Effect of Surface Preparation on Mechanical Properties of Anodized A6061 in Sulfuric Acid Bath

Amane Sahli, Oussama Djema, Mabrouk Bouabdallah, and Djaffar Saidi

Abstract− The aims of this work are to examine the effect of the pickling conditions on the mechanical behavior of anodized A6061. Pickling was explored at 60°C for 30, 60, 90 and 120 second. The growth rate of the anodic layer, surface roughness, microhardness and tensile strength of anodized samples treated by alkaline pickling were investigated. The experimental results showed that the thickness of the anodic layer is directly related to the roughness of the substrate. In addition an important growth in thickness layer affects slightly the mechanical proprieties.

***Keywords*− A6061 aluminum alloy, Sulfuric anodizing, Microstructure, Surface morphology, Surface roughness, Microhardness, Tensile strength.**

I. INTRODUCTION

The aluminum alloy A6061 is widely used in the aircraft, automotive industries and nuclear industry, because of their low density, good mechanical properties and easily manufactured [1]. However, due to his heterogeneous microstructure the A6061 is prone to localized corrosion, a drawback that limits its technical applications especially in an aggressive environment [2-4]. Currently, many researchers have investigated anodizing process to improve the surface performance and corrosion resistance of aluminum alloys [5-7].

Anodizing is an electrochemical process to produce an aluminum oxide film Al2O3 with a thickness that can reach 500 times the oxide layer naturally formed [8-10]. Aluminum anodization can be processed in an acid or alkaline bath [11]. The sulfuric acid anodizing is the most commonly bath used in the industry due to lower cost and rapid action. Researchers [12] focused their work on the influence of the current density, concentration, compositions, temperature of electrolyte and anodization time on the quality and thickness of the anodic film. T.C. Cheng et al [13] studied the effect of the sulfuric acid concentration, and anodizing voltage. They demonstrated that 0.3 M sulfuric acid bath and the range of 30 - 50 V are the optimum conditions to anodizing of A6061. C.C. Lee et al [14] prepared AA6061 oxide films in 10 % H2SO4 electrolyte solutions for 20 min at 0°C and 22°C. The results showed that anodic oxide film with a higher hardness can be obtained at a lower anodization temperature (0 °C) than at room temperature.

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In this aluminum anodizing experiment, the A6061 sheets anodized with sulfuric acid were obtained using different conditions of pickling. The surface morphology, surface roughness and the mechanical properties of the samples, including their microhardness, tensile strength and elongation were analyzed in order to observe the influence of surface preparation against the mechanical properties of anodized A6061.

II. MATERIALS AND METHODS

II.1. MATERIALS

The material investigated in this study was aluminum alloy 6061-T6 with the composition as given in Table 1. The tested samples were machined according to the tensile test specimen before anodizing treatment.

Table. I
Chemical composition of the A6061 (wt.%)

Element	Al	Mg	Si	Cr	Mn	Ti	Cu	Zn	Fe
Composition	Bal	1.06	0.64	0.24	0.77	0.06	0.2	0.01	0.04

II.2. SAMPLE PREPARATION

In order to investigate the effect of anodizing process on the mechanical behavior of A6061 alloy, a number of specimens were treated by means of sulfuric acid anodizing. Before the anodizing process, the samples were subjected to degreasing using Acetone and pickling sequences as defined in Table 2 in order to produce a chemically clean and decontaminated surface. According to the surface treatment applied to the samples, they were divided in different groups as summarized in Table. II.

Table. II
Surface pretreatment conditions

Groups	Pickling	Neutralization
G1	15 g / l of NaOH at 60°C for 30 s	910 g/l HNO_3 for 2 min
G2	15 g / l of NaOH at 60°C for 60 s	910 g/l HNO_3 for 2 min
G3	15 g / l of NaOH at 60°C for 90 s	910 g/l HNO_3 for 2 min
G4	15 g / l of NaOH at 60°C for 120 s	910 g/l HNO_3 for 2 min
G5	30 g / l of NaOH at 60°C for 30 s	910 g/l HNO_3 for 2 min
G6	30 g / l of NaOH at 60°C for 60 s	910 g/l HNO_3 for 2 min
G7	30 g / l of NaOH at 60°C for 90 s	910 g/l HNO_3 for 2 min
G8	30 g / l of NaOH at 60°C for 120 s	910 g/l HNO_3 for 2 min
G9	45 g / l of NaOH at 60°C for 30 s	910 g/l HNO_3 for 2 min
G10	45 g / l of NaOH at 60°C for 60 s	910 g/l HNO_3 for 2 min
G11	45 g / l of NaOH at 60°C for 90 s	910 g/l HNO_3 for 2 min
G12	45 g / l of NaOH at 60°C for 120 s	910 g/l HNO_3 for 2 min

