Fatigue Behavior of the Rare Earth Rich EV31A Mg Alloy: Influence of Plasma Electrolytic Oxidation

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Abstract: Rare earth rich magnesium alloys are used in aerospace and automotive fields because of their high specific strength and good castability. However, due to their low corrosion resistance, protective surface treatments, such as conversion coating or electroless plating are necessary, when they have to be used in humid or corrosive environments. The present study was aimed to evaluate the effect of Plasma Electrolytic Oxidation (PEO) and different surface roughness on the rotating bending fatigue of an innovative Mg alloy, with a high content of Nd (up to 3.1 wt%) and Gd (up to 1.7 wt %). Fatigue tests revealed a 15% decrease in the fatigue strength of the PEO treated alloy with respect to the bare alloy, probably due to the residual tensile stresses induced by the treatment. The effect of surface roughness on the bare alloy was, instead, negligible. The mechanisms of crack initiation were similar in the untreated and PEO treated alloy, with crack nucleation sites located in correspondence of large facets of the cleavage planes.

Keywords: rare earth rich magnesium alloy; Plasma Electrolytic Oxidation; PEO; microstructure; fatigue

1. Introduction

Due to their low density, high specific strength, good vibrational absorption and good castability [1–4], Mg alloys are increasingly used in aerospace and automotive fields. Structural components for the transport industry, such as camshaft covers, clutch and transmission housings, intake manifolds, engine blocks, steering components, radiator supports, and automobile wheels, are inevitably subjected to cyclic stresses in service. For this reason, investigation on the fatigue behaviour of both cast and wrought Mg alloys has recently drawn considerable interest [5–9]. Recent studies [7,9,10] have demonstrated that the addition of rare earth (RE) elements, such as Nd, Gd, Y, can remarkably enhance both the tensile and fatigue strength of Mg alloys. In the case of castings, this improvement was related both to the microstructural refinement and to the decreased size and amount of porosity and inclusions induced by RE elements [7,11,12], while in wrought Mg alloys it seems mainly due to the corresponding weakness of crystallographic textures and the reduction of twinning [13–16]. It was also found that the addition of RE and the reduction of impurities can effectively improve the corrosion resistance of Mg alloys [17–20].

However, when components have to be used in humid or corrosive environments and/or improved wear resistance is needed, also Mg-RE alloys can be inadequate and protective surface treatments, such as conversion coating or electroplating/electroless plating are necessary [21–23].

Among the conversion coating processes, plasma electrolytic oxidation (PEO) is gaining increasing attention, since it is able to produce relatively thick, dense and protective surface oxide layers with enhanced wear and corrosion resistance [24–26]. Moreover, the lower residual surface stresses and amount of microstructural defects in the conversion layer, with respect to traditional oxide coating, seem to reduce its adverse effect on the fatigue properties of Mg alloys.

To date, however, few data on the fatigue behaviour of RE-containing cast Mg alloys are available [6,12,27–29] and also the effect of anodisation or PEO has been mainly investigated on traditional wrought Mg-alloys without RE addition [21–23,30].

Based on the above, the present study was aimed to evaluate the rotating bending fatigue behaviour of an innovative casting Mg alloy, with a high content of Nd (up to 3.1 wt%) and Gd (up to 1.7 wt%). The excellent fluidity of this alloy, thanks to the high amount of Nd, enables the production of sand cast components, with very low defect content and an average grain size comparable to that of wrought alloys. The effect of different surface roughness on the untreated alloy, as well as of surface modification by Plasma Electrolytic Oxidation (PEO), was evaluated.

2. Materials and Methods

The RE-rich magnesium alloy used in the present study was provided by Magnesium Elektron® and its chemical composition is reported in Table 1.

Table 1. Chemical composition (wt%) of Elektron 21® (EV31A) magnesium alloy. (Magnesium Elektron® test report).

Nd	Gd	Zr	Zn	Cu	Fe	Ni	Si	Mg
2.8	1.5	0.53	0.25	<0.008	<0.003	<0.001	<0.01	Bal.

Fatigue test samples were machined from sand castings to the final geometry reported in Fig. 1 according to ISO-1143 [31] and then were T6 heat treated according to standard conditions (solutionising at 520°C for 8 h; quenching in water at 70°C; and aging at 200°C for 16 h).
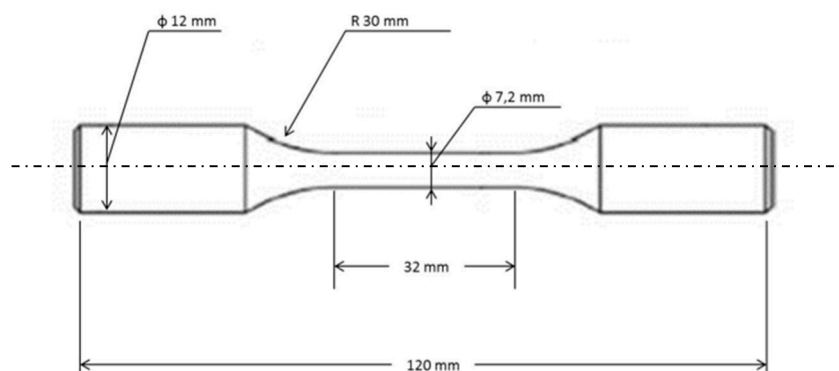
Figure 1. Geometry of rotating bending fatigue specimens

Subsequently, they were finished with emery papers in order to achieve two different surface roughness values, Ra=0.6÷0.8 μm (samples abbreviation S0.8) and Ra=0.2÷0.3 μm (samples abbreviation S0.3). A set of T6 heat-treated specimens was surface treated by Plasma Electrolytic Oxidation (PEO) at AHC® (Germany), using a proprietary production process (MAGOXID-COAT®). After PEO, they were sandblasted to a final surface roughness Ra=1.9÷2.1 μm (sample abbreviation SP). Surface roughness was checked by a stylus profilometer (tip radius: 5 μm).

