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Study on the Influence of Process Parameters on the Thickness and Micro hardness during the Sulfuric Acid Anodization of AA 6061

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*Abstract— **Aluminium alloys are now gaining their usage in gas turbine, space vehicles and aircraft because of their inherent properties like good formability, weldability, machinability, wear resistance and corrosion resistance. The anodization process of metals has been used by industry to increase the corrosion resistance. In the present work, the influence of various process parameters, i.e. electrolyte concentration, electrolyte temperature and voltage on the thickness and micro hardness was studied during anodization of aluminum alloy AA 6061 in sulfuric acid. The settings of the process parameters were determined by using Taguchi's experimental design method. Orthogonal arrays of Taguchi, the Signal-to-Noise (S/N) ratio, and Analysis of Variance (ANOVA) were employed to find the optimal process parameter levels and to analyze the effect of these parameters on thickness and micro hardness. The main parameter affecting thickness is identified as electrolyte temperature and the parameter affecting micro hardness is voltage. Confirmation test with the optimal levels of parameters was also carried out in order to illustrate the effectiveness of the Taguchi optimization method.***

Index Terms—**AA 6061, Anodization, ANOVA, Micro hardness, Taguchi DOE, Thickness**

I. INTRODUCTION

Anodization is an electrolytic process used to create a uniform oxide layer on the surface of the component to prevent it from corrosion, abrasion which also provides additional surface hardness [1]. This process is extensively used to provide surface protection to aluminum alloy components. The natural oxide layer on aluminum pieces is only 40-50 ^{o}A thick. This already existing film has high porosity and less mechanical strength. Hence it cannot provide desired corrosion resistance. Hence to obtain a uniform oxide layer with considerable thickness, we need to anodize the aluminum parts [2]. The properties of the films obtained by anodic oxidation can be considerably varied such as hardness, thickness, elasticity depending primarily upon the composition of the electrolyte and the degree of its reactivity towards the oxide films which are formed during the treatment [3, 4]. In this electro-chemical process, the part to be anodized is made as the anode of the electrical circuit. As the part undergoes the process, the thin, invisible natural aluminum oxide layer on the surface of the aluminum which provides some degree of corrosion protection for the underlying aluminum is converted into thicker oxide layer, which makes it resistant to corrosion, and also increases its hardness [5, 6]. The main anodization parameters are electrolyte concentration, electrolyte temperature and voltage. The effect of these parameters on the thickness and micro hardness of the anodic films has not been investigated till date.

II. MATERIALS AND METHODS

A. Work piece

AA 6061 is the base material used in this experiment. It is extremely useful in gas turbine, space vehicles, aircraft and nuclear reactors, submarine. AA 6061 is a precipitation hardening aluminum alloy, containing magnesium and silicon as its major alloying elements. It is commonly available in pre-tempered grades such as 6061-O (annealed) and tempered grades such as 6061-T6 (solutionized and artificially aged) and 6061-T651 (solutionized, stress-relieved stretched and artificially aged). T6 temper 6061 has an ultimate tensile strength of at least 300 MPa and yield strength of 241 MPa. The typical value for thermal conductivity for 6061-T6 at 80°C is around 152 W/m K. Subsequent topics discuss about the experimental set up, results & conclusions drawn out.

B. Experimental details

AA 6061 temper grade T652 containing 95.85-98.56 % Al, 0.4-0.8 % Si and 0.8-1.2 % Mg was used as the starting material. Disc specimens of diameter 46 mm by 10 mm thick were prepared by CNC turning and a jigging hole of diameter 4 mm was provided. A surface finish of Ra 0.3-Ra 0.5 was achieved. Anodizing process was carried out in three stages viz, pre- anodizing treatment, anodizing and post treatment. The pretreatment involved manual degreasing in Tri Chloro Ethylene followed by vapor degreasing in TCE at 85^{0}C for 20 minutes. This was followed by alkaline etching in 5% ±1% Sodium Hydroxide solution (by wt) at 55^{0}C for 10-30 seconds. The smut and residue left as a result of descaling was removed by desmutting in 50% ±2% by volume Std. Nitric Acid (HNO_3) with 70% concentration at room temperature for 120 seconds. Rinsing in deionized water was then done subsequently for 10 minutes. To ensure a regular structure chemical polishing to improve surface finish was done in orthophosphoric acid 95% and nitric acid 5 % at 70^{0}C for 2 minutes. Chemical polishing solution was replaced after each experiment. Sulfuric acid anodizing with variable parameter combinations comprising of voltage, temperature and bath concentration were carried out. Duration of 45 minutes was kept constant for all the experiments. After anodization, the specimens were rinsed in clean running water and were sealed in hot water for 1 hour.