Subsequently, the specimens without a natural oxide film were placed in a prepared sulfuric acid solution with a concentration of 20% under constant voltage at 20 V for 30 min. The aluminum sheet (anode) and the lead plate (cathode) were connected to the DC power supply. A schematic of the anodizing process is presented in Fig. 1.

All the specimens were anodized at 20 °C thanks to a thermostatically controlled electrochemical cell (±2 °C) and a continuous agitation to prevent local violent reactions and to keep initial electrolyte concentration and temperature.

After the anodizing process, the samples were immediately rinsed with deionized water to avoid acid attack and then sealed in boiling water at 97 °C for 30 min.

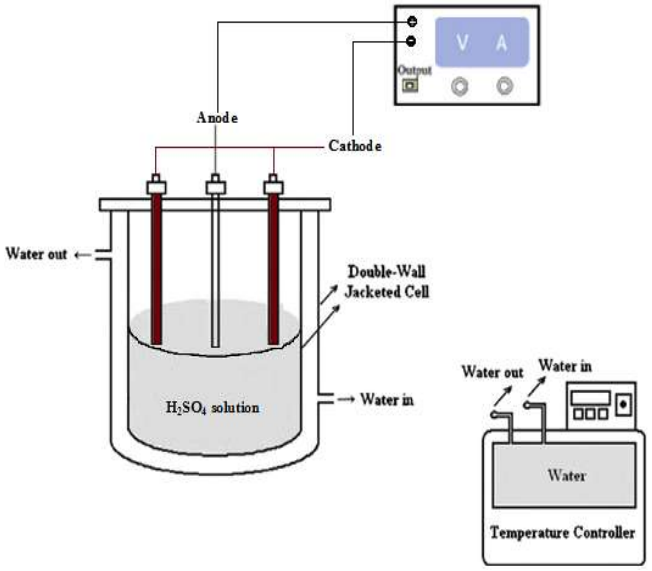

Fig. 1: Anodizing process

II.3. CHARACTERIZATION

II.3.1. METALLOGRAPHIC CHARACTERIZATION

The specimens were prepared using fine grinding, polishing and etched with Keller's reagent. The surface morphology and the thickness of the anodic films were examined using an optical microscope (Carl Zeiss-Axioteck 100).

II.3.2. MECHANICAL TESTING

Tensile strength testing is carried out using a MTS Criterion Model 45 X 100 KN machine with a strain rate of 1.2mm/min at room temperature.

Test specimens (Fig. 2) were machined according to the standards and specifications of ASTM E8 [15]

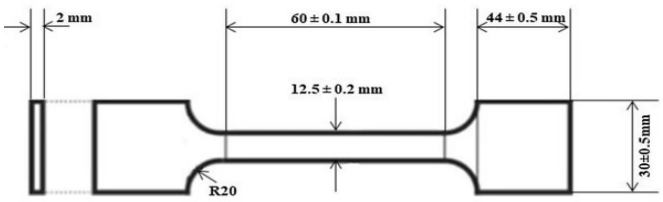

Fig. 2: Shape and dimension of tensile strength testing specimen

Vickers microhardness of the anodic layer was measured from the surface of the samples under a load of 0.5 N for 20 s. Each data point represents the mean of three values at least.

Surface roughness (Ra) was measured using RUGOSURF 10 G from three different regions of each sample with an evaluation length of 10 mm.

III. RESULTS AND DISCUSSION

III.1 Surface Morphology

Fig. 3 shows the relationship between the roughness of the pickled samples and the pickling parameters. It appears that samples pickled with 45g/l during 120s has a higher roughness compared to the others concentrations bath for the same time pickling.

