

## Research Article

# Experimental Investigation on Tribological Behaviour of Various Processes of Anodized Coated Piston for Engine Application

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In the automobile sector, pistons are anodized on the crown, on ring grooves, and also on skirts to improve its wear resistance and corrosion resistance properties. In this work, we have carried out crown anodizing along with skirt anodizing simultaneously to study the wear resistance of anodized samples. The surfaces are anodized with three different processes such as soft anodizing, hard anodizing, and microarc oxidation (MAO) or well known as plasma arc oxidation (PAO). All the three processes differ in their respective procedures. The hardness value and microstructure of all the samples were tested to find wear resistance values and the effect of coatings on the samples. In wear testing, piston samples are rubbed on cast iron to denote its wear resistance based on weight loss per unit time. Reciprocating wear testing is also carried out on every sample for testing their wear resistance value.

## 1. Introduction

Anodizing is a key for surface treatment of aluminum. Cheap density, high specific strength, high electric and thermal conductivity, low cost in comparison to other light metals, great workability, no health danger, and recyclability are just a few of the benefits of aluminum and its alloys [1]. Anodizing is a technique that coats certain metals and alloys in a protective coating which improves wear and corrosion resistance of materials [2]. Wang et al. studied a two-step current-decreasing mode derived from continuous current manner which was established to construct microarc oxidation (MAO) coatings on ZK60 magnesium alloy in a double

electrolyte scheme [3]. According to Zhang and Chen and Zhang et al., recently, the MAO method, which is resulting from the old-style anodic oxidation, has been working as a progressive method to adapt the exteriors of Mg alloys [4, 5]. Anodizing can be done as regular, hard, or MAO coatings, depending on the time period and circumstances applied. In this case, the time gap between normal and hard anodizing might be very different, and MAO coating is a form of anodizing that uses more voltage than hard and normal anodizing [6]. Anodizing's environmental friendliness and relative safety, according to the Aluminum Anodizers Council (2018), are among its greatest features, in an era of increased concern for human protection and

the quality of our land, water, and air [7]. Anodizing is done using simple water-based chemicals that are easy to clean and have no toxic by-products. The liquid by-products are recycled and used again in the manufacturing process. It is possible to extract solid byproducts for use in the production of alum, baking powder, cosmetics, newspaper, fertiliser, and water filtration systems [8]. An oxide coating may be produced on certain metals, such as aluminum, niobium, tantalum, titanium, tungsten, and zirconium, using an electrochemical technique known as anodizing, according to Alwitt [9]. There are process parameters for each of these metals that favour the formation of a thin, dense, uniform-thickness barrier oxide. Only the aluminum and tantalum films are of significant economic and technological use as capacitor dielectrics, as their thickness and characteristics vary substantially depending on the metal [10]. Aluminum is unusual among these metals in that anodizing aluminum alloys in certain acidic solutions creates a thick oxide covering with a high density of tiny holes in addition to the thin barrier oxide [11].

Shen et al. studied the microstructure and corrosion behaviour of microarc oxidation (MAO) coatings on 6061 aluminum alloy that had been pretreated with high-temperature oxidation (HTO). The findings show that the prefabricated HTO film has a significant impact on the creation of the MAO coating, resulting in an enhanced Mg content and decreased coating compactness. HTO, normal MAO (NMAO), and HTO pretreated MAO (HTO-MAO) treatments considerably enhanced the corrosion resistance of the 6061 aluminum alloy, with the NMAO coating exhibiting the highest corrosion performance. The Mg content of the HTO prefabricated film is significantly greater than that of the substrate, which has a significant impact on the MAO coating formation [12]. According to Chen and others, the preparation technique of black microarc ceramic coatings on Al alloys in silicate-based electrolyte was explored in order to create decorative and anticorrosive coatings on aluminum alloys. The ceramic coating got darker and thicker without combination additives as the  $\text{Na}_2\text{WO}_4$  content in the electrolyte increased, but the colour was not black; after adding combination additive, the coating became black; the black ceramic coating has a multihole form in the surface. Apart from the  $-\text{Al}_2\text{O}_3$  phase, there was a minor amount of tungsten in the black ceramic coating, and the aluminum alloy with black ceramic coating demonstrated outstanding anticorrosion properties in acid, basic, and neutral 3.5 percent NaCl solution [13]. Ly and Yang examined the corrosion resistance of these coatings which would deteriorate while the current increases during the final repairing stage, which is attributed to low compactness of coatings and uneven distribution of pores [14].

