Pedro Vergara Vera*
https://orcid.org/0000-0001-5914-8251

Oscar Magna**
https://orcid.org/0000-0002-0361-3553

Yohanna Palominos***
https://orcid.org/0000-0002-8520-1500

Ana Tapia****
https://orcid.org/0000-0003-4228-8885

Artículo

OPTIMIZATION OF CHEMICAL MILLING OF ALUMINUM FOR AIRCRAFT MANUFACTURING. AN EXPERIENCE IN REDUCING THE THICKNESS OF ALUMINUM SHEETS

OPTIMIZACIÓN DEL FRESADO QUÍMICO DE ALUMINIO PARA LA FABRICACIÓN DE AVIONES. UNA EXPERIENCIA EN LA REDUCCIÓN DEL ESPESOR DE LAS CHAPAS DE ALUMINIO

Received: June 23 2020 | Accepted: July 6 2020 | Final version: 26 de octubre de 2020

How to cite this article:
Vergara, P.; Magna, O.; Palominos, Y. y Tapia, A. (2020). Optimization of chemical milling of aluminum for aircraft manufacturing. An experience in reducing the thickness of aluminum sheets, *Trilogía, 33*(44), 12-25. Universidad Tecnológica Metropolitana.

* Doctor in Math-Science by Universitat Politècnica de Catalunya, Barcelona-Tech (UPC). Filiación institucional: Department of Statistics and Econometrics, Universidad Tecnológica Metropolitana (UTEM), Santiago de Chile. Correo electrónico: pvergara@utem.cl

**Doctor in BA & amp; Mgt. by Universitat Politècnica de Catalunya, Barcelona-Tech (UPC). Filiación institucional: Department of Informatics and Computing, Universidad Tecnológica Metropolitana (UTEM), Santiago de Chile. Correo electrónico:omagna@utem.cl

***Ing. Ejec. Geomensura, Universidad Santiago de Chile, Chile. Master in Industrial Design and Technological Bases, Jaume I University of Castellón, Barcelona, España. Filiación institucional: Department of Industry, Universidad Tecnológica Metropolitana (UTEM), Santiago de Chile. Correo electrónico: ypalominos@utem.cl.

****Master in Chemical Sciences of Universidad de Chile. Filiación institucional: Chemistry Department, Universidad Tecnológica Metropolitana (UTEM), Santiago de Chile. Correo electrónico:ana.tapia@utem.cl.

ABSTRACT

This paper presents an optimization model for aluminum plates by means of chemical milling to manufacture aircraft or parts thereof.

The aims of the experiments carried out were to obtain the best conditions to use aluminum plates, by chemically reducing their thickness in a selective and controlled manner according to the specifications of a piece.

From a factorial 2^4 design with 16 experiments and ensuring the maximum roughing depth (in mm), the most significant variables and interactions in the process were statistically determined, as well as the optimal conditions that maximize attack speed to achieve roughing in the shortest time possible. All of this using multiple regression models, response surfaces and contour lines.

The results make possible to reduce the thickness of the metal plates with the purpose of obtaining materials with dimensions not usually available from metal suppliers. By reducing the thickness of these machined components, part of the weight can be reduced as well, thus granting a longer flight range.

KEYWORDS, Aluminum Optimization, chemical Milling, aircraft alloy aluminum.

RESUMEN

Este artículo presenta un modelo de optimización para placas de aluminio mediante fresado químico para fabricar aeronaves o partes de ellas.

Los objetivos de los experimentos realizados fueron obtener las mejores condiciones para utilizar placas de aluminio, reduciendo químicamente su espesor de forma selectiva y controlada según la especificación de una pieza. Se realizaron experimentos utilizando un diseño factorial 2^4 con 16 experimentos para lograr la máxima profundidad de desbaste (en mm). A partir de los modelos de regresión múltiple obtenidos se determinó estadísticamente mediante superficies de respuesta y curvas de nivel qué variables e interacciones eran más significativas en el proceso, así como las condiciones óptimas que maximizan la velocidad de ataque para lograr el desbaste en el menor tiempo posible.

Los resultados permiten reducir el espesor de las planchas de metal para obtener el grosor adecuado de cada pieza, dado que estas dimensiones habitualmente no están disponibles por parte de los proveedores de metal, como también con la reducción del espesor de estos componentes mecanizados se logra reducir parte del peso permitiendo así una mayor autonomía de vuelo.

PALABRAS CLAVE: Optimización de aluminio, fresado químico, aleación de aluminio para aviones.