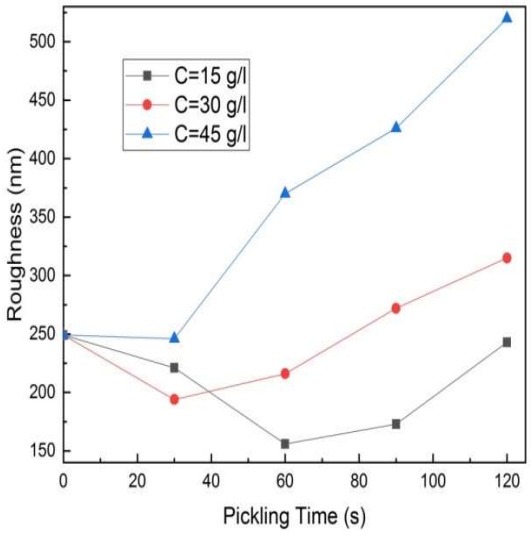

Fig. 3: Roughness of the substrate as function of pickling time and bath concentration

The effect of pickling treatment on A6061 substrate, slightly roughen the surface. Prolong the immersion time in the sodium hydroxide bath had slightly increased the surface roughness as shown in Fig.3. A long exposure to the caustic soda at high concentrations accelerates significantly the pickling process. This conclusion has been proven by other researchers [16].

To show the changes induced by the pickling treatment in the surface morphology of the substrate, optical microstructure images of A6061 as received and after pickling have been presented in Fig.4

The Fig.4 (a) shows the surface morphology of A6061before pickling. Figures 4 (b) and (c) reveal inhomogeneous morphologies which have the appearance of an eroded surface with random distribution of several small and large pits.

The curve of Fig. 5 shows the effect of the roughness of the substrate on the roughness of the anodic layer. It is clear that the roughness of the anodic layer increase with increasing the roughness of the substrate. This was due to the fact that on a rough surface, the localized current density is high; therefore, the electric field-assisted dissolution of the oxide is also high [17]. This could leads to the formation of a more porous anodic layer leading to high rough anodic layer. This result is supported by P. F. A. Bijlmer's results [18] which confirm that the anodic layer reproduce the roughness of the substrate in the case of anodizing in sulphuric acid solutions.

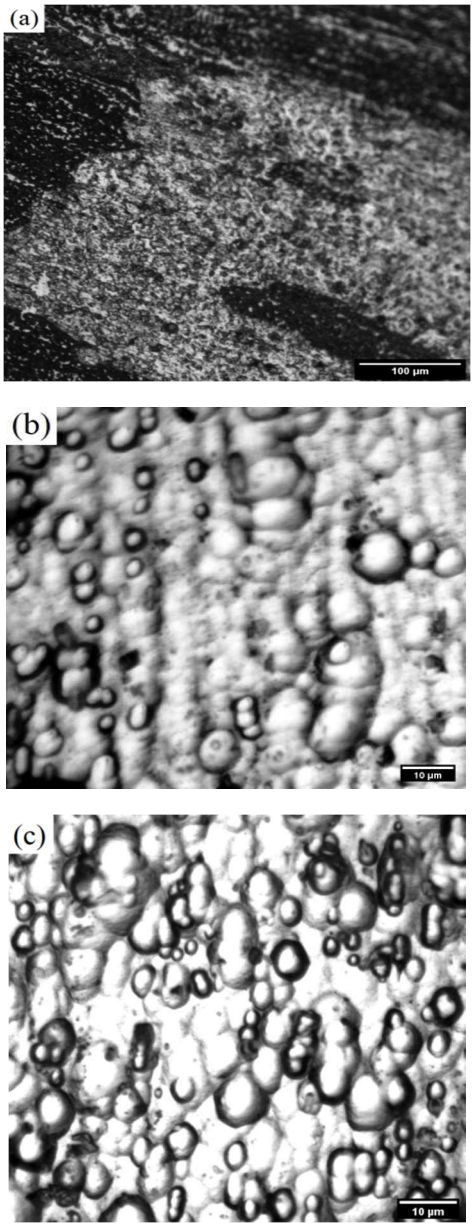

Fig. 4: Surface morphology of A6061-T6: (a) as received, (b) and (c) after pickling with 45g/l of NaOH during 60s and 120s respectively.