C. Measurement of Thickness and Micro hardness

Thickness of the anodic film is measured using Fischer scope MMS PC2 (Figure 1) which works on the principle of eddy current. This is a non- destructive method that has been widely adopted for production control purposes. When the probe is applied to a metal surface, it induces a current in the metal. The strength of this current is reduced if the probe is separated from the metal surface and this loss is picked up by a detector in the probe and fed back to the instrument. The induced current is converted to a scale showing coating thickness in microns.

Fig 1: Fischer scope MMS PC2

The micro hardness of the anodic oxide film is measured using Fully Automatic Micro- hardness tester MHT- Smart by Omnitech (Figure 2). Load range is from 10 gms to 1000gms. Automatic loading and releasing is possible. The magnification of microscope available are 100 x and 400 x.

Fig 2: Automatic Micro- hardness tester MHT- Smart by Omnitech

D. Taguchi's Design of Experiment

Design of Experiments (DOE) is an experimental strategy in which effects of multiple factors are studied simultaneously by running tests at various levels of the factors. Taguchi DOE is a powerful and efficient method over other traditional methods. The main difference between Taguchi DOE and full factorial method is regards the number of experiments to be done. For a 3 factor- 4 level combinations the total experiments to be conducted in full factorial method is 64. Whereas in Taguchi DOE only 16 experiments has to be done. For a DOE is a systematic approach to investigation of a system or process. A series of structured tests are designed in which planned changes are made to the input variables of a process or system. The effects of these changes on a pre-defined output are then assessed. For each input variable, a number of levels are defined that represent the range for which the effect of that variable is desired to be known. The response is then measured for each run. The method of analysis is to look for differences between response (output) readings for different groups of the input changes. These differences are then attributed to the input variables acting alone (called a single effect). MiniTab 16 software was used for generating the Taguchi's orthogonal array.

E. Experimental Procedure

For studying the degree of influence of the process parameters during anodization of AA 6061 in sulfuric acid, three factors, each at four levels are taken as shown in Table 1. In this research, sixteen experiments were conducted at different parameter levels. For this L16 orthogonal array was used, which has sixteen rows corresponding to the number of tests (Table 2).

Table 1: Anodization parameters and their levels

Parameter or Factor	Levels and values				Output Responses
	1	2	3	4	
Electrolyte concentration (%wt/vol)	14	16	18	20	1.Thickness 2.Micro-hardness
Electrolyte temperature (˚c)	5	10	15	20	
Voltage (V)	14.5	18	21.5	25	

Table 2: L16 orthogonal array

Expt	Electrolyte concentration (%wt/vol)	Electrolyte temperature (˚c)	Voltage (V)
1	1	1	1
2	1	2	2
3	1	3	3
4	1	4	4
5	2	1	2

6	2	2	1
7	2	3	4
8	2	4	3
9	3	1	3
10	3	2	4
11	3	3	1
12	3	4	2
13	4	1	4
14	4	2	3
15	4	3	2
16	4	4	1

III. RESULTS AND DISCUSSION

After the experiments were completed, the thickness and micro hardness of the anodized specimens were measured. Table 3 shows the thickness and micro hardness measured by Fischer scope MMS PC2 and fully automatic Micro- hardness tester MH Smart by Omnitech.

