



Advanced characterization study of commercial conversion and electrocoating structures on magnesium alloys AZ31B and ZE10A



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ABSTRACT

The local metal-coating interface microstructure and chemistry formed on commercial magnesium alloys Mg–3Al–1Zn (AZ31B) and Mg–1Zn–0.25Zr–0.5Nd (ZE10A, ZEK100 type) were analyzed as-chemical conversion coated with a commercial hexafluoro-titanate/zirconate type + organic polymer based treatment (Bonderite® 5200) and a commercial hexafluoro-zirconate type + trivalent chromium Cr³⁺ type treatment (Surtec® 650), and after the same conversion coatings followed by electrocoating with an epoxy based coating, Cathoguard® 525. Characterization techniques included scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS), and cross-section scanning transmission electron microscopy (STEM). Corrosion behavior was assessed in room temperature saturated aqueous Mg(OH)₂ solution with 1 wt.% NaCl. The goal of the effort was to assess the degree to which substrate alloy additions become enriched in the conversion coating, and how the conversion coating was impacted by subsequent electrocoating. Key findings included the enrichment of Al from AZ31B and Zr from ZE10A, respectively, into the conversion coating, with moderate corrosion resistance benefits for AZ31B when Al was incorporated. Varying degrees of increased porosity and modification of the initial conversion coating chemistry at the metal-coating interface were observed after electrocoating. These changes were postulated to result in degraded electrocoating protectiveness. These observations highlight the challenges of coating Mg, and the need to tailor electrocoating in light of potential degradation of the initial as-conversion coated Mg alloy surface.

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1. Introduction

Magnesium and its alloys are of great interest for engineering applications ranging from functional uses such as biomedical implants to structural alloys to achieve automotive and aircraft vehicle light weighting [1–3]. In vehicle light weighting applications, the rapid corrosion of Mg, particularly when exposed to salt species under aqueous conditions, is a key issue [4–16]. Alloying generally results in only moderate improvement in the aqueous corrosion resistance of Mg. Therefore, coatings are typically used to provide corrosion protection for Mg components [17–21].

Coatings for Mg structural components generally utilize a multi-layer strategy, involving an initial cleaning step; a surface pre-treatment such as chemical or electrochemical conversion coatings, surface alloying, and anodization; a second coating layer such as electrocoatings, platings, powder coatings, and organic coatings; frequently followed by a final

layer of sealant and/or paint [17–41]. In single- and multi-layer coating concepts, the interface between the substrate alloy and the initial coating layer is critical to coating adherence and performance. Delamination or through attack in this region can allow the environment to access the underlying alloy and result in local corrosion and coating failure.

Chemical conversion coatings are frequently used industrially as the first coating step after surface cleaning due to their relatively low cost and ease of implementation [17,19,29,30,33,38]. They are primarily used to provide a receptive surface for subsequent coating layer step adherence, but can also contribute to overall corrosion resistance of the multi-layer coating system [17,19,23,26]. Chromate conversion coatings provide the basis for good protection of Mg alloys, but result in significant safety and environmental issues related to hexavalent Cr, Cr⁶⁺ [17,19]. Alternatives to chromate conversion coatings for Mg alloys include phosphate-permanganates, fluorides, stannates, organics, vanadates, and rare earths [17,19].

Conversion coatings for Mg alloys have been the subject of intense study [e.g. reviews 17, 19]. Much of this work has emphasized coating processing development and subsequent corrosion resistance assessment. However, relatively few studies have pursued

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Table 1

Alloy chemical composition of AZ31B and E717 sheet determined by inductively coupled plasma atomic emission spectroscopy (ICP-AES) [after reference 16]. The E717 contains <0.5 wt.% Nd, the exact level of Nd is considered proprietary.

Alloy	Composition weight % (wt.%)									
	Mg	Al	Zn	Zr	Mn	Nd	Fe	Cu	Ni	
AZ31B sheet	95.47	3.05	1.00	<0.001	0.46	<0.001	0.001, 0.002	0.001	0.001	
E717 sheet	Bal.	0.004	1.49	0.31	0.008	<0.5	0.0007, 0.007, 0.008	0.001	0.001	

advanced microstructural characterization of the local chemistry, morphology, and structure of the coating and coating/substrate interface, e.g. for Mg substrates [42,43] and for Al substrates [44–46]. Such understanding is of particular importance for conversion coatings, as the resultant surface and its corrosion resistance are influenced not only by the coating solution chemistry and process conditions, but also by the substrate Mg alloy composition and microstructure [17,19,40].

The goal of the present work was to characterize the chemistry and morphology of several state-of-the-art Cr⁶⁺ free commercial conversion coatings for two representative Mg alloys of different composition classes using standard process conditions, and then after subsequent application of a commercial, state-of-the-art electrocoating. Such electrocoatings are one approach under consideration as the next protective layer above the conversion coating in multi-layered coating schemes used to protect Mg alloys [e.g. 22,23]. Conversion coatings and/or electrocoatings have been developed and successfully used to protect steels and aluminum, but are less well established for Mg alloys. Detailed knowledge of the chemistry and structure at the metal-coating interface as-conversion coated and after electrocoating is needed to provide baseline understanding for future coating development and optimization to protect Mg alloys.