1. INTRODUCTION

Aluminum is the third most abundantly available element after oxygen and silicon, totaling approximately 8.3% of mass in the earth's crust (Greenwood, 1997; Loria, 2001). It is widely used in various fields, especially in the aeronautical industry.

Chemical milling is a non-traditional machining process, by which materials are removed in strong corrosive solutions and components with complex geometries and accurate dimensions can be machined as well (Çakir, 2008). In addition, McCallion presents and evaluates the use of chemical milling to complete a highly efficient and environmentally-benign manufacturing (An, Zhao and Tu, 2002). As a result, chemical milling is widely applied in the steel manufacturing or aluminum alloy products (Li, Wang and Hu, 2017).

The current study presents an optimized protocol for chemical milling, a surface treatment process frequently used in the aeronautical industry. This process is used for chemically etch aluminum sheets, resulting in a selective and controlled decrease in thickness.

Lightening the weight of aeronautical parts is fundamental for the industry as the end-product (i.e. aircraft) needs to be supported by motors that are placed under increasing performance requirements to meet modern-day travel demands.

The National Aeronautical Company of Chile (Empresa Nacional de Aeronáutica de Chile, ENAER) designs, manufactures and repairs civil and military aircraft. In recent years, the company has invested in cutting-edge technology to improve the design and manufacturing processes of aircraft and parts thereof.

1.1 Process description

Chemical milling (Rikhtegar, Shabestari and Saghafian, 2015; Trda, Ocana and Grum, 2011) is a type of surface treatment that has the ability to decrease the thickness of an aluminum sheet through chemical etching. The aluminum sheets are plunged into an acidic solution containing the following:

A=Caustic soda	: 120	- 195 g/L
B=Sodium sulfide	: 6	- 10 g/L
C=Dissolved aluminum	: 20	- 80 g/L
D=Temperature	: 90	- 105 °C

The sheet is plunged into this solution for a specified number of minutes based on the etching rate, where the decreased thickness is determined in terms of mm/min.

This process is optimized when achieving the greatest etching depth within a given time. Currently, each component in the solution is measured and added as the median value allowed by regulations. This procedure can be improved.

All chemical-milling experiments, which required chemical baths, were carried out under laboratory conditions. The assays were performed in test tubes according to the following procedure:

1. The chemical milling solution (1 L) was prepared according to the compositions and temperatures provided in Table-1.
2. The aluminum sample was cleaned with a methyl ethyl ketone solution in order to remove any fats and oils.
3. The aluminum sample was cleaned with detergent and water.
4. The aluminum sample was dried in an oven for 10 min between 45 °C and 50 °C.
5. The aluminum sample was cooled on a drying rack for 5 min.
6. The initial weight and thickness of the aluminum sample was recorded.
7. The temperature of the chemical milling solution was verified, and then the aluminum sample was plunged into the solution for exactly 5 min. During submersion, the bath was agitated to ensure a homogenous etching on both faces of the sample.
8. The aluminum sample was rinsed off and neutralized with nitric acid, 50% (v/v) and dried for 5 min at 45-50 °C.
9. The final weight and thickness of the aluminum sample were registered.

1.2 Aluminum sample's description.

The Aluminum samples with the following specifications were used in the experimental assays:

• Sample material: Aluminum 2024-T3 BARE (a material used in the National Aeronautical Company of Chile).
"Known as the 'aircraft alloy', 2024 is heat-treatable aluminum alloy with copper as the primary alloying element, and magnesium. Its other metallic elements include iron, silicon, manganese and zinc, in descending order... [it] Can be machined to a high finish and... its corrosion resistance is fair. Due to its high strength and excellent fatigue resistance, 2024 it is commonly used on structures and components in the aviation and transportation industries" (Teknica4, 2020).

• Dimensions: 70 mm x 70 mm x 2.5 mm.

• Identification: Each sample was stamped with an identification number. All samples were also perforated (3 mm) on the central, superior surface.

1.3 Factors influencing the milling process.

The chemical milling bath had the following components:

• Caustic soda, or sodium hydroxide – a strong soluble base obtained via the electrolysis of brine. For chemical milling assays, the amount of caustic soda varied between 120 g/L and 195 g/L.

• Sodium sulfate aids in the precipitation of heavy metals, and in a better end-product in terms of roughness. For chemical milling assays, sodium sulfate concentration varied between 6 g/L and 10 g/L.

