditions was found to be present in both investigated alloys. The corrosion tests revealed that the corrosion behaviour of the investigated samples is not correlated to the mechanical properties of the thin films, as the rate of corrosion and the tendency to localized corrosion do not change significantly upon the deposition conditions, which dramatically influence the mechanical properties.

Introduction

Magnesium and its alloys are attractive construction materials, as they have the lowest density and the highest strength-to-weight-ratio of all metallic engineering materials [1]. Especially in thin films the corrosion behaviour can easily be varied [2]. Despite these favourable features the poor corrosion properties of magnesium are a major disadvantage, which limits the field of applications in the aircraft and automotive industry. WE magnesium alloys with yttrium and rare earths as main alloying elements are often used when creep resistance up to a temperature of 250 °C, high specific strength, good castability and corrosion resistance are needed [1,3].
Usually the alloyed rare earths are a mixture of Ce, La, Nd, Gd, Pd and others, but in this work alloys were prepared which contain yttrium and only one other rare earth element (either Gd or Nd). The alloys Mg4Y3Gd and Mg4Y3Nd were chosen, as Nd and Gd differ significantly in their solid solubility.
The aim of this work was to study the influence of sputtering parameters as Ar sputtering pressure and sputtering power on the mechanical behaviour and the corrosion properties of thin film samples consisting of both alloys.

Experimental

Test materials. The used target materials were cast by the Helmholtz-Zentrum Geesthacht using permanent mould direct chill casting. High-purity Mg was melted in a mild steel crucible under a protective atmosphere (Ar + 2% SF6). Pure REs were added at a melt temperature of 700 °C. The melt was stirred for 30 min at 140 rpm. Then the melt was transferred into a preheated thin walled mould, kept at 650 °C for 0.5h and the quenched in water with a temperature of 16 °C with 12 cm/min. The chemical composition measured by EDX is given in table 2. Both investigated alloys belong to the class of WE43 alloys, but differ largely in the solubility of the rare earth element in the magnesium matrix. Gd has a maximum solid solubility of c_s = 23.5 wt.%, whereas Nd exhibits a maximum solid solubility of only c_s = 3.6 wt.% [4].

Preparation. The thin film specimens with a film thickness of 10 µm were sputtered using a VonArdenne CS 730 S cluster magnetron sputtering system. The samples used for the corrosion tests were fabricated on oxidized silicon substrates with the dimensions of 15×15mm while the specimens used in the tensile tests were deposited on 3×50mm silicon substrates which were previously coated with an organic sacrifical layer to get a free standing film. After the deposition the samples were put in the solvent *N*-Methyl-2-pyrrolidone (NMP) and subsequently dried in warm flowing air. The various sputtering conditions used in this work are given in table 1.

Table 1: Sputtering conditions for the investigated samples

Sample	sputtering pressure [mbar]	Ar flow [sccm]	sputtering power [W_{DC}]
-A1	2.0×10^{-2}	30	50
-A2			500
-B1	2.3×10^{-3}	20	50
-B2			500

Characterization. The chemical composition of the used targets was determined using an Inca Oxford attached to a FEI Dualbeam Helios Nanolab.
Prior to the corrosion tests the microstructure of the thin film specimens was analyzed by X-ray diffraction with a Seifert XRD 3000 PTS.
A VersaSTAT 3-300 potentiostat connected to a three-electrode cell was used for the electrochemical measurements. The working electrode was the test material and the counter and reference electrode were Pt mesh and Ag/AgCl, respectively. As electrolyte PBS (8.0 g/l NaCl, 0.2 g/l KCl, 0.2 g/l KH_2PO_4, 1.15 g/l Na_2HPO_4) was used. The polarization measurements were carried out at a scan rate of 1 mV/s from – 300 mV to + 600 mV with respect to the free corrosion potential (E_{corr}). The measurement was stopped, when the current density exceeded significantly 10^{-3} A/cm². Previous to each measurement the open circuit potential was monitored for 10 minutes to ensure a steady state potential at the beginning of the polarization measurements. All measurements were performed at room temperature.
For the tensile tests the universal tensile test machine Messphysik Beta 5-5/6 x 10 was used. The tests were performed with a clamping length of 5 mm and a strain rate of 0.4% per minute.

Results

EDX-Measurements. The chemical composition of the target material and the investigated thin films is shown in table 2. The real contents of REs are approximately 10 % lower compared to the real measured values. This is a typical range for the burn of or RE during melt handling [5,6].

Table 2: Chemical composition of the used target material

Mg alloy	Y (wt.%)		Gd (wt.%)		Nd (wt.%)		Mg (wt.%)
	measured	nominal	measured	nominal	measured	nominal	
Mg4Y3Gd	3.5 ± 0.4	4	2.7 ± 0.3	3	-	-	balance
Mg4Y3Nd	3.6 ± 0.3	4	-	-	2.8 ± 0.3	3	balance

Microstructural characterization. The XRD measurements show that all investigated samples regardless of alloy composition or deposition parameters exhibit a microstructure consisting of a crystalline, textured, supersaturated solid solution with no detectable precipitates.

Corrosion tests. Fig. 1 a and b show the potentiodynamic polarization curves of Mg4Y3Gd and Mg4Y3Nd deposited at various sputtering pressures and sputtering powers. It can be seen, that neither the free corrosion potential E_{corr} nor the corrosion current density i_{corr} vary significantly with the changing deposition conditions. The corrosion rates are 15.3 ± 0.7 µm/month for the alloy Mg4Y3Gd and 16.2 ± 3.1 µm/month for the alloy Mg4Y3Nd. The free corrosion potential has been determined to be -1.85 ± 0.03 V vs. Ag/AgCl for both investigated alloys.

