

# Characterization and Tribological Properties of Hard Anodized and Micro Arc Oxidized 5754 Quality Aluminum Alloy

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Microstructure  
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## ABSTRACT

This study was initiated to compare the tribological performances of a 5754 quality aluminum alloy after hard anodic oxidation and micro arc oxidation processes. The structural analyses of the coatings were performed using XRD and SEM techniques. The hardness of the coatings was determined using a Vickers micro-indentation tester. Tribological performances of the hard anodized and micro arc oxidized samples were compared on a reciprocating wear tester under dry sliding conditions. The dry sliding wear tests showed that the wear resistance of the oxide coating generated by micro arc oxidation is remarkably higher than that of the hard anodized alloy.

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## 1. INTRODUCTION

Aluminum alloys are very attractive engineering materials for many applications in automotive, construction and aerospace industries due to their low density, good corrosion resistance, enhanced specific strength, high thermal and electrical conductivities [1–6]. Despite these attractive properties, the low wear resistance and relatively low hardness reduce their service time and therefore limit their extensive use in the industry [7–9]. In this respect, hard anodizing appeared as an attractive surface modification technique generating an amorphous oxide layer to enhance wear and

corrosion resistance of aluminum and its alloys. Although slight increase in hardness and wear resistance are achieved by hard anodizing, the enhancements in surface properties are still far from the requirements of the wear-related applications [10]. Moreover, serious concerns of hard anodizing process such as use of strong acids and recycling their disposal, lead to a seeking for an alternative surface modification process in the industry [11].

Recently, micro arc oxidation (MAO) process, also called "plasma electrolytic oxidation", appeared as an alternative electrochemical process to hard anodizing of aluminum and its

alloys [6]. MAO has potentiality to form thick, hard and crystalline oxide layers mainly on light metals. While MAO technique is a relatively new and is used fairly less in the industry compared to hard anodizing, it is one of the most efficient, simplest and fast methods to generate alumina coating, based on the creation of plasma discharges around a substrate immersed in an alkaline electrolyte. In the literature, MAO process is characterized by high and fast productivity, economical efficiency, relatively high hardness, excellent wear resistance, and good adhesion with the substrate [10].

In the present work, alumina coatings were fabricated on 5754 aluminum alloy in an alkaline electrolyte by MAO process. The microstructure, phase composition, hardness and wear resistance of MAO coating were compared with commercially produced hard anodized coating on 5754 aluminum alloy.

## 2. EXPERIMENTAL DETAILS

### 2.1 MAO Process

Samples (15 x 10 x 2 mm) cut from plates of commercial 5754 aluminum alloy were used as the substrate for MAO process. Chemical composition of 5754 aluminum alloy samples used in the present study was as follows (in wt.%): 0.4 Si, 2.6-3.2 Mg, 0.5 Mn, 0.4 Fe and balance Al. Prior to MAO process, the samples were ground using SiC abrasive papers up to #1200, washed with ethanol and distilled water, and completely dried in air at room temperature.

**Table 1.** Electrolyte composition and parameters of MAO and HA processes.

Process	Electrolyte	Applied Voltage	Processing Duration
HA	200 g/L H <sub>2</sub> SO <sub>4</sub> 25 g/L (COOH) <sub>2</sub>	30 V	50 min
MAO	15 g/L NaAl <sub>2</sub> O <sub>4</sub> 2 g/L KOH	440 V	10 min

MAO equipment was an electrochemical cell consisting of a 30 kW power supply capable of applying positive and negative voltage pulses in square form, a stainless steel electrolyte container, an air pump for stirring the electrolyte and an external cooling unit. Samples

were connected to the cell as the anode and the stainless steel container served as the cathode. The process parameters of the MAO are presented in Table 1 along with processing parameters of hard anodizing, which was conducted in an industrial plant.

