Impostation of an experimental design for studying the optimization of artificial aging process for the aluminium alloys

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Abstract: *New area industry of Albania, in recent years, factories with closed cycle of production and processing of aluminium alloys are being targeted. In Albania about 25,000 tons of aluminum alloys per year are produced and there is a tendency to increase this production. This reality has encouraged us to undertake a number of studies with the primary objective for optimizing the parameters of the basic process of this industry. In this article we focus on the process of artificially aging aluminum alloys. From early and newer studies [2], the importance and delicacy of temperature-time parameters in the evolution of microstructure and in the mechanical properties of the final product are recognized and underlined. We have impost an experimental planning based on the Response Surface Method by choosing Central Composite Design to optimize the temperature and time parameters for the artificial aging with the objective of maximizing the process indicator - mechanical properties, hardness in our case. The plan includes 13 tests with 5 replicas in the center of the experiment, built with the help of Design-Expert DX7 and DX13 software. The focus of our attention was the assessment of the design, analysis of residuals and diagnostic diagrams and forms of presentation of results: mathematical model, 3D response surface, isocontours and effects of interaction between factors. In the future works we will present the experimental results of the optimization of the artificial aging process for different aluminium alloys produced in Albania.*

Keywords: *RSM, CCD, ALUMINIUM ALLOY, AGING, OPTIMIZATION, DESING EXPERT*

1. Introduction

On the production and processing industry of aluminum in Albania

Starting from the 90s and especially after the 2000s in Albania, many factories and companies have been set up with the main object of processing aluminum and its alloys, mainly for the production of aluminum profiles. Today there are several dozen of them. Some of the largest companies have included in their production scheme the foundry line for the production of aluminum alloys based on recycled materials. Each of these companies produces from the foundry approximately five thousand tons per year. The aluminum produced is processed almost entirely in the country. To respond to the challenges of meeting the ever-increasing demands of quality products as most of them are mainly destined for export to developed countries. This industry is turning its attention to the university unlike what happened before. To respond to this new industrial reality we have planned and are undertaking a series of researches starting with the optimization of the parameters of artificial aging of aluminum alloys.

On the importance of the process of artificial aging of aluminum alloys

The industry of production and processing of aluminum alloys in Albania is in the early stages of its development and as such feels the need for studies in support of it and especially for the optimization of key processes. One of the processes that gives the imprint to the mechanical properties of products made of aluminum alloys is that of artificial aging, as the final process of thermal processing. From earlier, but also newer studies, the importance and delicacy of artificial aging of aluminum alloys is recognized and underlined. Indeed, it is a process of strengthing oo the alloys by managing the transformation of a supersatureted solid solution. During this process, the formation and growth of precipitate particles of the second phases from the supersaturated solid solution occurs. It is a process that is controlled by the diffusion of atoms of dissolved elements and as such significantly influenced by the parameters of the process temperature and time, for a given chemical composition of the alloys and a optimized homogenization annealing. The characteristics of precipitated particles depend on the temperature and time parameters of artificial aging: size, distribution, relative quantities which determine the degree of strengthening of the alloy. Precisely for these reasons, we are undertaking a series of studies to optimize the parameters of the artificial aging process of aluminum alloys produced in Albania with the objective of maximizing mechanical properties, starting from hardness.

2. Choosing the Experiment Plan

Among the plans that offer optimization of the indicators we have chosen the Response Surface Method - **RSM**, and Central Composite Design - **CCD**. The CCD plan offers more experiment points compared to the simple factorial plan, while maintaining a minimum total number of tests. The CCD plane also offers the rotary property, which means that the same error is stored at a certain distance from the center of the experiment. The response surface method, as an optimization method, was developed in the 1950s and was first applied in the process industries (chemical, metallurgical, etc.). This method constitutes the optimization technique based on the programming of the most widespread and successful experiments even today. [5] Our e experimental plan was draft on recent industrial development in which was applied this experimental plan and also on the latest articles, scientific research regarding the experimental plan. [6]

3. Impostation of our CCD experimental plan

The primary objective of this study is to optimize the process of artificial aging of aluminum alloys, in order to maximize the mechanical properties, strength, tensile strength, hardness. For the construction of the plan we have chosen as the center of the experiment the value of the process parameters, temperature and aging time, approximate to the values applied by the manufacturers for some aluminum alloys: temperature $T = 185$ ^{o}C, time $t = 8h$. As a step value for these two parameters we have chosen: $\Delta T = 15$ ^{o}C, $\Delta t = 3h$. These step values provide approximately the same change / effect on the process indicator (hardness), considered different bibliographic sources (Fig.1).