Microarc oxidation was used by Li et al. to create a black ceramic layer containing  $\text{V}_2\text{O}_3$  on an aluminum alloy surface. It is suggested that the  $\text{V}_2\text{O}_3$  generated in this sublayer is responsible for the achieved black look, and it was discovered that the black ceramic layer grows in two phases. Microzones containing vanadium are generated and enlarged in the first step. The adsorption of  $\text{V}_2\text{O}_3$  on the metastable oxides plays a significant role in the formation of the black ceramic

layers after the microarc oxidation procedure was initiated for 3 minutes [15]. The protection or decoration of Al surfaces by anodization has been used commercially since at least 1923. Self-organized “nanopore” structures in anodic alumina films called alumite have attracted great attention due to their high pore density and their potential use for masking or information storage. When the pores are filled with metals or semiconductors in a subsequent alternating current reductive electrolysis, these films can be fabricated into interesting magnetic recording, electronic, and electro-optical devices. In anodized aluminum oxide, there is formation of highly organized pores. It has been proven that repeated anodization and peeling of the porous oxide can result in hexagonally organized domain structures in anodic alumina films. The domain size was discovered to be a linear function of time and grows with temperature. The pore density is initially high, but as prominent pores deepen, it declines with anodizing time. In native oxide in the air or nucleate after electropolishing, very tiny holes occur. When the electric field at the pore bottoms grows and acid dissolves the oxide locally, pore development may begin [16–18].

Existing journals already published about the anodizing process, types of anodizing, and metals that can be anodized. But our work leads to denote the wear resistance of anodized piston samples in two ways like normal and hard anodizing, whereas normal anodizing represents thin coating of the anodized layer than hard anodizing. The main objective of our project is to compare the wear resistance of anodized and MAO-coated samples to denote and categorize the best based on requirement which helps to improve the lifetime and corrosion resistance of pistons.

## 2. Materials and Method

Aluminum piston is the selected sample for this work, and those samples are cut into the required shape to be fixed for wear resistance testing [19, 20]. Samples will be cut by using different cutting processes like wire cutting or cutting using saw. Samples will be structured using a lathe and other tools to get the required shape. The samples for wear resistance will be in smaller size, and in such situation, wire cutting will help for better results [21–23]. The prepared samples are ready for anodizing, and a setup will be fabricated for anodizing the samples.

*2.1. Methodology for Sample Preparation.* There are various steps involved in the process of sample preparation which are shown in Figure 1.

From the flowchart as shown in Figure 2, the procedure for sample preparation is mentioned in a sequence of operation. Based on this sequence, samples for wear resistance are prepared. These samples are later utilized for wear resistance [24].

*2.2. Methodology for the Entire Process.* Anodizing is an electrolysis process so electrolysis setup will be fabricated using anode, cathode, rectifier, tank, electrolyte, and chemicals (sulfuric acid and oxalic acid). Samples are anodized and inspected to remove the defected outputs [25]. Anodized