• Dissolved aluminum prevents aggressiveness of the bath during chemical etching. For technical reasons, the amount of dissolved aluminum varied between 20 g/L and 80 g/L. For this component, 6000-series pure aluminum was used.

• Temperature provides homogeneity to the chemical bath. The temperature was tested in a range of 90-105 °C.

For all solution preparations, 1000 mL of demineralized water was used as a constant, standardized variable.

In the process carried out in the National Aeronautical Company of Chile, the aluminum sample was not considered an influential factor, since the reaction of the aluminum sample to the chemical attack agents mentioned above is irrelevant for the behavior of the chemical grinding process.

Another important factor to consider is the exposure time. However, to achieve significant etching values, experimental times were standardized to 5 min. Within this time frame along with the etching depth, it was possible to determine the etching rate.

2. MATERIALS AND METHODS

2.1 Materials and Variables.

A deep throat micrometer was used in all the assays to determine the thickness of the aluminum samples. The characteristics of this micrometer are as follows: Units: inches / millimeters; Model: 389-711-30; Measurement range: 0-1 in; LCD Resolution: 0.00005 in; and Measurement precision: 0.0002 in.

Table 1. Variable levels used

Variable	Variables level		Units
	-1	**+1**	
A: Caustic soda	120	195	g/L
B: Sodium sulfide	6	10	g/L
C: Dissolved Aluminum	20	80	g/L
D: Temperature	90	105	°C

2.2 Method

To construct the design matrix, the variables were codified using the following transformation:

$$\text{Codified Variable}_i = \frac{\text{Variable unit}_i - \text{Variable average}_i}{\frac{\text{Range}_i}{2}}$$

Then:

- Caustic soda : $A = \frac{\frac{g}{L} - 157.5}{37.5}$
- Sodium sulfide : $B = \frac{\frac{g}{L} - 8}{2}$
- Dissolved Aluminum: $C = \frac{\frac{g}{L} - 50}{30}$
- Temperature : $D = \frac{(°C) - 97.5}{7.5}$

Therefore, the research was carried out using a 2^4 complete factorial design with 16 experiments (Almimi et al., 2008; Barra-Bucarei, Vergara and Cortes, 2016; Box et al., 2005; Carvajal, González and Lozano, 2011; Hwan and Lee, 2010; Ogronik et al., 2011; Pepió and Poló, 1990). Table-1 presents the factorial design and the etching depth for each of the 16 experiments. The order of the experiments was randomly determined. For the etching of each aluminum sample, the chemical milling baths were prepared according to the matrix in Table-2.

Table 2. Design matrix, etching depth

variables				Etching depth (mm)
A	**B**	**C**	**D**	
+1	+1	-1	-1	0.285
+1	-1	+1	-1	0.135
-1	+1	-1	+1	0.210
+1	-1	+1	+1	0.125
-1	+1	-1	-1	0.170
-1	-1	-1	+1	0.190
-1	-1	+1	-1	0.090
+1	+1	+1	-1	0.115
+1	+1	+1	+1	0.135
+1	-1	-1	-1	0.275
-1	+1	+1	+1	0.105
-1	-1	-1	-1	0.175
+1	+1	-1	+1	0.295
+1	-1	-1	+1	0.285
-1	+1	+1	-1	0.115
-1	-1	+1	+1	0.110

