

## The Immersion Corrosion Resistance of Shot Peening and MAO Applied on AZ31–0.5% Nd Sheets

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### Abstract

The effect of shot peening (SP) and Micro Arc Oxidation (MAO) were utilized to characterize the performance of the corrosion resistance process at 0.5 % Nt with add AZ31 Mg alloy. Hot rolling at different rolling speeds has been investigated in this work. The surface of the rolling materials a rolling speed of 4.7 m/min and the rolling materials a rolling speed of 10 m/min were measured to create a clear definition of corrosion resistance physical properties. Experimental results show the surface of the rolling materials a rolling speed of 4.7 m/min had higher surface smoothness values than the one of the rolling at 10 m/min. It was observed that the corrosion rate changed in the first 24 of 168 hours. In the following hours, the corrosion rate showed different results according to the initial microstructure properties of the base materials. Initially, pore size was the dominant factor determining corrosion resistance, although, after coating the corrosion rate was affected by the twins formed, based on rolling speed, which enhanced the corrosion rate between 24 and 168 hours.

**Keyword:** Shot peening; micro arc oxidation; AZ31; Nd; hot rolling; corrosion resistance.

## مقاومة غمر التآكل عند الصقل بالخرق وتطبيق الاكسدة الدقيقة عند 0.5% من النيتروجين مع اضافة سبيكة الماغنيسيوم AZ31 Mg

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### الملخص

في هذا للبحث تم استخدام تأثير الصقل بالخرق (SP) وأكسدة القوس الدقيقة (MAO) لتوصيف أداء عملية مقاومة التآكل عند 0.5% من النيتروجين مع إضافة سبيكة AZ31 Mg. تم فحص الدرفلة على الساخن بسرعات درفلة مختلفة في هذا العمل. وكذلك تم قياس اسطح مواد الدرفلة بسرعة درفلة 4.7 م/دقيقة ومواد الدرفلة بسرعة درفلة 10 م/دقيقة لوضع تعريف واضح للخصائص الفيزيائية لمقاومة التآكل. أظهرت وجود نتائج مختلفة عند أسطح المعادن عند السرعات المختلفة. وكانت النتائج التجريبية على أسطح مواد الدرفلة بسرعة درفلة 4.7 م/دقيقة كان له قيم نعومة سطح أعلى من سطح الدرفلة بسرعة 10 م/دقيقة. لوحظ تغير معدل التآكل في أول 24 ساعة من 168 ساعة. في تلك الساعات، أظهر معدل التآكل نتائج مختلفة وفقاً لخصائص البنية المجهرية الأولية للمواد الأساسية. في البداية، كان حجم المسام هو العامل المهيمن الذي يحدد مقاومة التآكل، على الرغم من أن معدل التآكل تأثر بعد الطلاء بالتوائم المتكونة، بناءً على سرعة الدرفلة، مما عزز معدل التآكل بين 24 و168 ساعة.

**الكلمات المفتاحية:** الصقل بالخرق، الاكسدة الدقيقة، النيتروجين، مقامة التآكل، سبيكة الماغنيسيوم و الدرفلة على الساخن.

## I. INTRODUCTION

The material surface plays an important role in the response of the engineering components. Surfaces are frequently subjected to various surface treatment processes to achieve certain qualities that are not available from the primary manufacturing processes. The process is conducted for various reasons including improving the performance of materials, changing physical properties, varying appearance and altering dimensions. A diverse range of thermal, mechanical and chemical treatments has been developed to modify the surface characteristics. Various surface treatment processes have been used for a wide range of materials from semiconductors to metals, ceramics, polymers, bio and nonmaterial [1]. The quality and performance of a product are directly related to its surface integrity produced from different surface treatment processes. Surface integrity comprises the topography (e.g. roughness, erosion), the mechanical properties (e.g. residual stress, hardness), metallurgical states (e.g. phase transformation, microstructure) and other related property variations of the work material during surface processing procedures [2].