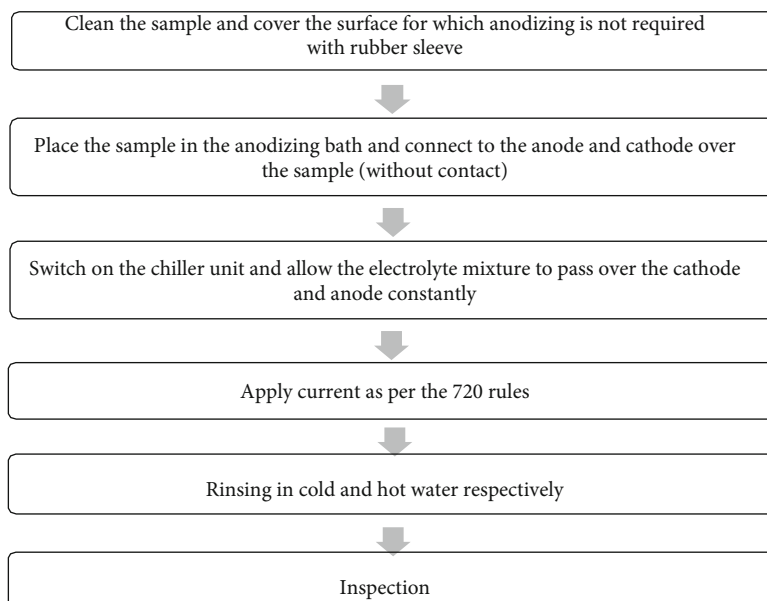


FIGURE 1: Methodology for sample preparation.

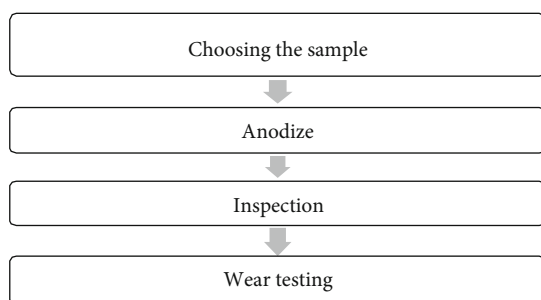


FIGURE 2: Methodology for the entire process.

samples are basically inspected physically like visual inspection. Samples were tested for hardness and also checked for its thickness. Finally, the samples are tested for wear using different wear testing methods [26].

From Figure 3, we can see that anodizing setup resembled as electrolysis process is made using leads as cathode and sample as anode, rectifier, electrolyte mixture, and wire connectors. Current will be supplied based on the requirement, and it can be adjusted based on the time interval [27].

### 3. Experimentation

The anodized aluminum layer is grown by passing a DC current through an electrolytic solution with the aluminum object serving as an anode (positive electrode) and lead as cathode (negative electrode). Table 1 shows the percentage of acids in electrolyte mixture. Electrolytic solution consists of sulfuric acid and oxalic acid in proportions as follows [28, 29].

- (i) The lead sheet which is acting as cathode is semiautomatically placed over the piston samples covered

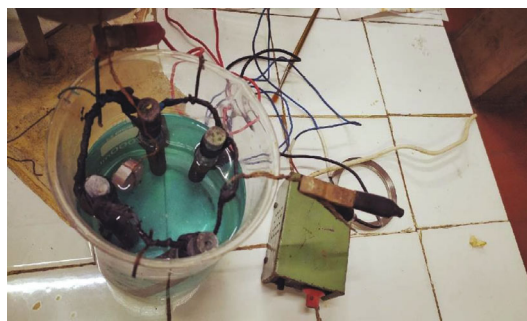


FIGURE 3: Setup used for sample preparation.

TABLE 1: Percentage of acids in electrolyte mixture.

Acids	Composition (%)
Sulfuric acid	12-15% for normal anodizing 18-20% for hard anodizing
Oxalic acid	4%

with sleeves whereas the pistons are placed on the stainless-steel sheet which acts as positive terminal, the electrolyte is made to pass through PVC pipe holes which is placed over the lead sheet, and it is a cyclic process [30]

- (ii) Cooling system is connected externally to this setup, and the temperature is maintained by using ethylene glycol  $[(\text{CH}_2\text{OH})_2]$  for antifreeze formulation
- (iii) Cathode and anode are connected to a rectifier externally which is used for setting current supply