The microstructure of the base and PEO treated alloy, both in surface and cross-section, was analysed by optical microscopy (OM) and Scanning Electron Microscopy (SEM) Zeiss EVO® equipped with an Energy Dispersive Spectroscopy (EDS) (Oxford INCA 350) microprobe.

Microstructural characterisation was carried out on samples prepared by standard metallographic techniques (grinding with SiC emery papers 800, 1200, 2000 grit; polishing with diamond 9, 3, 1 μm) and chemically etched with Nital 2 (HNO3 2% vol. with ethanol). Image

analyses on the optical micrographs (OM) were carried out using the ®Image Pro-Plus software and grain size was evaluated using the linear intercept method, according to ASTM E112-10 [32].

The adhesion/cohesion of the PEO layer was evaluated by the scratch test method, using a Revetest system (CSM Instruments SA, Switzerland) equipped with a diamond indenter (200 µm diameter). The scratch indentations were carried out using a linear progressive load mode, from 1 to 20 N, with a linear speed rate of 10 mm/min-1 and for a scratch length of 6 mm. The testing conditions were set based on experimental trials.

Rotating bending fatigue tests were carried out both on the untreated specimens with different surface roughness (S0.3 and S0.8 series), and on the PEO treated ones (SP series), according to UNI 3964 [33] using the staircase method, at a frequency of 90 Hz and at a stress ratio R=-1. The maximum number of cycles was set to 2x106 cycles. To relate the different surface conditions to the fatigue behaviour, SEM analyses of the fracture surfaces were carried out.

3. Results and discussion

3.1 Microstructure of the untreated alloy

OM and SEM images of the EV31A-T6 alloy are reported in Fig. 2. The microstructure is composed of equiaxed grains with an average size of 45 ± 2 µm (Fig. 2a). The T6 heat treatment did not completely dissolve the ternary eutectic compound Mg12NdxGd(1-x) located at the grain boundaries, as highlighted in Fig. 2b and SEM-EDS of Fig. 2c. Moreover, clusters of nanometric and micrometric Zr-Zn precipitates are clearly visible at the grain centre, as shown by SEM-EDS analysis reported in Fig. 3. A more detailed microstructural investigation on the strengthening precipitates, formed by the aging treatment, was reported by the authors in [34].
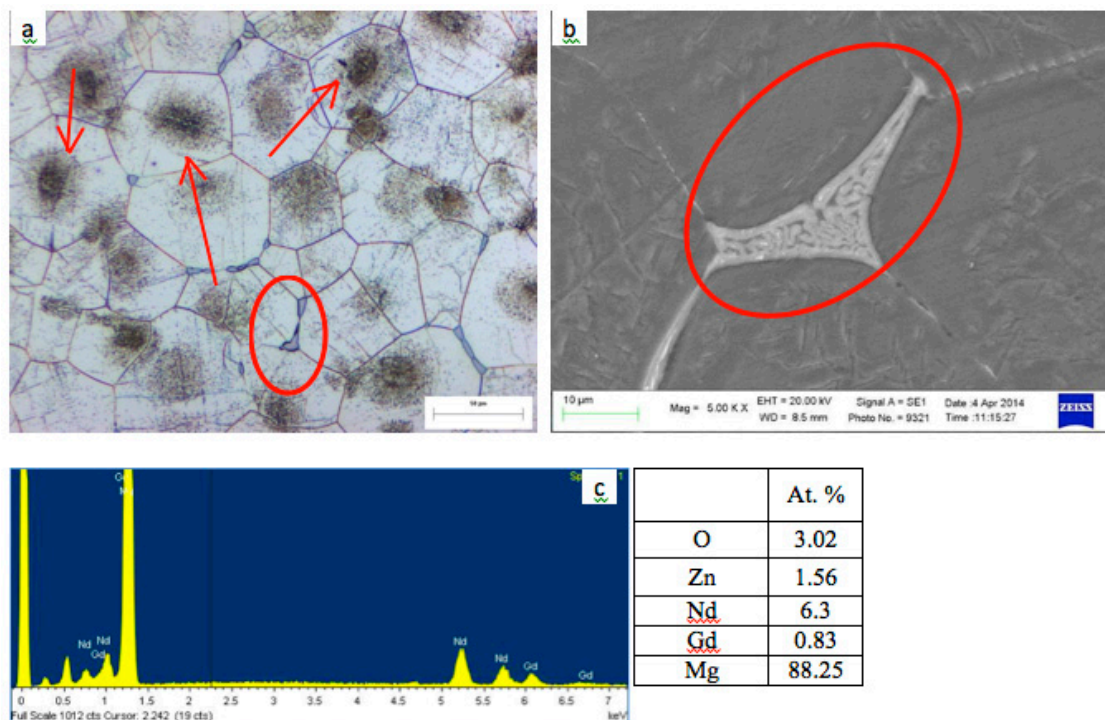
Figure 2. OM image EV31A etched with Nital2. Zr-Zn cluster and eutectic compounds highlighted respectively by red arrows and a red circles (a) and SEM image of the eutectic compound at the grain boundaries (b) with corresponding EDS analysis (c).