Mg alloys attract attention especially from the automotive and aerospace industries due to their low density and high strength [3]. HCP (hexagonal package structure) reduces the easy forming of Mg alloys and weakens the competitiveness of sheet Mg products [4]. It is known that the rare earth elements such as Nd, La, increase the formability of AZ31 Mg alloy [5]. On the other hand, the surface of Mg alloy required coating to improve the corrosion resistance are quite popular [6-7]. Moreover, it is desired to improve the mechanical properties of Mg alloys by creating a tension on the surface of the material by processes such as shot peening (SP) [8]. In addition, the micro arc oxidation (MAO) process improves corrosion resistance perfectly [9]. AZ31 Mg alloys that have been treated with only SP or MAO have been reported [6-8]. The aim of this study is to improve the corrosion resistance of AZ31 Mg alloy metal sheets by addition of Nd as well as SP and MAO applications. In addition, the effect of rolling speed and rolling parameters on the final result was investigated.

## II. EXPERIMENTAL STUDY

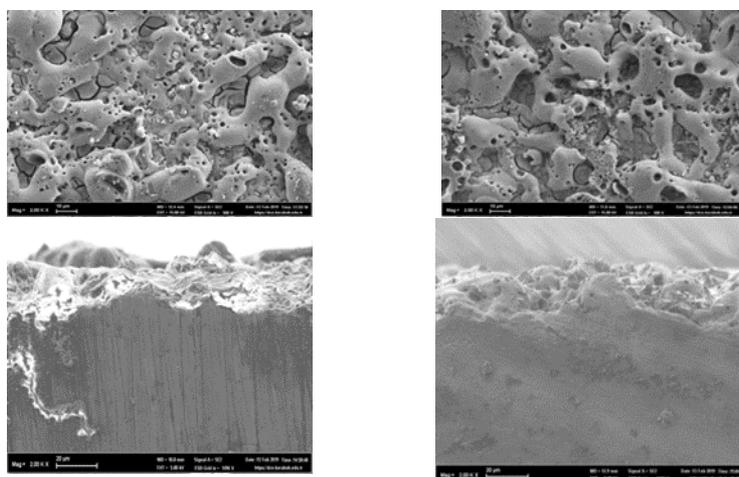
AZ31 Mg alloys containing 0.5 Nd (% wt.) were produced by using low pressure gravity die casting method. The composition of the produced material, determined by an XRF machine, was 2.93%wt Al, 1.03%wt Zn, 0.12%wt Mn, and 0.49%wt Nd. In an argon gas protected environment, pure Mg alloys were allowed to melt at 750°C for 1 hour and then the Nd-Mg master alloy was added to the ladle and alloyed. In SF<sub>6</sub>- CO<sub>2</sub> blended protective atmosphere, the alloy was transferred with 2atm pressure to a stainless steel 36x36x200mm mold, which was heated at 350°C. Billets with dimensions 36x12x80mm were cut from the billets left to cool in the molds to be used in the homogenization process before rolling. The surfaces of the billets were homogenized for 24 hours at 400°C and were sanded with 800 sanding sheets before rolling. Before rolling the specimens were heat treated at 400°C for 30min. The samples were rolled at 400°C. The thickness of billets reduced from 12mm to 2mm in 5 passes and they were allowed to cool in air after the last pass. The rolling parameters were 30% cross-sectional reduction per pass and the rolling speeds were 4.7m/min and 10m/min. The used rollers of the rolling stand were from stainless steel with a diameter of 110mm. No lubricant was used in the rolling process. The SP process was carried out for 20min with TAB company ball SP machine by using Z850 metal balls on the sheet materials, which were previously sanded with 1200 grit by giving maximum 8bar air pressure according to AISI 1070 standard. After the SP process, the surface of the samples was cleaned with alcohol and dried. Micro arc oxidation process followed: 25x25x12mm samples previously sanded up and cleaned with ultrasonic cleaning device were immersed in 4g/lit sodium silicate, 1g/lit potassium hydroxide and 3g/lit disodium hydrogen phosphate containing 4lit pure water medium under 85mA/cm<sup>2</sup> current density and 250Hz frequency for 5min. Immersion corrosion test in 3.5% NaCl solution was applied to the 2mm sheets for 168 hours. After the corrosion test, the corrosion waste of the samples was extracted using chromic acid solution and then dried with alcohol and the weight loss was measured using 0.0001 precision balance machine. Microstructure