### 3.1. Equipment for Anodizing

- (i) Tank: is used to store electrolyte and is lined with lead, rubber, or PVC and suitably equipped for air agitation and cooling
- (ii) Air agitation: used to extract the gases like hydrogen formed during the process
- (iii) Cooling unit: a chiller unit is used to maintain the temperature of the process in which chemicals like ethylene glycol are used for antifreeze formulation
- (iv) Filtering unit: used to remove the foreign particulates in the electrolyte
- (v) Masking: rubber sleeves are used for masking the unwanted anodize surface layer
- (vi) Rectifier: used for current supply and the current supply will be as per 720 rule
- (vii) 720 rule: we can get a good estimate of the processing time using the 720 rule which states that it takes 720 amp minutes per square foot to produce one mil of oxide layer. The application of this rule is simple. Take 720, and divide it by the current density using in amps per square foot, so that we can get the time interval based on our requirement [31]
- (viii) Chemicals: sulfuric acid, oxalic acid, DM water, and ethylene glycol
- (ix) Cathodes: mainly lead sheet will be used, and the distance between the anode and cathode should be a minimum of 75 mm

**3.2. Anodizing Process for Sample Preparation.** The prepared piston samples are cleaned to anodize and cover the overexposure surface with rubber sleeve. Ensure that the sleeve is fixed tight where the samples should not anodize. Place the piston sample on the copper sheet which is located inside the bath tank [32]. This copper sheet will act as anode of the process, and the piston sample is placed on this sheet which makes the sample act as anode of this electrolysis process. A lead sheet which acts as cathode is placed over the sample (without any physical contact). A chiller unit is used to maintain the temperature below 10 degrees Celsius to help the process and optimize it. The chiller unit is maintained by using ethylene glycol which helps for antifreeze formulations. A rectifier is used for current supply for the electrolysis process which is connected to the setup, and anode and cathode connections are done, respectively [33]. Current supply will be given based on the 720 rule which will give the current density value based on the time interval or vice versa. The electrolyte which is a mixture of DM water, sulfuric acid, and oxalic acid in definite proportions is made to flow cyclic from over the lead sheet and on to the samples [34].

During the process, hydrogen will be segregated at the cathode and oxide ions will be released which will react with

the anode, i.e., sample, and form as aluminum oxidic layer over the surface of aluminum [35]. This layer is a highly ordered and porous structure that allows for thickness build up and also allows for secondary processes such as coloring and sealing. The rate of oxidic layer formation is constant with time, so based on time interval, the required amount of thickness can be achieved [36]. This differentiates the anodizing to normal and hard. After anodizing, the pistons are rinsed in cold and hot water, respectively. Cold water rinsing is for clearing acid particulates, and hot water rinsing is for rapid drying and speeding up the production. Later, the pistons are inspected to detect any defects. And if a full body anodizing is necessary, the sample is just dipped inside a jar or tank containing the electrolyte mixture and a lead sheet or rod is placed inside the electrolyte tank which will act as cathode and a sample will be connected to the anode terminal and the process is repeated as explained before [37].

### 3.3. Chemical Equation of Anodizing of Aluminum Sample.



The anodizing process requires electrolyte (water) and sulfuric acid as main chemical mixture which will react to the Al metal, and a layer of aluminum oxide will form on the metal surface which can also be denoted as alumina. The latter eminent like hydrogen is removed using air agitation [38].

**3.4. Thickness Formation of Anodized Samples.** The thickness value for anodized samples varies for hard and normal anodized samples and MAO-coated samples. As mentioned before, the thickness for normal anodized samples will be 10-25 microns and for hard anodized, it will be in the range of 25-75 microns. For MAO-coated samples, thickness ranges as 50-75 microns. But the colour of MAO-coated samples will be little deviated from normal and hard anodized samples. Normal and hard anodized samples' colour will be like grey, and for MAO coating, it will be light grey colour. Based on the time interval and input current supply maintained, the thickness formation will be varied. For requirement of specific thickness value, the 720 rule is applied for better results which will give the respective current density value for particular time interval and vice versa. Sometimes, the thickness may vary based on the environmental conditions and the condition of the setup created and also for the electrolytic mixture. If the acid content of the electrolyte mixture is decreased below the required quantity, the oxide layer formation will get less.