3. RESULTS

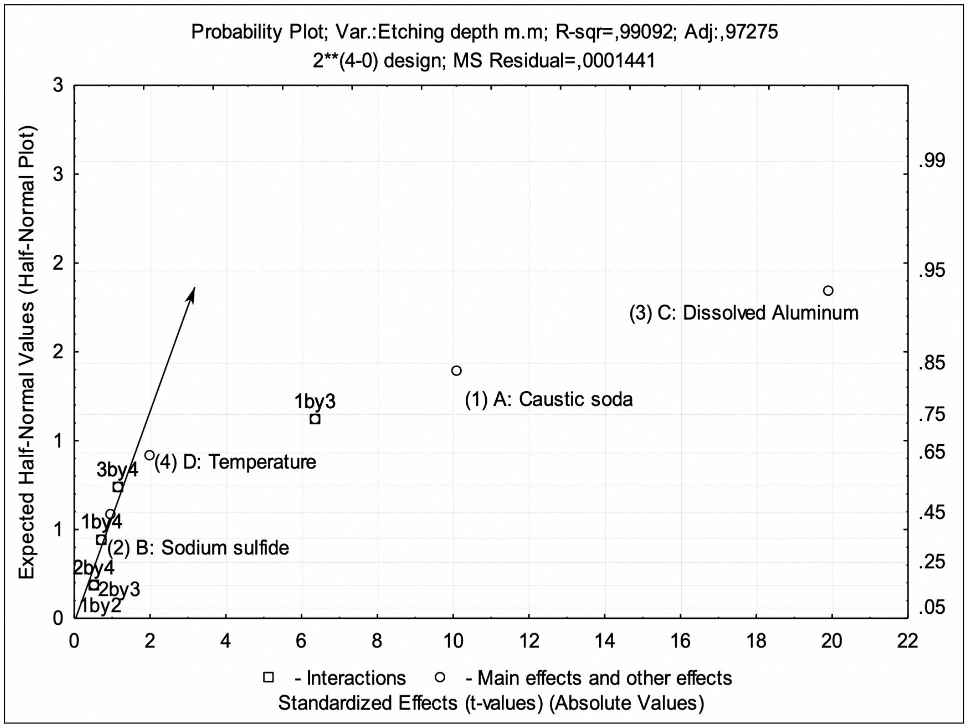
The effects and interactions of these variables were obtained as follows:

Table 3. Regression coefficients for etching depth

	Regression Coef.	Standard Error
Mean/Interc.	0.175938	0.003001
(1) Caustic soda	0.030313	0.003001
(2) Sodium sulfide	0.002813	0.003001
(3) Dissolved Aluminum	-0.059688	0.003001
(4) Temperature	0.005938	0.003001
Interaction 12	-0.001563	0.003001
Interaction 13	-0.019063	0.003001
Interaction 14	-0.002188	0.003001
Interaction 23	-0.001562	0.003001
Interaction 24	0.001563	0.003001
Interaction 34	-0.003438	0.003001

To determine the statistically significant variables and interactions, a half-normal probability plot was used. Their behavior is shown in Figure-1 (Barra-Bucarei et al., 2016; Ogronik et al., 2011; Pepió and Poló, 1990; Daniel, 1959; Guirguis, 2008 and Iyappan, 2015; Vergara; 2003; Vergara, Uribe and Cortés, 2013).

Figure 1. Half-normal probability plot



The most significant variable was dissolved aluminum, followed by caustic soda; then, the interaction between dissolved aluminum and caustic soda; and, finally, temperature.

To reduce the costs associated with chemical milling, the non-significant variables were considered at the lowest values. Specifically, sodium sulfide, set at a low amount (-1).

Therefore, the following model was obtained (Barra-Bucarei et al., 2016; Box, 2005; Ogrodnik et al., 201; Pepió and Poló, 1990; Iyappan, 2015; Hwan, 2015; Vergara; 2003; Vergara et al., 2013) to estimate etching depth:

$$y = 0.175938 + 0.030313 \cdot A - 0.059688 \cdot C - 0.019063 \cdot A \cdot C + 0.005938 \cdot D$$

Using a regression model, the corresponding surface response was determined (Barra-Bucarei et al., Vergara et al., 2016; Ogrodnik et al., 2011; Pepió and Poló, 1990; Iyappan, 2015; Chun and Ko, 2011; Nikman, 2015), thus defining the optimal conditions for chemical milling.

By setting temperature at its highest value (+1), the model is reduced to:

$$D = +1 \Rightarrow y = 0.181876 + 0.030313 \cdot A - 0.059688 \cdot C - 0.019063 \cdot A \cdot C$$

Figure-2 shows the corresponding surface response, while Figure-3 shows the model's level curves.

According to this result, to obtain the greatest etching depth, aluminum was set at a low concentration while caustic soda at a high concentration.

The calculation of the contour lines for variables A and C performed as follows:

• When temperature was considered at a low value (-1), the model was reduced to the following:

$$D = -1 \Rightarrow y = 0.17 + 0.030313 \cdot A - 0.059688 \cdot C - 0.019063 \cdot A \cdot C$$

The surface response (Figure-4) and level curves (Figure-5) of this model show the conditions needed to maximize etching depth.