	At. %				
	Zn	Zr	Nd	Gd	Mg
Sp.1	0.00	0.00	0.50	0.17	99.34
Sp.2	0.00	0.34	0.47	0.21	98.98
Sp.3	0.68	1.72	0.49	0.17	96.94
Sp.4	0.35	2.97	0.47	0.00	96.21
Sp.5	0.73	1.79	0.44	0.00	97.04
Sp.6	0.00	0.33	0.53	0.24	98.90
Sp.7	0.00	0.00	0.48	0.14	99.37
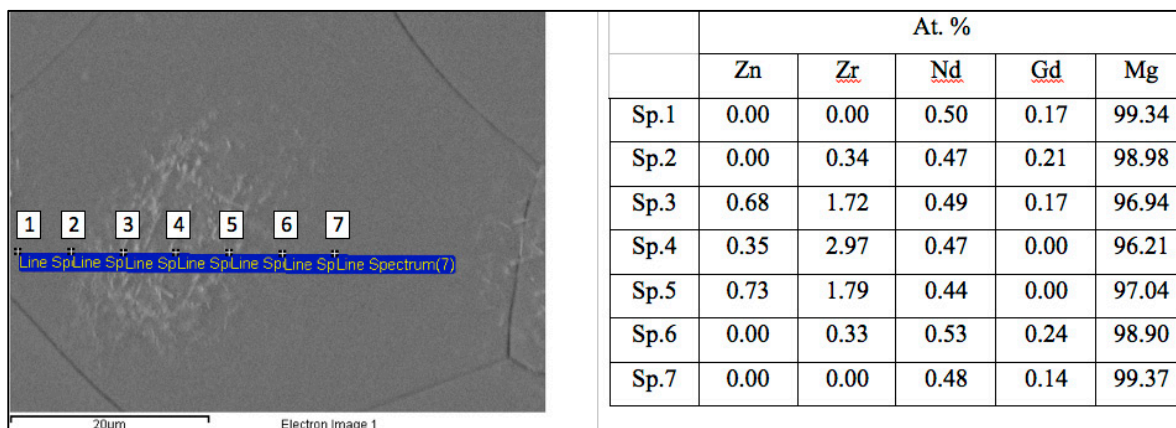
Figure 3. SEM image and EDS analyses of Zr-Zn intermetallic precipitates at the grain interior

No solidification defects, such as gas and shrinkage porosities or oxides, which can adversely affect the fatigue behaviour of the alloy have been observed. This can be related both to the increased castability of the EV31A alloy, induced by the presence of RE, and to the optimised sand casting process used for samples production [35,36].

3.2 Microstructure of the PEO-treated alloy

The PEO process was carried out by AHC® (MAGOXID-COAT®) in a saline electrolyte solution. During the process, a high voltage between the anode and the sample was generated, inducing micro-discharges on the specimen surface and converting the alloy surface into a hard oxide layer. PEO layers on Mg alloys can be usually divided into two or three regions, according to the operative treatment conditions: (i) a thin barrier layer (generally hundreds of nanometres) at the interface with the base material; (ii) a compact intermediate inner layer, with a small amount of porosities and cavities; (iii) an outer region with a high number of pores and crater density [37–39].

SEM images on the outer surface of the PEO treated and sandblasted fatigue samples are shown in Fig. 4. The sandblast process led to a relatively smooth surface, with a significant reduction of the typical cracks and microcracks present on the outer surface of the PEO layer. However, some small cracks through the pores are still present, as shown at higher magnification in Fig. 4-b.
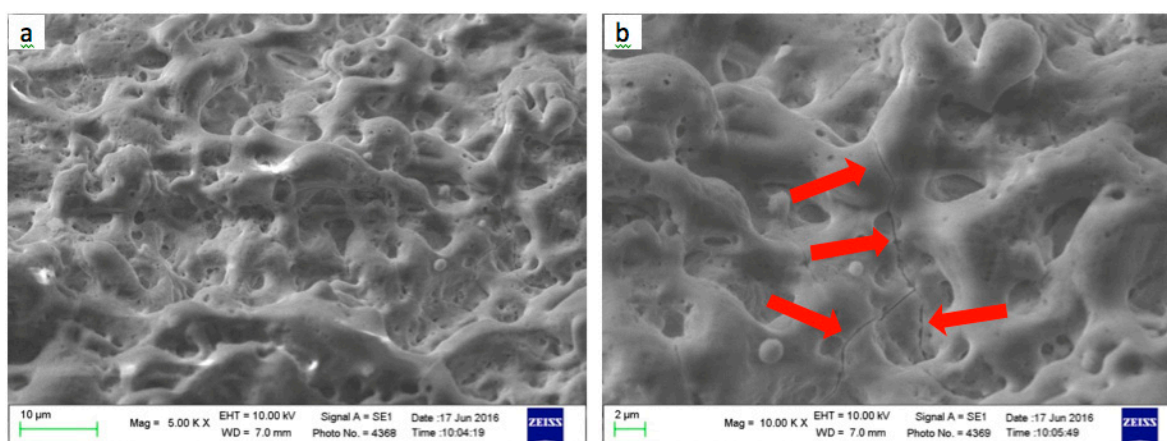
Figure 4. SEM images at different magnification of the surface of PEO treated and sandblasted fatigue specimens: smooth surface (a) with very few small cracks through the pores (b).