**3.5. Thickness of the Oxide Layer.** Samples for finding thickness of the oxide/anodic layer are made using mount sample equipment in which bakelite powder is used to bind the sample tight as shown in Figure 4. The mount is made at a temperature of 70 degrees Celsius. The anodized sample is made to stand on the flat surface, and it is covered with bakelite powder around and is pressurized using hydraulic pressure system and is closed tightly until the temperature reaches to 70 degree Celsius. Later, the sample is made to





FIGURE 4: Mounted sample for measuring thickness of the oxide layer.

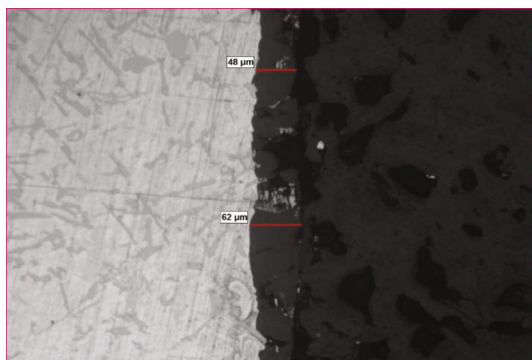


FIGURE 5: Microscopic photo of the oxide layer of the hard anodized sample.

cool and is polished/grinded using a manual grinding machine and later polished using a semiautomatic polishing machine. The polished surface is tested for its required smoothness to test it for microscopic thickness measurement.

#### 4. Results and Discussion

The microscopic photocopies of anodized samples are shown, and thickness variation is explained. Photos of both hard and normal anodized samples are discussed and explained separately.

**4.1. Microscopic Photo of the Oxide Layer of Hard Anodized Samples.** Figure 5 shows the thickness measurement of the hard anodized sample through an electronic microscope. This photo is taken in a scale of 100x zoom onto the oxide layer.

As shown in Figure 5, the thickness of the anodic layer will vary and it will not be continuous for the whole layer formation. So, the thickness varies from one point to another and so a range of thickness measurement will be dis-



FIGURE 6: Microscopic photo of the oxide layer of the normal anodized sample.

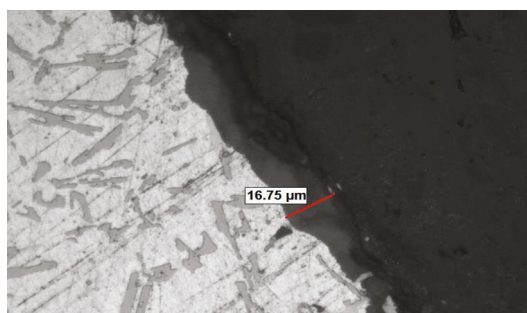


FIGURE 7: Microscopic photo of the MAO-coated sample (crown part).

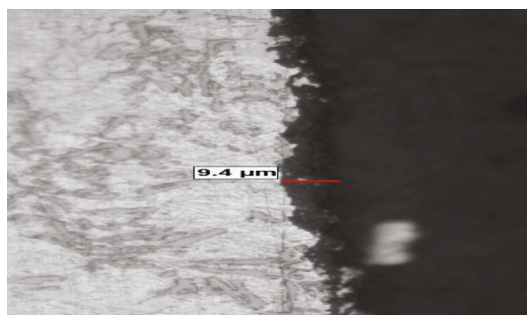


FIGURE 8: Microscopic photo of the MAO-coated sample (skirt portion).

played for any sample. Here, as observed, a good continual layer of oxide is formed and a range of 40-65-micron thickness can be observed.