Figure 2. Surface response with D = +1 for etching depth

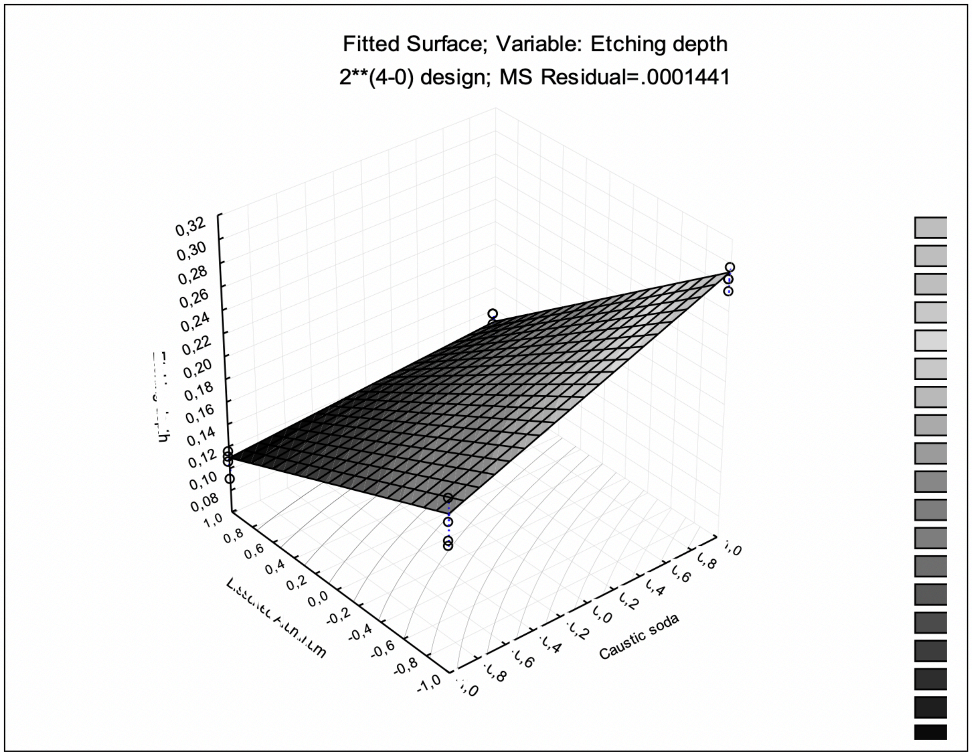
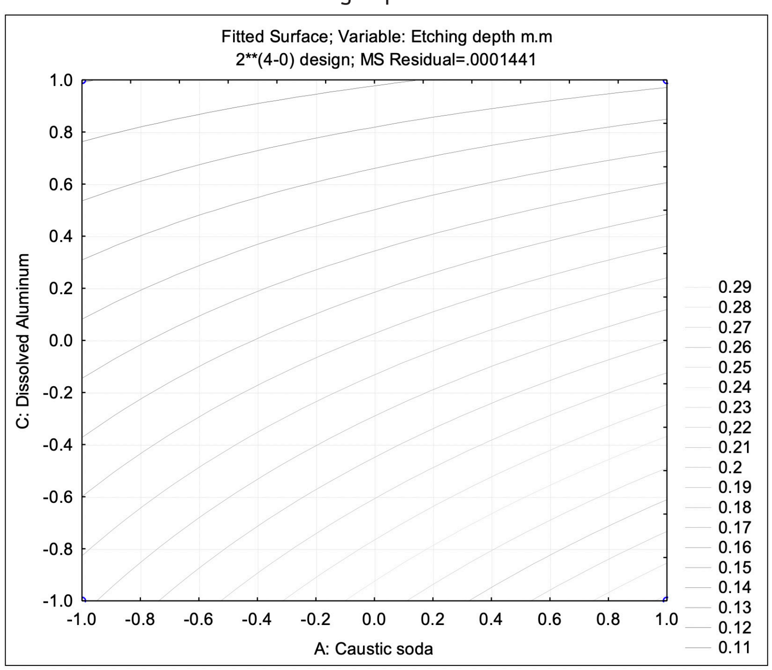
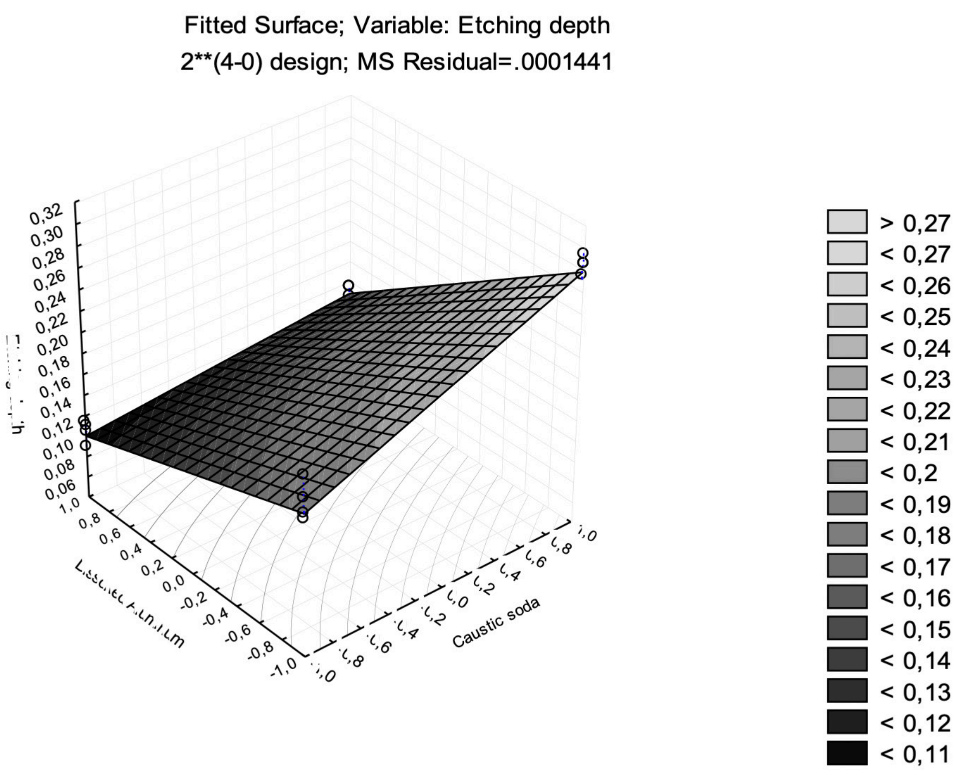