Table 3: Measured values of thickness and micro hardness

Electrolyte concentration (%wt/vol)	Electrolyte temperature (˚c)	Voltage (V)	Thickness (µm)	Microhardness (HV)
14	5	14.5	2.33333	85
14	10	18	11.3333	186
14	15	21.5	22.6666	274
14	20	25	65.3333	365
16	5	18	13.6666	143
16	10	14.5	4.6666	100
16	15	25	27.6666	296
16	20	21.5	54.6666	347
18	5	21.5	13	128
18	10	25	6.3333	326
18	15	14.5	13	205
18	20	18	23.3333	285
20	5	25	46	333
20	10	21.5	27.6666	301
20	15	18	19.6666	257
20	20	14.5	22.6666	281

A. Analysis of S/N Ratio

Taguchi method stresses the importance of studying the response variation using the signal – to – noise (S/N) ratio, resulting in minimization of quality characteristic variation due to uncontrollable parameter. The thickness and micro hardness was considered as the quality characteristic with the concept of “nominal- the- best” and "larger-the-better" respectively.

The S/N ratio for nominal the best is:

$$S/N = 10\log\left[\frac{\mu^2}{\sigma^2}\right] \qquad (1)$$

Where μ is the mean and σ is the standard deviation.

The S/N ratio for the larger-the-better is:

$$S/N = -10\log\left[\frac{1}{n}\sum_{i=1}^{n}\left(\frac{1}{{y_i}^2}\right)\right] \qquad (2)$$

Where n is the number of observations, and y is the measured output value.

The thickness and micro hardness values and their corresponding S/N ratio values are listed in Table 4.

Table 4: Thickness and micro hardness values along with their corresponding S/N ratio values

Exp. No.	THICKNESS		MICROHARDNESS	
	Thickness (µm)	S/N ratio (dB)	Microhardness (HV)	S/N ratio (dB)
1	2.33333	12.1307	85	38.5884
2	11.3333	25.8584	186	45.3903
3	22.6666	23.4280	274	48.7550
4	65.3333	29.9345	365	51.2459
5	13.6666	19.0335	143	43.1067
6	4.6666	18.1513	100	40
7	27.6666	27.5897	296	49.4258
8	54.6666	31.0747	347	50.8066
9	13	22.2789	128	42.1442
10	6.3333	14.7833	326	50.2644
11	13	22.2789	205	46.2351
12	23.3333	23.6798	285	49.0969
13	46	28.4839	333	50.4489
14	27.6666	27.5897	301	49.5713
15	19.6666	24.6252	257	48.1987
16	22.6666	25.8584	281	48.9741

The S/N ratio values for thickness by factor level are shown in Table 5 and S/N ratio values for micro hardness by factor level are shown in Table 6. Regardless of the category of the performance characteristics, a larger S/N value corresponds to a better performance. Based on the analysis of the S/N ratio, the optimal combination for nominal thickness is obtained at 20 wt%/volume electrolyte concentration (level 4), 20 °c electrolyte temperature (level 4) and 21.5 volts voltage (level 3). Similarly from the analysis of the S/N ratio, the optimal combination for micro hardness is obtained at 20 wt%/volume electrolyte concentration (level 4), 20 °c electrolyte temperatures (level 4) and 25 volts voltage (level 4).

Table 5: Response Table for thickness S/N Ratios (Nominal the best)

Level	Electrolyte concentration	Electrolyte Temperature	Voltage
1	22.84	20.48	19.60
2	23.96	21.60	23.30
3	20.76	24.48	26.09
4	26.64	27.64	25.20

Delta	5.88	7.16	6.49
Rank	3	1	2

Table 6: Response Table for micro hardness S/N Ratios (Larger is better)

Level	Electrolyte concentration	Electrolyte Temperature	Voltage
1	45.99	43.57	43.45
2	45.83	46.31	46.45
3	46.94	48.15	47.82
4	49.30	50.03	50.35
Delta	3.46	6.46	6.90
Rank	3	2	1

Figure 3 shows the effect of process parameters on the thickness of anodic oxide films and figures 4 show the effect of process parameters on the micro hardness of anodic oxide films.