Representative SEM micrographs of the cross-section of the PEO-treated alloy are reported in Fig. 5 a-b. The conversion layer was about 23±3 µm in thickness and appears uniform and compact, with low density of pores and cracks, thus confirming that the sandblasting process was effective in removing the outer region of the PEO layer.
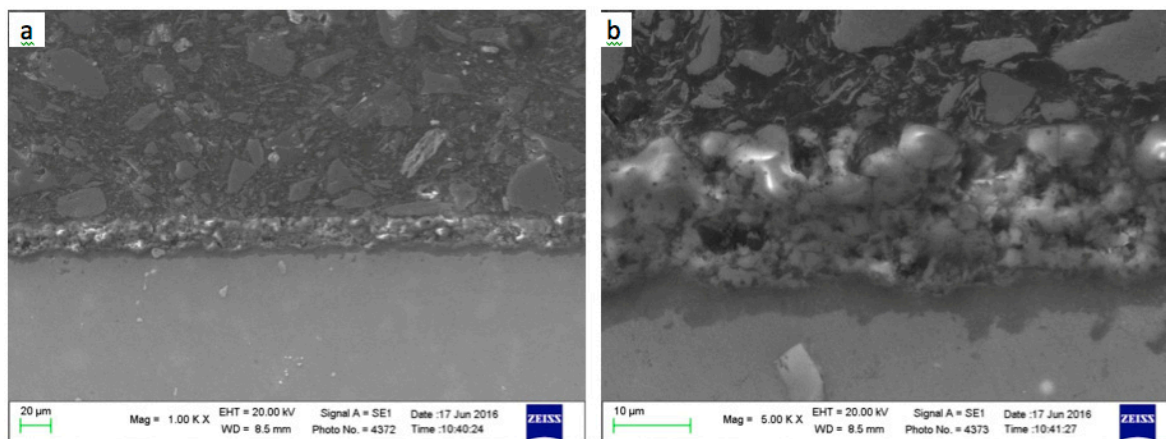
Figure 5. SEM images of PEO layer cross-section.

SEM-EDS element maps on the cross-section, shown in Fig 6, revealed a homogenous distribution of phosphorous, potassium and oxygen due to the phosphate, silicate and potassium fluorides present in the electrolytic bath. A thin fluorine layer in the substrate material, underneath the PEO layer, can also be detected.
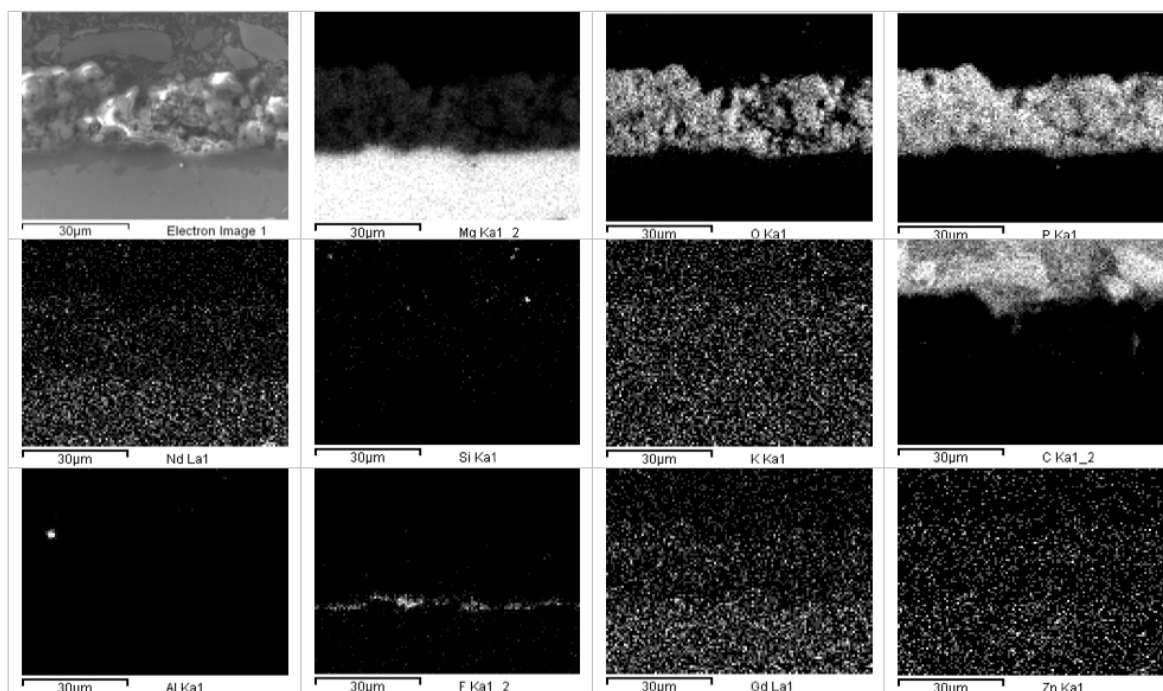
Figure 6. SEM-EDS cross section maps of the PEO layer.

This layer plays an important role during the PEO process, reducing electrolyte conductivity, thus decreasing the working voltage. Moreover, the fluorine addition helps obtain a more compact layer, reducing porosity and correspondingly improving mechanical performance, tribological behaviour and corrosion resistance [40].