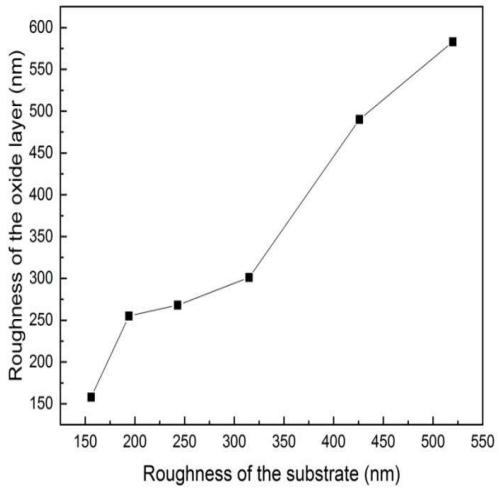

Fig. 5: Roughness of the anodic layer as function of substrate roughness

III.2 ANODIC LAYER THICKNESS

The thickness of the anodized layer depends on the roughness of A6061 after pickling .This dependences is presented in Fig. 6. The thickness of the anodic layer did change significantly with the substrate roughness. A significant increase in the anodic layer thickness until a roughness of 310 nm, and then a reduction has been noticed.

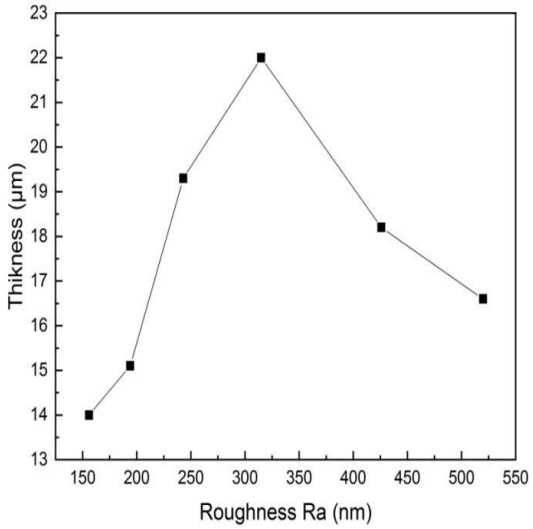

Fig. 6: Thickness of the anodic layer as function of substrate roughness

Fig.7 (a), (b) and (c) show an example for the anodic layer thickness measurement, of anodized A6061 formed on aluminum substrates having Ra value of 187, 310 and 521 um, respectively.

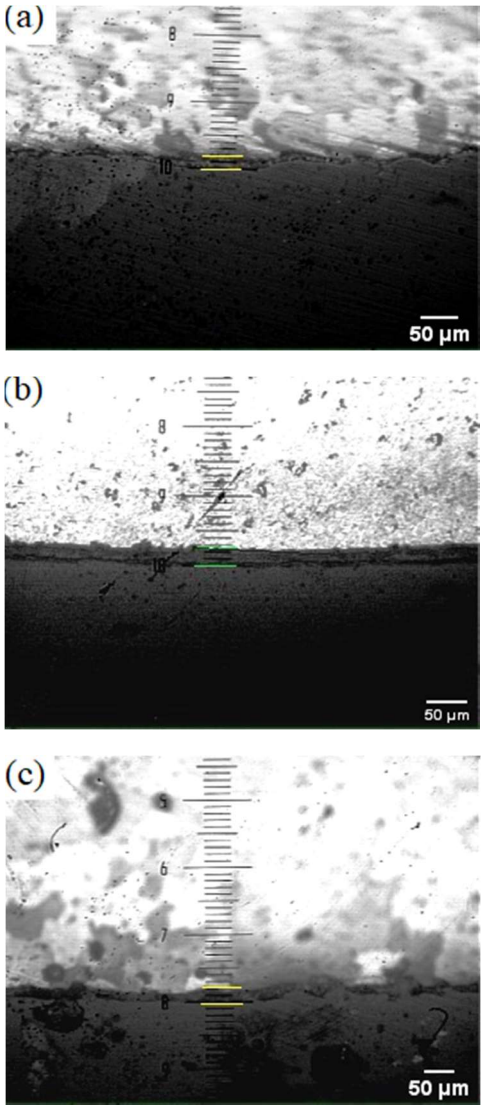

Fig. 7: Cross-sectional optical micrographs of anodized A6061 alloy after different surface preparation conditions: (a) pickling with 15g/l of NaOH during 60s, (b) pickling with 30g/l of NaOH during 120s and (c) pickling with 45g/l of NaOH during 120s