images of the samples were taken before and after the corrosion test by SEM (Carl Zeiss Ultra plus Gemini Fesem). In addition, information was obtained about the secondary phases by using XRD (RIGAKU Ultima IV). The surface smoothness of the samples was measured according to the ISO 1997 standard (Mitutoya Surftest SV-2100M4).

### III. RESULTS AND DISCUSSION

#### A. Microstructure Results

As shown in Figure 1, the base metal of AZ31-0.5 Nd alloys rolled at 4.7m/min had twins dominated microstructure and bigger sized secondary phases shaped as globular, although the rolled at 10m/min speed specimen includes dynamic recrystallization grains and finer secondary phases distributed mostly continuously on grain boundaries.



(a)

(b)

Figure 1. SEM images of surface and cross-section of materials rolled at: (a) 4.7m/min, (b) 10m/min

The surface morphology and cross-sectional images of the samples after MAO treatment are different. However, the typical coating

properties of the MAO process were obtained, for example, from islets of different sizes and micropores of different diameters formed on these islets. Melting and rapid solidification during MAO ensure that the pores are circular. At a rolling speed of 4.7m/min, the extruded sample has smaller diameter micropores, which are denser in the islets. The rolled material at 10m/ min has larger pores with larger diameters, but they are distributed less on the islets.

The images taken show us that the rolled material at 4.7m/min is coated non-uniformly. The coating is more uniform at 10m/min. The XRD results of the rolled material at a rolling speed of 4.7m/min coated with MAO are given in Figure 2. It is seen that the coating of each sample contains Mg, MgO and MgAl<sub>2</sub>O<sub>4</sub> phases. The Mg phase attributed to the AZ31 substrate, exhibits a peak in the XRD pattern, the MgO phase is least influenced by the control parameters. The MgAl<sub>2</sub>O<sub>4</sub> phase has a low intensity for all the samples due mainly to the same amount of Al present in the substrate AZ31 alloy.

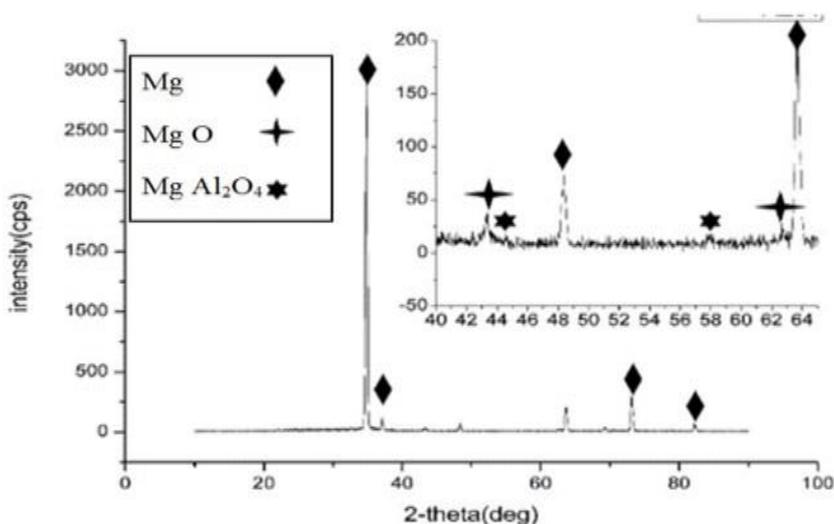


Figure 2. XRD patterns of MAO coated material rolled at 4.7m/min

## B. Surface Smoothness

As can be seen from Figure 3, the surface images of the samples after coating are mostly similar. However, surface smoothness was found to be lower in the material rolled at 10m/min.