3.3. Adhesion of the coating

PEO treatment on Mg alloys could be classified as a hard coating on a softer substrate material [41]. Scratch testing is a commonly used method to evaluate the adhesion of thin hard coatings. Failure depends on both substrate and coating properties, namely hardness and microstructural characteristics. A representative image of a scratch test performed on the PEO treated EV31A alloy is

shown in Fig. 7a at a relatively low magnification, whereas more detailed images of the failure induced by the scratches are reported in Fig. 7b.
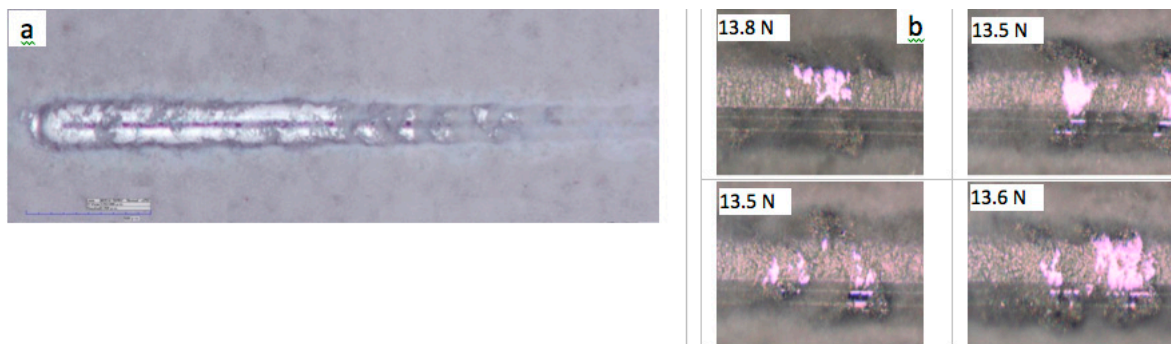
Figure 7. OM scratch test image (a); OM images of the failure area for different scratch tests with corresponding failure load (b).

Reproducible results, in terms of failure mode and critical load, were obtained. SEM images at different magnifications of a representative scratch are shown in Fig. 8 a-b, with corresponding EDS X-ray maps. Buckling and spallation failure are both present in Fig. 8 a-b, where coating detachment from the substrate can be clearly seen.
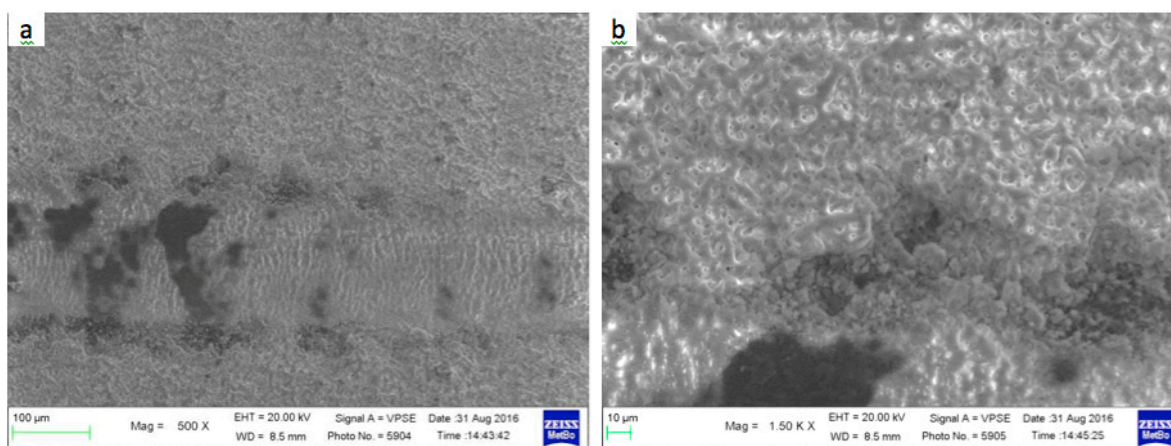
Figure 8. SEM images of the scratch test track at different magnification.
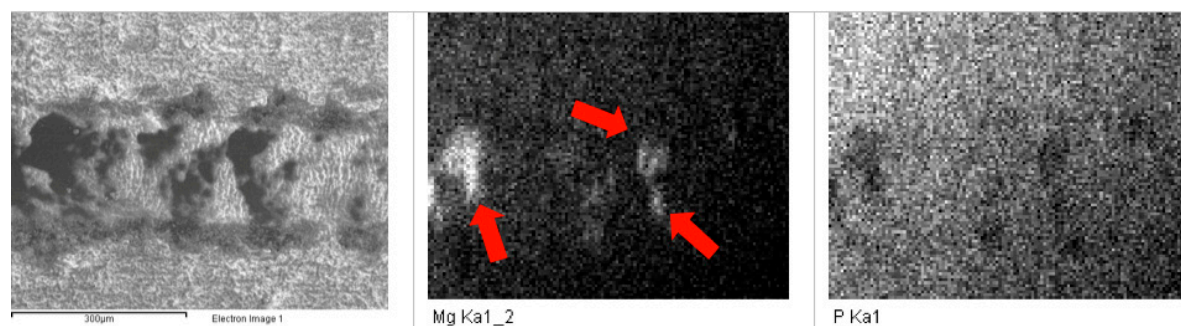
Figure 9. Representative SEM-EDS maps of the scratch tests, with the substrate highlighted by red arrows.