2. Materials and methods

Two substrate alloys relevant to automotive applications were selected for study, AZ31B, a Mg–3Al–1Zn type, and Elektron® 717 (E717 for brevity), a ZE10A type rare earth and Zr modified Mg–1Zn–0.25Zr–<0.5Nd. These alloys are both near-single-phase, but do contain second phase Al–Mn (AZ31B) and Zn–Zr/Nd rich (E717) intermetallic

second phases [16,47–49]. The alloy test samples were electro-discharged machine (EDM) cut from wrought coil sheet ~1.5–1.6 mm thick obtained from Magnesium Elektron North America (MENA), Madison, IL USA. The E717 is of particular interest for automotive applications as it was recently demonstrated to be amenable to warm forming of door panels [50]. Analyzed compositions are shown in Table 1 [16]. (It should be noted that some scatter in the range of Fe impurity level was found in the E717 sheet material [16]. Data for Fe levels from 2 different measurements for AZ31B and 3 different measurements for E717 are shown in Table 1. Note that the test samples were polished to remove all residual surface finish prior to compositional analysis, as steel processing rolls can impart Fe impurities to the sheet surface during manufacture [16]. Such surface contamination does not appear to be the source of the variation in Fe level detected for the E717 sheet [16].

Bare (uncoated) AZ31B and E717 for corrosion studies were prepared as 15 mm diameter disks to a 1200 grit surface finish by wet grinding with SiC paper (Buehler, 41 Waukegan Road, Lake Bluff, Illinois 60044 USA), cleaned with acetone and deionized water, and dried with an air stream. Samples were then stored in a desiccator for at least 24 h prior to the corrosion exposures. Samples for coating were EDM cut to 15 mm diameter disks or 25 mm × 25 mm squares from the as-received sheet (no surface grinding). Two commercially available, state-of-the-art conversion coatings were selected for study: Bonderite® 5200 (chromium free hexafluoro-titanate/zirconate type + organic polymer based [26,28,45,51] (Henkel Corporation, North America, One Henkel Way, Rocky Hill, CT 06067 USA) and Surtec® 650 (hexafluoro-zirconate type with addition of trivalent chromium Cr³⁺ [46] (Surtec International, Neuhofstraße 9, 64625 Bensheim Germany). Additional coated samples were made after the same conversion coating treatments followed by electrocoating with Cathoguard® 525 (epoxy based) (BASF Corporation 26701 Telegraph Rd, Southfield, MI 48033 USA).

The conversion coatings were processed for AZ31B and E717 substrates by MENA, and by a commercial vendor for the subsequent electrocoat layer according to standard industry practices. The test samples for coating were in the as-mill received sheet state. Surfaces for conversion coating were prepared by cleaning in 10% nitric acid, followed by purified water rinse. The Bonderite® 5200 treatment was prepared using the commercially procured coating solution, diluted to 5% as directed, with sample immersion for 2 min at ~21 °C, purified

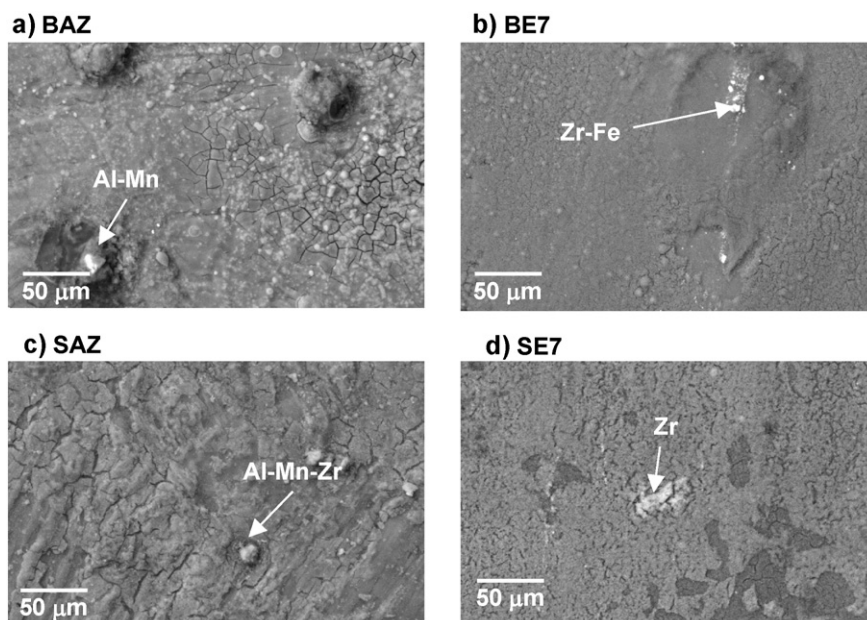


Fig. 1. Backscatter mode SEM images of the as-conversion coated surfaces formed on AZ31B and E717.

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