Figure 3. Level curves with D=+1 for etching depth



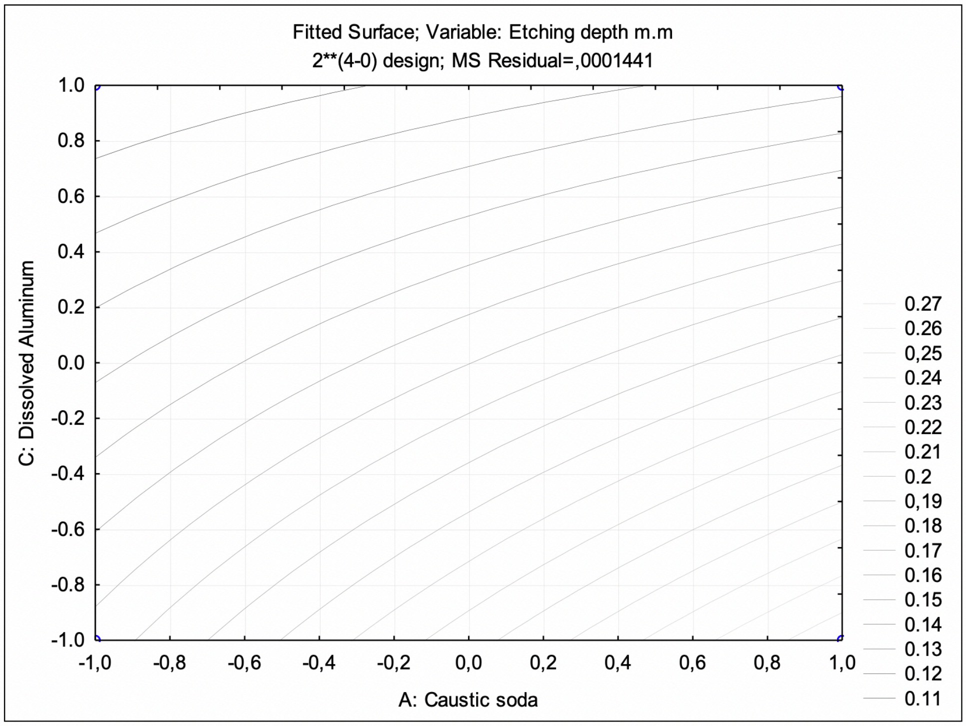
As in the prior model, to obtain the greatest etching depth, it was necessary to set dissolved aluminum low (-1) and caustic soda high (+1). Given the differences produced by each model, an engraving depth along with an optimal cost were obtained at low temperature.