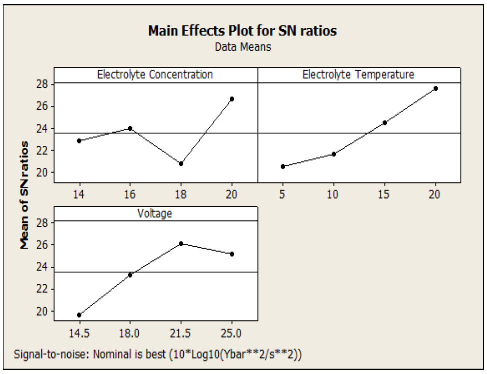

Fig 3: S/N ratio plot for thickness

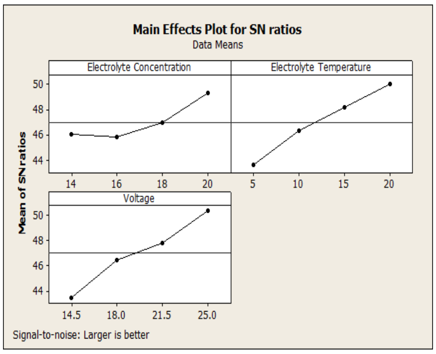

Fig 4: S/N ratio plot for micro hardness

B. Analysis of Variance

Statistical analysis of variance (ANOVA) is done to investigate which design parameter significantly affects the micro hardness and thickness. Based on the ANOVA, the relative importance of the anodizing parameters with respect to thickness and micro hardness was investigated to determine the optimum combination of the anodizing parameters. All analysis is carried out for a significance level of α=0.05, i.e., for confidence level of 95%. ANOVA table also has probability level that is the realized significance level, associated with the F-tests for each source of variation. The sources with a probability level less than 0.05 are considered to have a statistically significant contribution to the performance measures. Also the percentage of contribution of each source to the total variation indicates the degree of influence on the result by each source. The P-value reports the significance level (suitable and unsuitable). Percent (%) is defined as the significance rate of the process parameters on the output responses. It can observed from Table 7 that the electrolyte concentration, electrolyte temperature and voltage affect the thickness by 16.92%, 28.90% and 23.28% respectively during the anodization of AA 6061 in sulfuric acid.

Table 7: Analysis of Variance for thickness

Source	DOF	Sum of squares	Mean of squares	F	P	% contribution
Electrolyte concentration	3	72.13	24.04	1.10	0.42	16.9
Electrolyte temperature	3	123.2	41.07	1.87	0.23	28.9
Voltage	3	99.23	33.08	1.51	0.30	23.2
Error	6	131.6	21.95			30.8
Total	15	426.2				

Table 8: Analysis of Variance for micro hardness

Source	DOF	Sum of squares	Mean of squares	F	P	% contribution
Electrolyte concentration	3	30.61	10.20	2.8	0.12	12.6
Electrolyte temperature	3	90.99	30.33	8.5	0.01	37.5
Voltage	3	99.12	33.03	9.3	0.01	40.9
Error	6	21.32	3.55			8.8
Total	15	242.0				

C. Confirmation Test

The confirmation test is the final step in verifying the results obtained from Taguchi's design approach. The optimal conditions are set for the significant factors and experiments are run under specified anodizing conditions. The confirmation experiment is a crucial step and is highly recommended by Taguchi to verify the experimental results.

In this study, a confirmation experiment was conducted by utilizing the levels of the optimal process parameters. The thickness obtained at 20 wt%/volume electrolyte concentration (level 4), 20 °c electrolyte temperature (level 4) and 21.5 volts voltage (level 3) is 35.85 µm. Similarly, the micro hardness obtained at 20 wt%/volume electrolyte concentration (level 4), 20 °c electrolyte temperatures (level 4) and 25 volts voltage (level 4) is 408 HV.

IV. CONCLUSION

In this study, aluminum oxide layer was produced under various experimental conditions and the thickness and micro hardness were measured. 16 levels of experiments had been done. Moreover, thickness and concentration have a direct relation. Higher concentrations aid in high oxide growth rate because of the higher conductivity resulting in larger current densities. It was also observed that anodizing thickness and micro hardness have direct relation. AA 6061 tends to have good anodizability. Similar studies can be extended to AA 7075 alloys which due to the presence of Zinc as an impurity, tends to have poor anodizability

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