

Influence of ion energy on morphology and corrosion properties of Mg alloys formed by energetic PVD processes

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Abstract

Different PVD processes – magnetron sputtering, ion beam sputtering and vacuum arc deposition – are used to produce novel corrosion resistant Mg alloys with the average energy per deposited atom increasing from 5–10 to 15–45 eV. Correspondingly, a transition from a 3-dimensional columnar growth regime towards a layer-by-layer growth at increased energies was observed with the film texture changing from a weak basal texture towards a highly oriented film with the *c*-axis normal to the surface. Additionally, a higher energy helped stabilizing supersaturation, especially for Mg–Ti alloys. However, only a weak influence of the morphology on the corrosion rates was observed, which were mainly governed by the chemical composition of the films.

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1. Introduction

Physical vapour deposition (PVD) is an established technology to obtain individually tailored surface coatings on various substrates. One of the most important parameters is the average energy per incoming particle where higher values lead to an increased surface mobility [1,2]. Additionally, changes in the surface morphology and texture are observed with higher energies leading to films with larger grains and less defects while the momentum of the incoming particles can align or orientate the growing crystallites [3,4].

Magnesium is an attractive material for transport applications as automotive and aerospace, primarily of its light weight [5,6]. Mg alloys have the highest strength-to-weight-ratio of all the structural metals [7,8]. However, several drawbacks, among them a relatively poor corrosion resistance, restrict the application of unprotected magnesium

alloys [9,10]. Consequently, corrosion protection is of great importance. In this presentation, the influence of PVD process parameters on selected binary and ternary Mg-based corrosion protective coatings are investigated. The investigated PVD processes include magnetron sputtering (MS), ion beam sputtering (IBS) and vacuum arc deposition (VAD).

2. Experiment

Table 1 shows a summary of the different PVD processes used for these investigations. Magnetron sputtering sources were used to prepare pure Mg-films with a thickness of about 3 μm, at a source power of about 160 W and an argon gas pressure of 0.2 Pa. The base pressure of the ultra-high vacuum chamber was below 10⁻⁵ Pa. The sputtering gas used was high purity argon gas (7.0). The samples were coated under an angle of about 25° to the substrate normal. The target-to-source distance was about 15 cm, resulting in a deposition rate of approximately 3–4 μm/h at average particle energies of 5–10 eV.

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Table 1
Deposition process parameters for the different PVD processes

	Magnetron sputtering	Ion beam sputtering	IBS + Arc	Vacuum Arc
Ion source/targets	1 Magnetron source: Mg + alloy targets	1 Ion source: Mg + alloys & composite targets	2 Ion sources: alloy target + Ti cathode	1 Ion source: Mg or Ti cathode
Process parameters	Ar+ ions Power: 160 W Pressure : 0.2 Pa	Ar+ ions Energy: 0.8–1.2 kV Current: 30–34 mA		Current: 100 A Voltage: 15–25 V
Average energy (eV)	5–10	8–15	10–25	15–45
Deposition rate ($\mu\text{m/h}$)	3–5	1–3 (–10)	1–3 (–10)	1–3 (–10)

The ion beam sputtering experiments were performed in an UHV chamber using Argon ions from 800 to 1200 eV [11]. The angle between the ion source and the substrate normal was 45° with the target normal dividing this angle into two equal parts. The respective distances were 15 cm. Beside sputtered atoms with a kinetic energy between 1 and 5 eV, high energy backscattered Ar ions were impinging on the substrate during the film deposition, yielding an average ion energy of 8–15 eV per deposited particle. VAD was either used in conjunction with IBS or as a stand-alone process. At a total current of 100 A, an ion current of about 10 could be extracted from the cathode. Using appropriate filtering techniques, macroparticles emitted from the cathode were suppressed at the expense of a reduced ion current. Average energies of 15–45 eV were typical for the stand-alone mode while values of 10–25 eV can be estimated for the combination with IBS, depending on the relative ion current densities.

Beside commercially pure Mg (Mg 99.93 wt.%, impurities: Ni, Cu, Fe, Be and Si in the ppm range), the alloy systems Mg–Al, Mg–Si, Mg–Sn, Mg–Ti, Mg–RE (Rare Earth: La, Ce, Pr, Nd), Mg–Ti–Al and Mg–Zr–Al were used as starting materials to obtain coatings on Si and Mg alloy substrates.

Scanning electron microscopy (SEM) was performed to analyze the morphology while X-ray diffraction (XRD) measurements were used to identify the phase components. Additionally, ion beam methods – Rutherford backscattering spectroscopy (RBS) and elastic recoil detection analysis (ERDA) – were employed to obtain qualitative and quantitative information on the composition of the films. Information about the general corrosion properties of the coating systems and the as-cast alloys were gathered with potentiodynamic polarization measurements. The specimens were tested in 0.5% NaCl solution at a pH of 11 using an exposed surface area of around 0.5 cm^2 .

3. Results and discussion

Except for a thin surface layer of less than 5 nm, which is enriched with oxygen, homogeneous films were obtained with ion beam analysis in all cases [12]. Bulk contaminations with C, H and O are below 0.5 at.%, comparable to

the purity of the sputter targets. Any influences of concentration gradients or contaminations on the microstructure and the corrosion can be excluded in the present experiments.

Fig. 1 shows SEM images of the microstructure of pure Mg coatings deposited using the three different methods. For the first two processes, the hexagonal structure is still visible, while smoother surfaces with fewer visible topological structures are formed for the latter process. The total layer thickness for all these samples was about $3 \mu\text{m}$ to eliminate influences of the layer thickness on the morphology. With increasing average ion energy from MS via IBS to VAD, a transition from columnar growth to layer-by-layer growth is observed, which is in agreement with theoretical considerations originally propagated by Thornton [13]. With increasing the deposited energy per particle, a higher (transient) mobility of the atoms impinging on the surface is obtained, thus increasing the apparent surface temperature. At the same deposition rate, the higher mobility will lead to a larger diffusivity, thus allowing a transport from the original arrival site.

Small influences of the total layer thickness on the morphology cannot be ruled out, however, using similar particle fluxes and deposition times should minimize such secondary effects. It is known that with increasing thickness, a reduction of the average crystallite size is observed while smaller deposition rates improve the crystalline quality [14]. Furthermore, recrystallisation effects and tilting of the crystallites by varying the incident angle of the energetic particles are reported in the literature [15,16]. Further investigations of these effects during deposition of Mg alloys are in progress and will be published separately.

For magnesium alloys also a significant influence of the chemical composition on the morphology was observed, with all alloying elements generally remaining in solid solution and promoting layer by layer growth already at lower ion energies. Only at very high contents of alloying elements (far beyond equilibrium solubility), decomposition of solid solution can occur and the morphology is influenced. This phase segregation will always lead to rougher surfaces and smaller crystallites.

The high degree of preferential orientation, improving with increasing energy which could be inferred from the

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