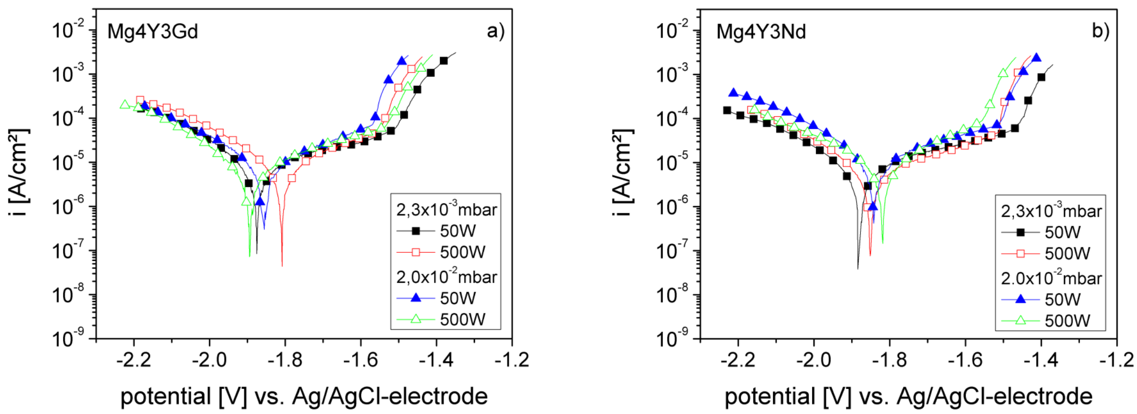

Fig. 1: Potentiodynamic polarization curves of (a) Mg4Y3Gd and (b) Mg4Y3Nd deposited at two different sputtering pressures and sputtering powers

Tensile tests. Fig. 2 shows exemplarily the results of the tensile tests performed on the samples deposited with a sputtering power of 50W at various Ar sputtering pressures.

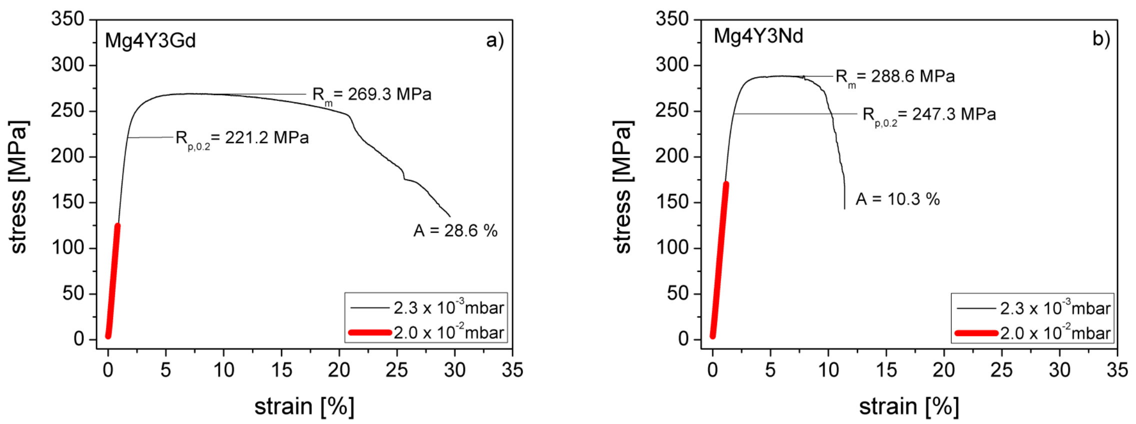

Fig. 2: Results of the tensile tests for (a) Mg4Y3Gdand (b) Mg4Y3Nd deposited at an Ar sputtering pressure of 2.3×10^{-3} mbar and 2.0×10^{-2} mbar with a sputtering power of 50W. Displayed are the values for maximum strain (A), 0.2% proof stress ($R_{p,0.2}$) and tensile strength (R_m).

The mechanical properties of the investigated alloys are displayed in fig.2 a and b. The maximum strain, yield and tensile strength of the thin films deposited at 2.0×10^{-2} mbar Ar sputtering pressure could not be determined, as all investigated samples broke in the elastic region.

The Young's modulus was determined to be 15.5 ± 0.5 GPa for all investigated samples regardless of the alloy composition and the sputtering parameters. In addition the 0.2 % proof stress and the tensile strength are similar for both investigated alloys, as can be seen in fig. 2 a and b. A large impact of the Ar sputtering pressure on the breaking elongation can be seen regardless of the investigated alloy. The samples deposited at 2.3×10^{-3} mbar show a ductile behaviour, whereas the samples deposited at 2.0×10^{-2} mbar exhibit a brittle fracture.

Conclusions

The results of our work show that the mechanical behaviour of thin film WE alloys can easily be influenced by a variation of the sputtering conditions without a negative impact on the corrosion rate.

This finding can be explained by the different microstructures of the deposited films originating in the different sputter parameters used in this study. The higher the kinetic energy of the sputtered particles the more diffusion can take place and the more ductile is the resulting film. In all cases the kinetic energy was not high enough to enhance the formation of precipitates, what explains the fact that the corrosion rate does not vary upon the change of deposition parameters.

Acknowledgement

The authors want to thank the German Research Foundation (DFG) for the financial support.

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