Fig.1 *From application to software for imposting the experiment plan*

For the star points arm, based on the recommendation in the Desing Expert tutorial, we have accepted the value +/- √2 (α = +/- 1.4214).

By entering this data, for *Factor A - Temperature* and *Factor B - Time*, in the software Design - Expert (RSM Method, CCD Plan), we obtain the Experimental Plan Table (Tab.1). In the experimental plan table are presented according to the factor combination and according to the order of the performing tests: *run*.

Table 1: Experimental plan table for the experiment

	Std	Run	Block	Factor 1 A: Temperature ºC	Factor 2 B: Time h	Response 1 Hardness HBN
	5	1	Block 1	163.7867966	8	
	4	2	Block 1	200	11	
	1	3	Block 1	170	5	
	7	4	Block 1	185	3.757359313	
	8	5	Block 1	185	12.24264069	
	13	6	Block 1	185	8	
	3	7	Block 1	170	11	
	6	8	Block 1	206.2132034	8	
	12	9	Block 1	185	8	
	10	10	Block 1	185	8	
	11	11	Block 1	185	8	
	2	12	Block 1	200	5	
	9	13	Block 1	185	8	

In the experimental plan table all the tests are presented according to the combination of factors as well as according to the order of performing the tests (run). To demonstrate the method, we have obtained, for the indicator *"hardness-HRB"*, approximate results from various bibliographic sources and our unpublished works.

4. Analysis of Variance, ANOVA

The result of the analysis of variance (ANOVA) processed by the software is given in the following table (Tab.2)

Table 2: Anova for response surface quadratic model

Interpretation of ANOVA table for our ezperimental data: The Model F-value of 30.17 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case A, B, AB, A2, B2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model. The "Lack of Fit F-value" of 5.47 implies there is a 6.71% chance that a "Lack of Fit F-value" this large could occur due to noise. [1,2,3]

5. Analysis of residuals and diagnostic diagrams

Before we can see and evaluate the results, we have to verify/diagnose the model. The assumptions and assertions made in the theory of Analysis of Variance must be fulfilled by the results of the experiment. As an illustrative example, we bring one of the assumptions made, that regarding to the "normality of the random size of residuals". For this, in the following we are bringing, the dependence on our experiment as well as two versions of this behavior (when not fulfilled and when the assumption is fulfilled) offered by the tutorial of Design - Expert 7 and 13.

5.1 Normal probability plot of the studentized residuals to check for normality of residuals.
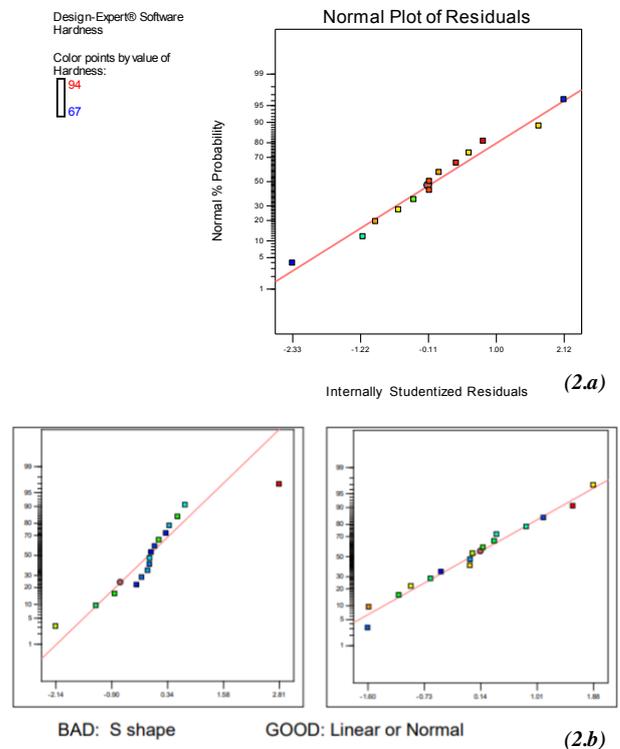

(2.a)

(2.b)

Fig. 2 *Normal Plot of residuals a) from our experiment b) from the DX software*

If the test points are placed in the form of an "S" the normality is not met (Fig. 2.b on the left) and when they are placed by random distribution around a straight line, the normality is met (Fig 2.b on the right). By carefully observing the graphs (Fig. 2a) it is concluded that in our experiment the assumption on the Normality of the random size of the residuals is fulfilled.