The EDS maps confirmed adhesion failure of the PEO coating, since the Mg substrate was detected at the bottom of the scratch, as shown in Fig. 9. Buckling failure occurs in response to the compressive stresses generated ahead of the moving indenter and typically occurs for a coating thickness less than 10 µm. Spallation occurs due to compressive shear stresses ahead of the indenter through the coating, resulting in crack nucleation that could propagate through the thickness. The

continued forward motion of the indenter leads to an interfacial crack and consequent coating failure [41]. Also in this case, where the PEO layer is about 20 μm and the EV31A substrate is relatively soft, buckling and spallation are the main failure modes.

3.4 Fatigue tests

The results of the rotating bending fatigue tests carried out on the untreated sand cast EV31A Mg alloy with different surface roughness (S0.8 and S0.3 samples), and on the PEO-treated alloy are reported in Tab. 2. It can be noted that the effect of surface roughness on the base alloy was negligible, since the difference in fatigue strength between the S0.8 and S0.3 samples was within the standard deviation (103 and 101 MPa, respectively). The investigated alloy displayed a fatigue behaviour comparable or even better than that of other rare earth rich sand cast magnesium alloys; in fact, a fatigue strength of 98 MPa was measured on the AM-SC1 alloy (Mg-Nd 1.7-Other RE 1.0-Zn 0.5 Zn-Zr 0.50) [42], while the NZ30K2 alloy (Mg-Nd 2.8-Zn 0.19-Zr 0.50) [12] showed a fatigue strength of 84 MPa (15% lower compared to that measured on the present alloy). It is also worth noting that the fatigue strength of the EV31A alloy used in the present study is comparable with that of the most common wrought magnesium alloy AZ31 [43].

Table 2. Results of the rotating bending fatigue tests.   S0.8 abbreviation for samples with Ra=0.6÷0.8, S0.3 abbreviation for samples with Ra=0.2÷0.3   m, PEO abbreviation for samples PEO-treated.

Sample series	S0.3	S0.8	PEO
$\sigma_{D[50\%]}$ [MPa]	101	103	88
SD [MPa]	12	17	13

The excellent fatigue response shown by the investigated alloy can be mainly ascribed to the soundness of the specimens, also due to a careful control of the sand casting process; in fact, no solidification defects were detected close to the free surface of the samples, which experiences the maximum stresses, or in the bulk.

Concerning, instead, the effect of plasma electrolytic oxidation on the fatigue performance of the EV31A Mg alloy, it can be noted that it induced a 15% reduction in fatigue strength (equal to 88 MPa for the PEO-treated samples). This reduction in fatigue strength is probably mainly related to the intrinsic defectiveness of the ceramic conversion layer and the residual tensile stresses [41]. The micro-discharges, in fact, cause local melting of the surface, inducing pores and microcracks, as well as tensile stresses, during the subsequent rapid solidification [24]. Similar results were also found by Yerokhin et al. [21] who reported a fatigue strength reduction in the range of 3–10% on PEO-treated magnesium alloy rods (Mg-Al 2-Zn 1-Mn 0.2) with respect to the bare material, while A. Nemcova et al. [23] showed a 40–50% reduction in fatigue strength of PEO treated wrought AZ61 Mg alloys with respect to the untreated alloy.

3.5 Fracture surfaces analysis

It is well known that in high cycle fatigue, the crack initiation period may take up to 90% of the total fatigue life of the material, thus particular attention has been given to the crack initiation mechanisms, both in the untreated and PEO-treated alloy.

A representative SEM micrograph, at relatively low magnification, of the fracture surface of the untreated alloy is reported in Fig. 10 a. The three typical regions of fatigue failure can be clearly identified: i) crack initiation; ii) crack growth; iii) final failure due to overloading.

In rotating bending fatigue tests, the surface experiences the maximum stresses, thus the cracks mainly nucleate from it. SEM image in Fig. 10 b shows, at a higher magnification, the crack initiation region in the untreated alloy, where it can be clearly seen that crack nucleation occurs at the isolated facets of cleavage planes, near the free surface of the specimen. The facets are flat and smooth,

resulting from cleavage induced by the limited number of operative slip systems characteristic of HCP Mg alloys, and appear surrounded by fatigue striations. Similar crack nucleation features were observed on other magnesium alloys by Z. Li et al. [27].
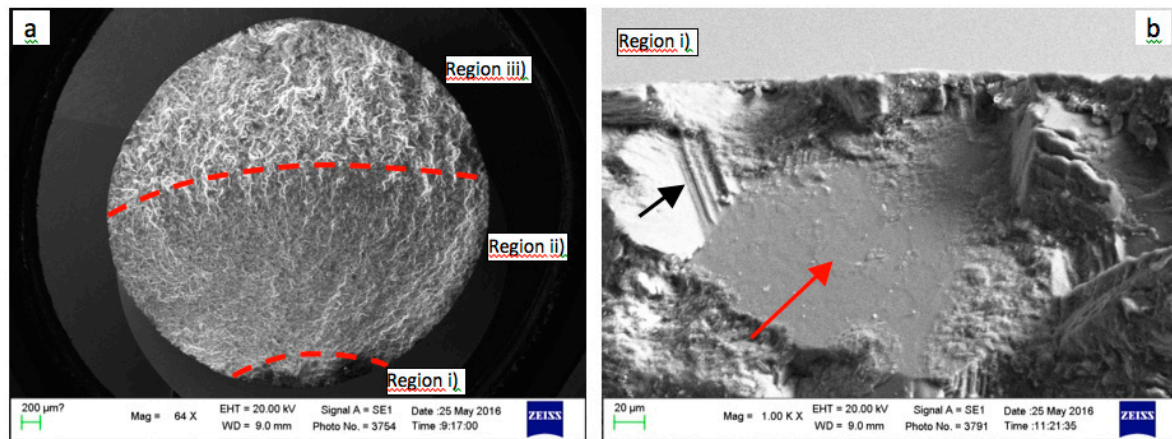
Figure 10. Representative SEM images of an untreated sample. Typical regions of fatigue failure at low magnification (a) and high magnification of crack initiation at isolated facets of cleavage planes (red arrow) surrounded by fatigue striations (black arrows) (b).