III.3 MICROHARDNESS EVOLUTION

The evolution of the microhardness as a function of the substrate roughness is represented in Fig. 8. In these analyses, the model of Jonsson and Hogmark [21] has been used to measure the microhardness of the anodic layer. This model uses a geometrical approach to combine the hardness of the anodic layer and of the substrate according to the area mixture model.

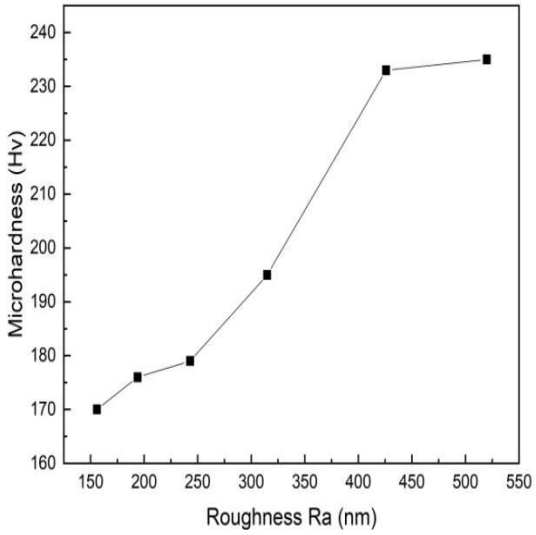

Fig. 8: Microhardness of the anodic layer as function of the substrate roughness

From Fig.8, it can be observed that the microhardness of the anodic layer increased significantly with increasing roughness of the substrate. The measured average microhardness values were in the range of 170 and 240 Hv.

The low microhardness of the anodic layer was attributed to the low O/Al ratio of the layer [22]. As there is lower oxygen content in the sample the film is more metallic and soft. According to R.K. Choudhary et al [19], the O/Al ratio of the anodic layer was found to be relatively high for the layer formed on a more rough aluminum surface and also resulted in increased hardness.

III.4 TENSILE PROPERTIES

Fig.9 shows the changes in the mechanical properties of A6061 alloy before and after anodization, as a function of the thickness of the anodic layer.

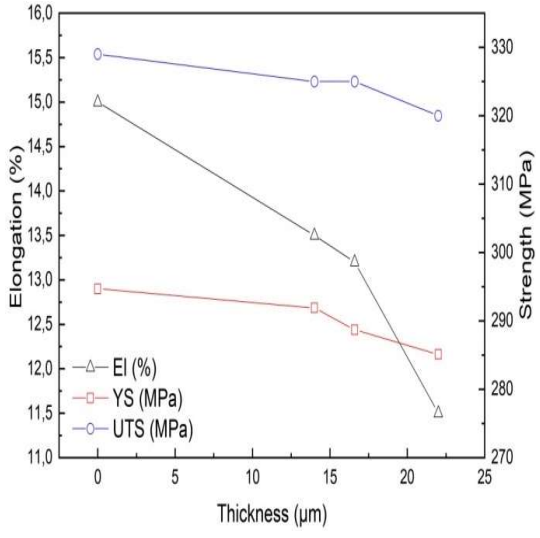

Fig. 9: Mechanical properties as a function of thickness of anodic layer

IV. CONCLUSION

In this paper, the influence of surface preparation on mechanical properties of A6061 anodized in sulfuric acid bath was investigated, and compared with those of A6061 without anodization. The results obtained were summarized below:

(1) - The roughness of the substrate A6061 increases with increasing time of pickling, and/or pickling bath concentration.

(2) - The thickness of the anodic layer and its roughness are directly related to the roughness of the substrate

(3) - The surface microhardness of the anodized A6061 is improved by increasing the thickness of the anodic layer

(4) - A slight decrease in yield strength, ultimate tensile strength and elongation compared to non-anodized A6061 alloy.

(5) - Mechanical proprieties of anodized A6061 decrease slightly with increasing anodic layer thickness.

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