Figure 4. Surface response with D = -1 for etching depth



Considering that dissolved aluminum prevents a violent chemical reaction, and that the greatest etching depth was obtained at a low dissolved aluminum content, over time (when aluminum accumulates in the solution), the solution is not affected by excessive aluminum contamination. Low dissolved aluminum content would also translate into reduced costs associated with chemical milling.

Figure 5. Level curves with D=-1 for etching depth



By setting a low amount of aluminum (C = -1) and low temperature (D = -1), which would result in reduced chemical milling costs, the model equation was reduced to only the behavior of caustic soda (A), as in:

$$y = 0.1229688 - 0.040625 \cdot A$$

Under this model, an interesting phenomenon occurred. Although initial dissolved aluminum content was expected to be low, over the course of chemical milling, additional aluminum was deposited by the etched aluminum parts.

Therefore, dissolved aluminum was considered a dynamic factor. By substituting low caustic soda and temperature in the initial model equation, the etching depth behavior became lineal and dependent on caustic soda, according to the following equation:

$$y = 0.175938 + 0.030313 \cdot A - 0.059688 \cdot C - 0.019063 \cdot A \cdot C + 0.005938 \cdot D$$

$$\mathrm{y} = 0.200313 - 0.078751 \cdot \mathrm{C}$$

Considering the variable C, this equation can be improved as follows:

$$y = 0.200313 - 0.078751 \cdot \frac{[g/L] - 50}{30}$$

then: $y = 0.33156 - 0.078751[g/L]$

4. CONCLUSIONS

The statistically significant variables in the model were dissolved aluminum, caustic sodium, the interaction between dissolved aluminum and caustic sodium, and temperature.

Considering the high amount of caustic soda, it was possible to determine the etching depth through the rate of aluminum deposition in the solution.

Related to this, it was possible to determine the etching rate or the needed exposure time to obtain the required thickness.

The chemical milling process, optimized to obtain the greatest etching depth with fixed process variables, was as follows:

- A: Caustic soda : High=195 g/L
- B: Sodium sulfate : Low = 6 g/L
- C: Dissolved aluminum : Low = 20 g/L
- D: Temperature : Low = 90 °C

The use of these values would cut down on the chemical milling process, as compared to the quantities normally used for each variable.

Through changes in dissolved aluminum a greater control of the milling process, a better control of the grinding process and thereby a better control of the etching rate regarding the amount of aluminum deposited in the solution can be achieved.

As sodium sulfate was not significant, this variable was set at a low concentration (6 g/L), thus lowering the chemical milling costs.

Table 4 shows the estimated engraving depths in relation to the variables considered in this study, ordered from greater to lesser engraving depth.

Table 4. Estimates

A: Caustic soda	C: Aluminum	D: Temperature	Etching depth - estimates [mm]
+1	-1	+1	0.291
+1	-1	-1	0.279
-1	-1	+1	0.192

As it can be observed, the greatest etching depths were obtained by setting dissolved aluminum at a low concentration, thus reducing costs, and by setting temperature high, with associated costs. By decreasing the etching depth, going from 0.291 mm to 0.279 mm, the temperature could be set within a low range (i.e. 90 °C). Through this modification an acceptable etching depth was achieved, lowering the costs associated with reducing the aluminum sheets thickness as much as possible.

In terms of research and experimentation lines, the proposed model and indications could be used in the chemistry, process control and optimization of aluminum parts in the automotive industry. In this industry, it could be applied in the machining of interior automotive components to achieve high-definition logos, surface textures and aluminum profiles in, for example, dials, instrument panels and instrument covers.

ACKNOWLEDGEMENTS

The authors thank the reviewers for their comments. The authors also thank the National Aeronautical Company of Chile for the support and the opportunity to use their facilities for the experiments.

DECLARATION OF COMPETING INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

REFERENCES

Almimi, A.; Kulahci, M. and Montgomery, D. (2008). Estimation of missing observations in two-level split-plot designs. *Qual. Reliab. Eng. Int.*, *24*(2), 127-152. https://doi.org/10.1002/qre.871.

An, M. Z.; Zhao, L. C. and Tu, Z. M. (2002). Graining technology by electrolytic etching on the surface of aluminum alloys. *Mater. Chem. Phys.*, *77*(1), 170-178. https://doi.org/10.1016/S0254-0584(01)00581-8.

Barra-Bucarei, L.; Vergara, P. and Cortés, A. (2016). Mass-production conditions of Metarhizium anisopliae (Metschn.). Sorokin 1883 optimized on different substrates. *Chilean Journal of Agricultural Research*, *76*(4), December. https://doi:10.4067/S0718-58392016000400008

Box, G.; Stuart Hunter, J. and Hunter, W. (2005). *Statistics for Experimenters: Design, Innovation, and Discovery* (2nd edition). New York, Unites States: Wiley-Interscience.