### 2.2 Characterization Tests

Surface and cross-sectional morphologies of the hard anodized (HA) and MAO samples were examined by using a scanning electron microscope (SEM, Hitachi TM-1000). Qualitative phase analysis of the coatings was determined by an X-ray diffractometer (XRD, GBC MMA-027) using a Cu-K $\alpha$  radiation. Accelerating voltage and applied current were 35 kV and 28.5 mA, respectively. The samples were scanned over 2 $\theta$  angles of 20-80° at a step of 0.02° with a scanning speed of 2°/min. Mean surface roughness (Ra) of the samples was evaluated by a surface profilometer (Veeco Dektak 6M) and coating thickness was determined by the cross-sectional observations of the coatings performed in SEM. Hardness of the coatings was measured from their cross-sections by a depth sensing micro-indentation tester (CSM Instruments Micro-Combi Tester) using a Vickers indenter. Maximum test load was 100 mN, and loading and unloading rates of the indenter were both 200 mN/min. At least ten different indentations on each sample were made to minimize the standard deviation.

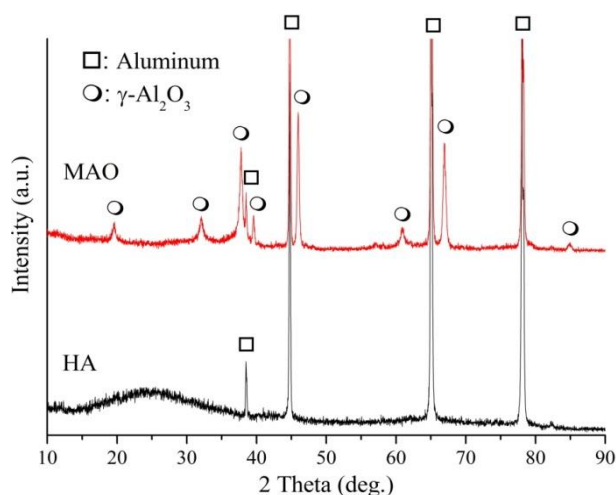
### 2.3 Wear Tests

Dry sliding wear tests of the HA and MAO samples were performed on a reciprocating wear tester (Tribotech Oscillating Tribotester) operating in ball-on-disc configuration at room temperature. In this configuration, a Si<sub>3</sub>N<sub>4</sub> ball with a diameter of 6 mm was sliding forward and backward against the samples with a sliding speed of 6 mm/s. Normal load of the test, sliding amplitude (wear track length) of the reciprocating motion and overall sliding distance were 4 N, 2 mm and 80 m, respectively. During the wear tests, the temperature and the relative humidity were maintained as 23±2 °C and 32±3 %, respectively. The friction coefficient force was continuously recorded during the tests. Width and depth of the wear tracks were measured by a surface profilometer (Veeco Dektak 6M) to calculate worn volume of the

samples and relative wear resistance. Following the wear tests, wear tracks were examined by a scanning electron microscope (SEM, Hitachi TM-1000).

### 3. RESULTS AND DISCUSSION

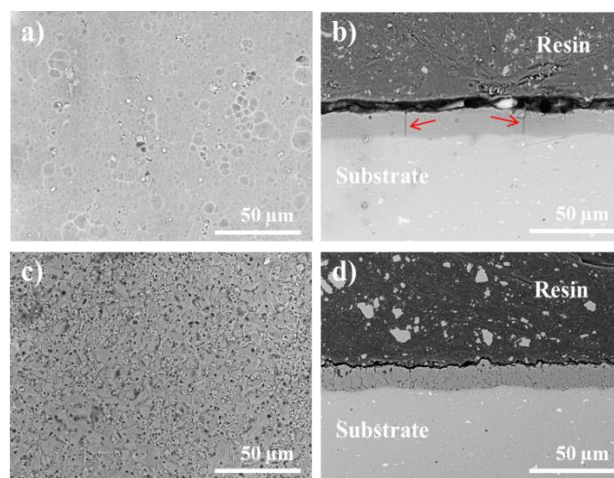
The XRD patterns of HA and MAO samples are given in Fig. 1. According to the XRD analyses, the surface of MAO samples consists of highly crystalline  $\gamma$ -alumina phases, while the coating of HA sample was composed of amorphous oxide phase that was indicated by a wide peak around 15-35°. Aluminum peaks appeared on the XRD patterns are due the penetration of X-rays beyond the oxide layers.