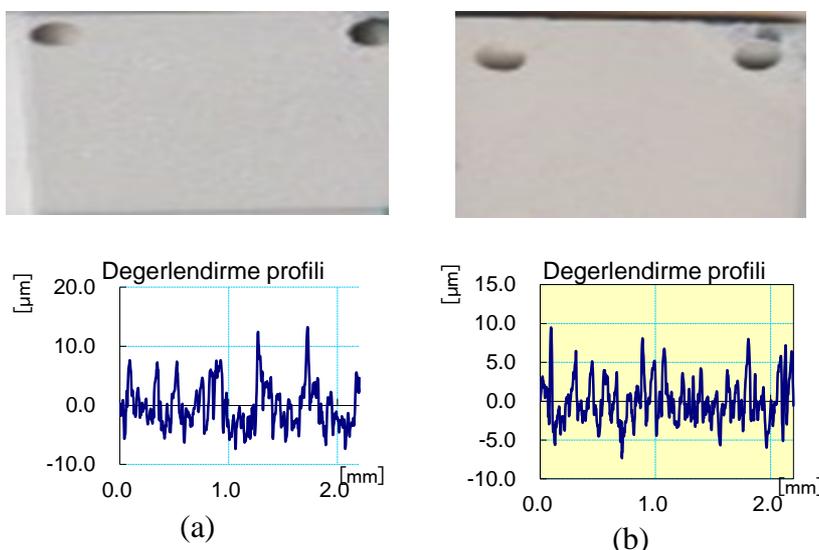


Figure 3. Surface properties of the examined materials at: (a) 4.7m/min (b) 10 m/min

## C. Corrosion Test

Immersion test results are shown in Figure 4. The corrosion rates of the rolled material at the rolling speed of 4.7m/min and 10m/min sample for the first 24 hours are the same, but afterwards, the corrosion rate increased more in the 4.7m/min sample. The corrosion rate of the first 24 hours is under the influence of the coating layer. However, the corrosion rate was influenced by the properties of the base material in the following hours. Firstly, the relationship between corrosion rate and MAO coating was determined by the diameter and the number of pores of the coating. The more pores or the larger the diameter, the faster the corrosion tendency. It is believed that the large diameter pores of the rolled material at 4,7m/min cause a high corrosion rate starts after the 24 hours [10]. The corrosion attacks find a way to enter inside the

coating due to the pores formed during MAO resulting in destruction of the coating layer. On the other hand, the degrading of coating firstly occurs in the porous/outer layer and after that the degrading encounters with the barrier/inner layer which is denser than the porous layer, wherein the corrosion rate is changed by the time of immersion. As seen in Figure 4, the 10m/min rolled sample had the most liner line, decreasing corrosion rate till 168 hours. The dense inner layer is the main determiner of the corrosion rate of the coating under increasing corrosion attacks with increasing immersion time [11]. However, it is known that the rolled materials at high speeds contain fewer twins and more recrystallized grains. It is reported that twinning adversely affects the corrosion resistance due to their different stress zones. However, the fact is that the recrystallized grains contain more grain boundaries and less dislocation density contributes to corrosion resistance [12]. The reason why corrosion resistance is weaker after 24 hours in the material with 4.7m/min rolling speed may be attributed to this.