Çakir, O. (2008). Chemical etching of aluminum. *Journal of Materials Processing Technology*, *199*(1-3), 337-340. https://doi.org/10.1016/j.jmatprotec.2007.08.012.

Carvajal, R.; González, P. and Lozano, S. (2011). Research study of factors affecting difference between hole diameters in hybrid metal-composite drilling. Proceedings of the Institution of Mechanical Engineers, Part B. *Journal of Engineering Manufacture*, *225*(7), 991-1000. https://doi.org/10.1177/2041297510393444.

Chun, S-H. and Ko, T. J. (2011). Study on the response surface model of machining error in internal lathe boring. *Int. J. Precis. Eng. Manuf.*, *12*(2), 177-182.

Daniel C. (1959). Use of Half-Normal Plots in Interpreting Factorial Two-Level Experiments. *Technometrics*, *1*(4), 311-341.

Greenwood, N. and Earnshaw, A. (1997). *Chemistry of the Elements* (2nd edition). Oxford, United Kingdom: Butterworth-Heinemann. https://doi.org/10.1016/C2009-0-30414-6.

Guirguis, G. H. (2008). A Note on Computing the Probability and Critical Values for the Half-Normal Plot. *Technometrics*, *50*(2), 228-229.

Hwang, Y. and Lee, Ch. (2010). Surface roughness and cutting force prediction in MQL and wet turning process of AISI 1045 using design of experiments. *Journal of Mechanical Science and Technology*, *24*(8), 1669-1677. Recovered from: https://link.springer.com/article/10.1007/s12206-010-0522-1.

Hwang, S. (2015). Synthesis of aluminum oxide dispersed -Fe with nano sized grains by simple milling. *J Alloys Compd.*, (638), 136-140.

Iyappan, G. (2015). Comparison of Tribological Properties of MoS2 and Graphite-PTFE Coatings and its Impact on Machining of Aluminium by HSS End Mills. *Mater Manuf. Process.*, *30*(7), 912-920.

Li, Q.; Wang, J. and Hu, W. (2017). Optimizations of electric current assisted chemical milling condition of 2219 aluminum alloy. *Journal of Materials Processing Technology*, (249), 379-385. https://doi.org/10.1016/j.jmatprotec.2017.06.028.

Loria, E. (2001). Quo vadis gamma titanium aluminide. *Intermetallics*, *9*(12), 997-1001. https://doi.org/10.1016/S0966-9795(01)00064-4

Niknam, S. (2015). Milling burr formation, modeling and control: A review. *Proc. Inst. Mech. Eng. Part. B. J Eng Manuf.*, *229*(6), 893-909.

Ogrodnik, P.; Xu, W., Goodwin, M. and Bancroft, G. (2011). A theoretical investigation of the use of 2^K factorial analysis to determine the effects of dimensional manufacturing tolerances on the stability of hydrodynamic journal bearing systems. *Proc. Inst. Mech. Eng. Part. J. J Eng Tribol*, July. https://doi.org/10.1177/1350650111408180.

Pepió, M. and Poló, C. (1990). Planificación de Experiencias. *Publicaciones del Laboratorio de Estadística ETSEIT-UPC*, Terrassa.

Rikhtegar, F.; Shabestari, S. and Saghafian, H. (2015). The homogenizing of carbon nanotube dispersion in aluminium matrix nanocomposite using flake powder metallurgy and ball milling methods. *Powder Technology*, (280), 26-34.

Tecnika4 (2020). *Description of 2024 Aluminum Alloy*. Madrid, Spain: Tecnika4. Recovered from: https://www.teknika4.com/2024-aluminium-alloy

Trdan, U.; Ocana, J. and Grum, J. (2011). Surface Modification of Aluminium Alloys with Laser Shock processing. *Strojniski Vestnik - Journal of Mechanical Engineering*, *57*(5), 385-393.

Vergara P.; Uribe, E. and Cortés, A. (2013). Optimization of a quality model for CCA industrial impregnation of radiate pine. D. Don agricultural fencing stakes. *Wood Research*, *58*(1), 131-140.

Vergara, P. (2003). *Process Optimization and Quality Control*. Santiago de Chile: Ediciones Universidad Tecnológica Metropolitana.