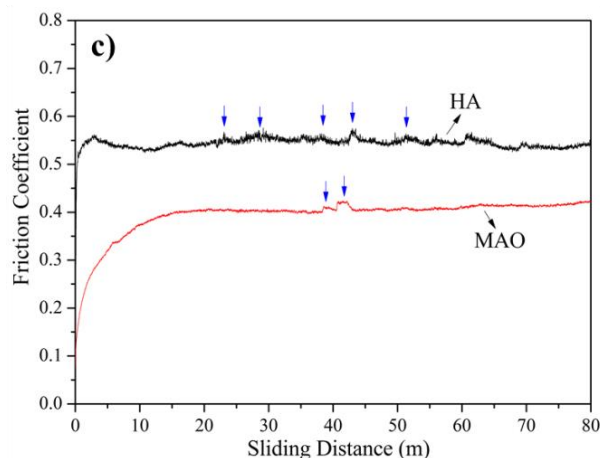
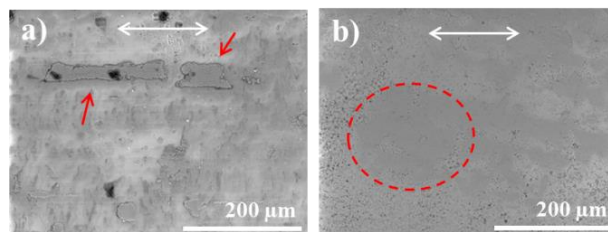
**Fig. 1.** XRD patterns of HA and MAO coatings formed on 5754 aluminum alloy.

The surface and cross-sectional SEM micrographs of the HA and MAO samples are illustrated in Fig. 2. The surface of the MAO sample exhibited typical surface characteristics of MAO by composing of spherical micro pores of various sizes that spread on the surface homogenously. The diameter of the pores was mainly under 3  $\mu\text{m}$ . The formation of micropores have been explained by existence of discharge channels under high voltage (up to 600 V) which leads to pulling out molten oxide and gases during the sparking stage of oxidation process [12]. No visible cracking was observed on the surface for both of the coatings. The presence of micro-cracks in the microstructure of the HA sample is mainly expounded with tensile stress which contributes to local cracking of the coating [13]. Despite of vertical cracks in the microstructure of HA coatings, there were no

cracks or discontinuity at the coating-substrate interfaces of HA and MAO samples which showed that the adhesion of the coating to the substrate is good. The surface roughness of the MAO coating was higher than HA coating due to the sparking on the surface of the 5754 aluminum substrate which creating volcano shaped micro-craters on the surface [14]. The thickness of the HA and MAO coatings were about 22 and 16  $\mu\text{m}$ , respectively. The hardness of the oxide layers generated by HA and MAO were measured as 360  $\text{HV}_{0.01}$  and 690  $\text{HV}_{0.01}$ , respectively.



**Fig. 2.** SEM micrographs of surface and cross-section of (a,b) HA and (c,d) MAO coatings.



**Fig. 3.** SEM micrographs of wear tracks generated on (a) HA and (b) MAO coatings and (c) friction coefficient curves recorded during wear tests.

Appearances of the wear tracks developed on the HA and MAO coatings along with the friction curves are shown in Fig. 3. Rubbing action of the counter face caused smoothening of on the MAO coating, while the wear was progressed by delamination and spallation on HA coating. As the result of the delamination (pointed out with the arrows on friction curves), instantaneous increases in the friction coefficient of HA coating were observed frequently. However, MAO coating kept mostly its steady state regime. In general, steady state friction coefficients of about 0.55 and 0.40 were measured for the HA and MAO samples, respectively. When the wear are (calculated by considering width and depth of wear track) are of concern (Table 2), MAO coating exhibited nearly six times higher wear resistance than the HA coating. Superior wear resistance of the MAO coating compared to HA coating can be attributed to higher hardness of the crystalline alumina coating (360 HV<sub>0.01</sub> and 690 HV<sub>0.01</sub> for HA and MAO samples, respectively).

**Table 2.** Average width and depth of the wear tracks developed on MAO and HA coatings.

Sample	Width of Wear Track (µm)	Depth of Wear Track (µm)
MAO	701 ± 178	0.54 ± 0.09
HA	749 ± 132	3.06 ± 0.16

#### 4. CONCLUSION

MAO generated hard and crystalline alumina layer on wrought 5754 aluminum alloy, while relatively soft and amorphous layer was developed by hard anodizing. Under dry sliding conditions MAO coating exhibited superior tribological performance than HA coating, in terms of higher wear resistance and lower friction coefficient.

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