**4.2. Microscopic Photo of the Oxide Layer of the Normal Anodized Sample.** The microscopic photo shown in Figure 6 is about the thickness of the oxide layer formed on the Al sample. It was taken in a 500x zoom scale from an electronic microscope.

As shown in Figure 6, thickness is varied from one point to another. It is discussed before that the thickness of the anodic layer is not continuous. The maximum thickness achieved from normal anodizing of the sample is 25.2

TABLE 2: Results for normal anodized samples.

S. No	Current	Thickness of the anodized layer (microns)	Hardness value (HV)	Conductive test	Time interval (minutes)
1	0.5 amps & 7.5 V	6.2	350	Pass	40
2	0.5 amps & 12 V	5	330	Pass	25
3	0.5 amps & 12 V	31	550	Pass	60
4	0.5 amps & 12 V	22	468	Pass	90
5	0.5 amps & 12 V	27	520	Pass	90

TABLE 3: Results for hard anodized samples.

S. No	Current	Thickness of the anodized layer (microns)	Hardness value (HV)	Conductive test	Time interval (minutes)
1	2.5 amps & 20 V	57	632	Pass	45
2	2.5 amps & 20 V	56	575	Pass	45

microns as shown in Figure 7. The alloy used for this normal anodizing is LM13 of aluminum which consists of about 82-85 percentage in volume; the rest will be of iron, silicon, magnesium, and copper.

**4.3. Microscopic Photo of the Oxide Layer of the Microarc Oxide- (MAO-) Coated Sample.** The microscopic photos shown in Figures 7 and 8 are about the thickness of the oxide layer formed on the Al sample. Figure 7 is taken from the crown part of the piston sample.

As we can see from Figures 7 and 8, the thickness observed is in the range of 10~16 microns. Due to lower crown depth, it is unable to check for the hardness value.

**4.4. Normal Anodized Samples.** Results obtained for normal anodized samples are shown in Table 2. For the results like hardness value (if obtained), conductive test at the oxide layer should be nonconductive and thickness of the anodized layer for the current applied should be observed.

In Table 2, the variation in the thickness of the oxide layer is due to the environmental conditions and alloy of the Al sample used for anodizing. The hardness value for few samples not mentioned is because of imperfect formation of the indent over the oxide layer, as the hardness of the samples is measured using a Vickers hardness tester. It requires at least of 22-25 microns of thickness to form an indentation over the oxide layer. The load in the Vickers hardness test is constantly maintained as 25 grams for each sample; at this load condition, an indenter of a shape of square is made to form an indentation over the oxide layer and based on the diagonal lengths formed, the hardness value is determined. Normally, the time interval for normal anodized samples is less and it is about 15-20 minutes but the increase in the time interval here is mainly due to the low application of the current and secondarily for the requirement of the thickness of the oxide layer, as discussed to maintain at least of 20 microns of thickness for wear testing.

**4.5. Hard Anodized Samples.** Results obtained for hard anodized samples are shown in Table 3. Results like thickness were observed for the applied current supply, hardness value

for the obtained oxide layer, and time interval of the sample of anodization, and conductive test is done to denote the conductivity nature of the oxide layer formed as it should not be conductive.

## 5. Conclusion

In this work, we have carried out crown anodizing along with skirt anodizing simultaneously to study the wear resistance of anodized samples. We have observed the external effecting factors for anodizing and the thickness formation effecting factors and looking to optimize the condition of the sample preparation process.

- (i) For normal anodizing, the condition of the temperature is atmospheric as the process can be done to achieve for lower thickness
- (ii) Time taken for normal anodizing will be low, but for achieving higher thickness, the time interval should be high and for hard anodizing, thickness formation is higher and so the temperature is to maintained for optimization
- (iii) Wear resistance will be greater for MAO-coated samples then for hard anodized and normal anodized samples, respectively

## Data Availability

The data used to support the findings of this study are included in the article. Should further data or information be required, these are available from the corresponding author upon request.

## Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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