The facet cleavage is due to the development of slip bands during the cycling loading, acting as stress concentration sites; thus, once the damage takes place, the crack could quickly propagate. Cleavage was observed in a large grain with a size of about 200 μm, the average grain size of the alloy being 45 ± 2 μm. Coarse grains, in fact, can easily form longer initial cracks, due to a large number of dislocations and defects, resulting in a higher probability of localised damage under cyclic loading compared to fine grains.

The PEO-treated samples showed the same crack nucleation mechanisms, as can be seen in Fig. 11 a-b, where cleavage facets and fatigue striations can be clearly identified.
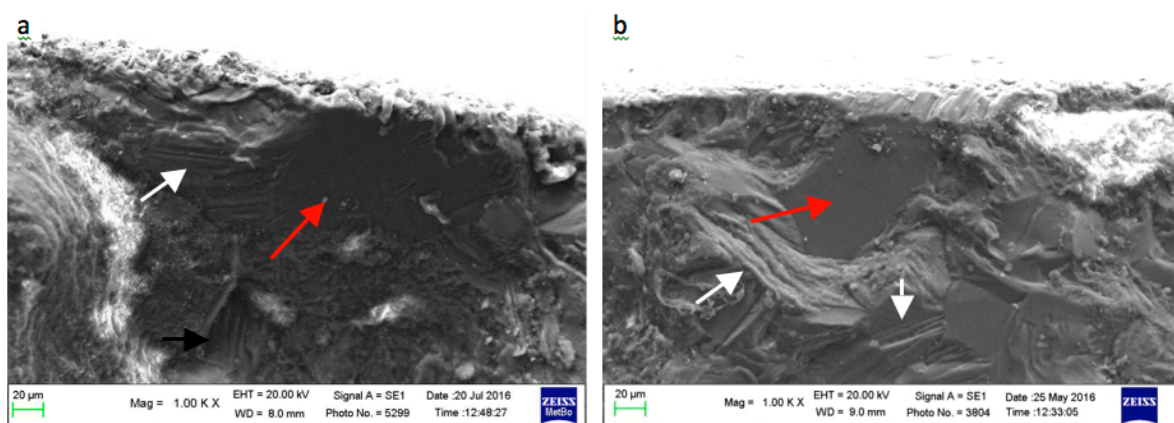
Figure 11. SEM images of crack initiation at isolated facets of cleavage planes (red arrow) surrounded by fatigue striations (white arrows) of two different PEO treated samples.

It can therefore be inferred that the lower fatigue strength of PEO treated samples is mainly due to the residual tensile stresses induced by the treatment, rather than to the defects present in the conversion layer. As previously reported, the presence of fluorine in the electrolytic bath and the sandblast process, in fact, reduced the typical solidification defects in the conversion layer (section 3.2) and consequently their influence on the fatigue behaviour of the alloy.

Representative SEM images of the crack growth region for the PEO treated and untreated samples are, instead, reported in Fig 12 a-b, showing the presence of classic fatigue striations. In Mg

alloys their formation is generally associated with the dislocation slip and twinning mechanisms in the plastic zone ahead of the fatigue crack tip [4,6,44].
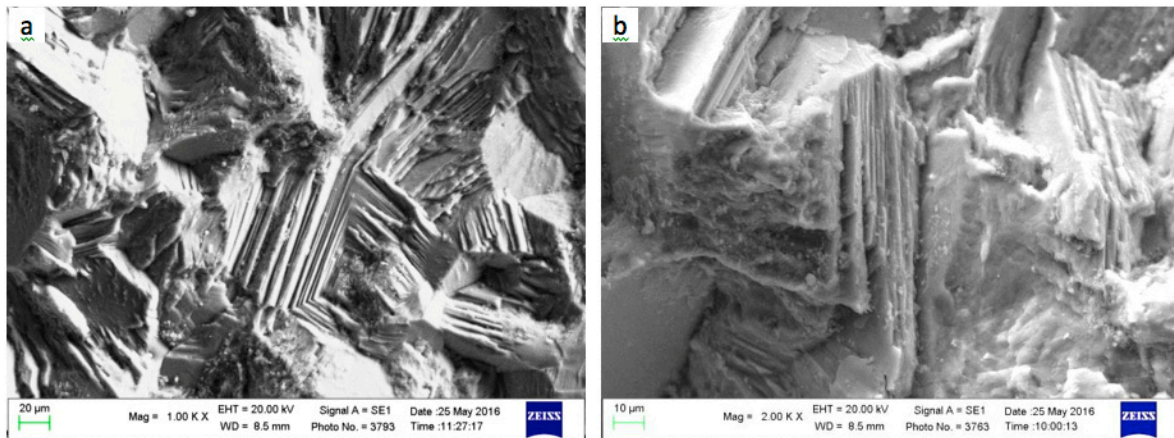
Figure 12. Representative SEM images of the crack growth region for the PEO treated (a) and untreated (b) samples