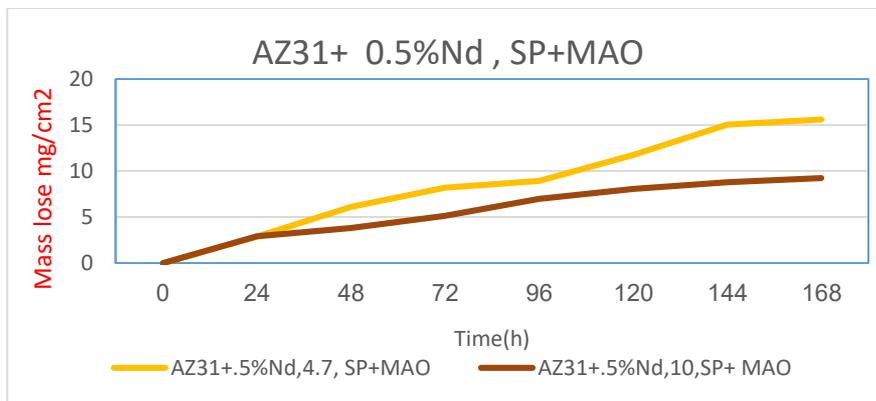


Figure 4. Corrosion rates of samples that were corroded for 168 hours in 3.5% NaCl

#### D. After Corrosion

After the corrosion test, pictures and SEM images were taken from the surfaces of the materials and are exhibited in Figure 5. As it can be seen from the macro pictures and the SEM images, it is

understood that the rolled material with a rolling speed of 4.7m/min has deeper and wider corrosion pits. As can be seen from Figure 6, Mg, O and Al elements remain in the coating material. However, as it progresses towards the base material, Mg becomes more dominant. During corrosion, oxidation occurs on the surface of the material and forms a compound with Al and Mg.

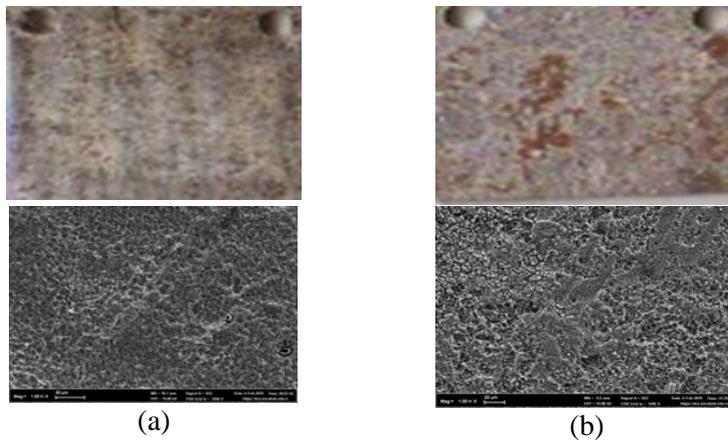


Figure 5. SEM investigations after the corrosion test, (a) 4.7m/min, (b) 10m/min

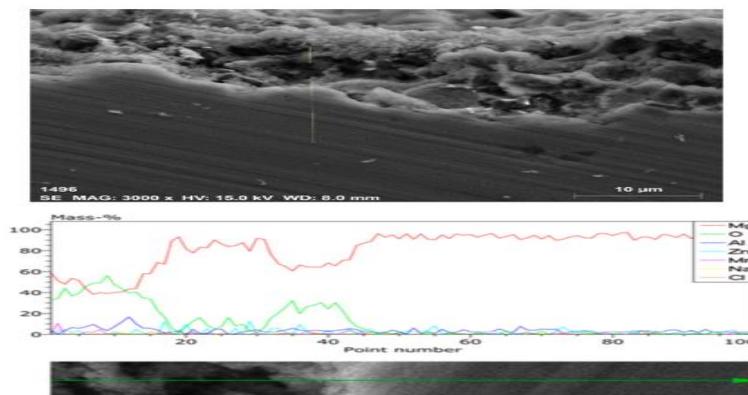


Figure 6. Linear EDS analysis from section to base material after corrosion test for the 4.7 SP-MAO sample

#### IV. CONCLUSION

The corrosion resistance of AZ31Mg alloy with 0.5% Nd of rolling speed 10m/min was higher after the first 24 hours compared to the rolled material at 4.7m/min rolling speed due to the size of micro pores which is narrower at 4.7m/min. and the corrosion rate of 4.7m/min deteriorated by the twins dominated microstructure.

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