In this region also secondary transgranular cracks are clearly visible, as shown by representative OM images on longitudinal sections of fatigue samples (Fig. 13 a); they originated from the main cracks, confirming a transgranular and intergranular mixed mode of crack propagation. Slip bands can be clearly seen inside the grains (Fig 13 b), confirming the previously discussed mechanism for fatigue striations formation.
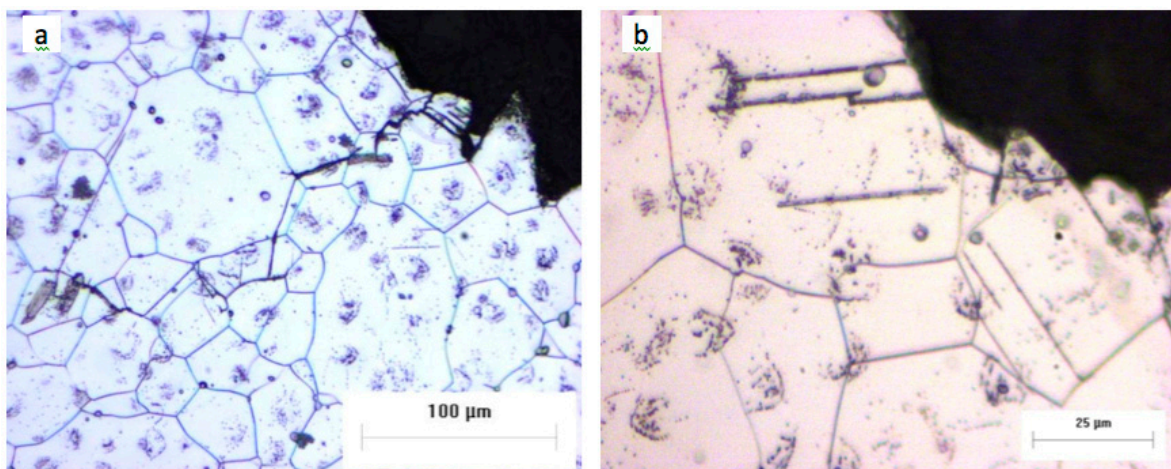
Figure 13. Representative OM images of longitudinal sections (parallel to the gauge length), of fatigue samples: secondary transgranular cracks (a) and slip bands inside the grains (b).

Finally, representative SEM images of the overloading regions are reported in Fig 14, where a typical brittle morphology, with cleavage planes and secondary cracks, can be observed.
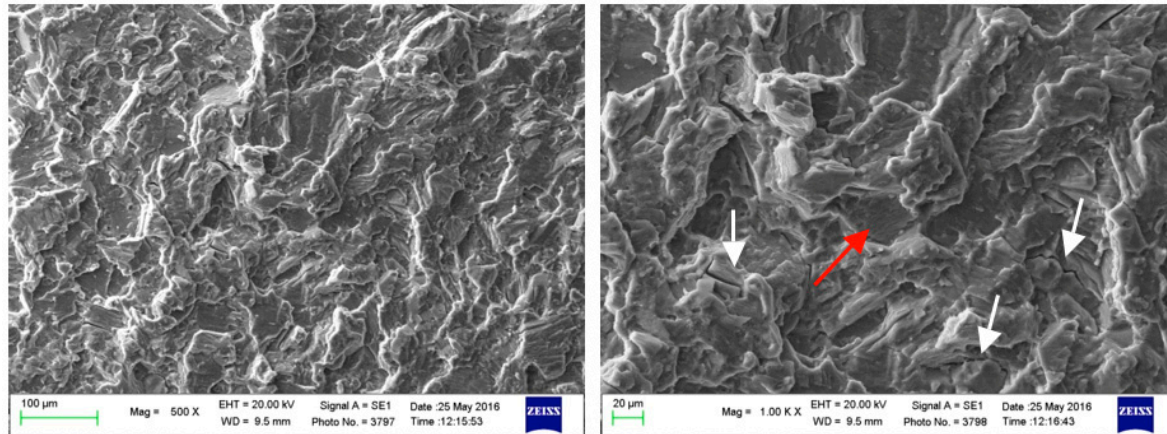
Figure 14. Representative SEM images at of the overloading area at low magnification (a) and high magnification with cleavage planes (red arrow) and secondary microcracks (white arrows) (b).

4. Conclusions

The effect of surface roughness and plasma electrolytic oxidation on the fatigue behaviour of the innovative rare-earth rich sand cast EV31A Mg alloy was investigated.

The fatigue strength of the untreated alloy was comparable with that of wrought Mg alloy, probably due to its chemical composition and also to the high quality of the cast, substantially free from solidification defects due to a well-controlled sand casting process. The effect of specimen surface roughness was negligible.

The PEO treatment caused no more than 15% reduction in the alloy fatigue strength. The relatively low reduction in fatigue strength was related to the compact and well adherent conversion layer. A positive effect on its fatigue behaviour was probably also induced by the sandblasting process, carried out on the PEO-treated specimens, that removed the outer layer with the largest pores, channels and craters, typically induced by plasma electrolytic oxidation.

Similar mechanisms of fatigue failure were observed in base and PEO-treated alloys, with crack nucleation sites located close to the large facets of the cleavage planes. This allows us to suppose that the observed reduction in fatigue performance was mainly induced by the residual tensile stresses due to PEO treatment in the substrate material, rather than to the defects present in the conversion layer.

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