In the following we are bringing the diagnostic graphs for two other assumptions for which the same reasoning logic is followed also the graph for the power of transformation.

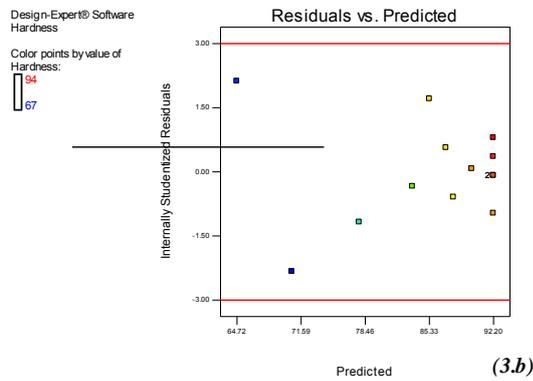*5.2 Studentized residuals versus predicted values to check for constant error.*

(3.a)
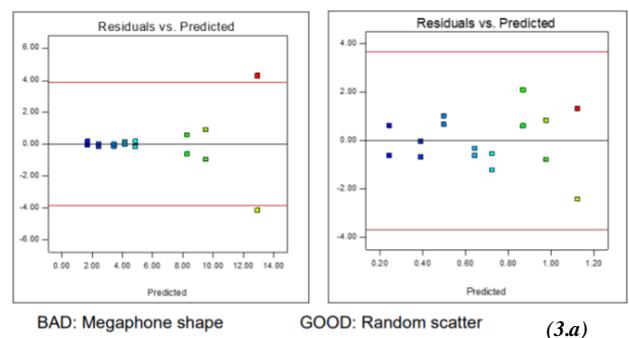
Fig. 3 *Residuals vs Predicted a) from the DX software b) from our experiment*

If the test points are distributed randomly, within the limit limits, the assumption is met and if the distribution gains certain geometry (megaphone in this case) the assumption is not met (Fig. 3.a). By carefully observing the graphs it is concluded that in our experiment the assumption on constant error is fulfilled (Fig. 3.b).

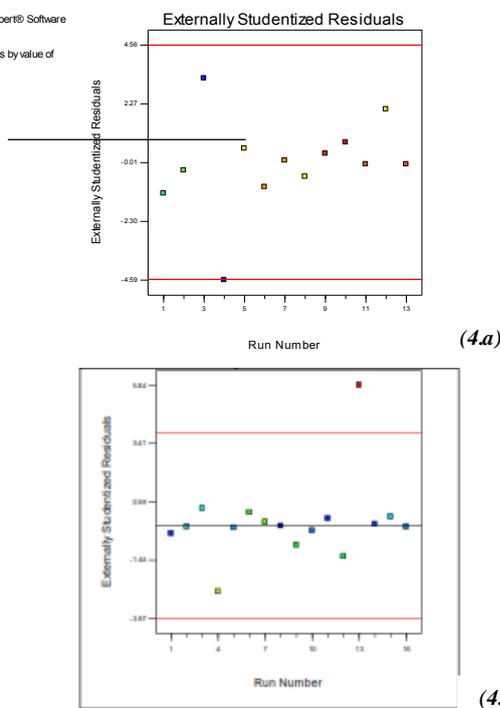*5.3 Externally Studentized Residuals to look for outliers, i.e., influential values.*
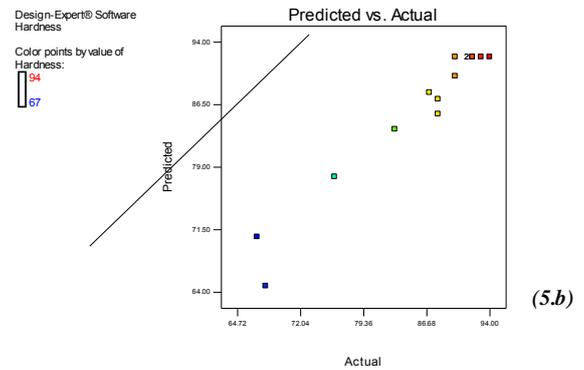

Fig. 4 *Externally Studentized Residuals vs Run : a) from our experiment b) from the DX software*

By carefully observing the graphs it is concluded that in our experiment the assumption on the affected values is fulfilled, we have no evidence points outside the limits (Fig.4.a).

5.4 Predicted vs. Actual
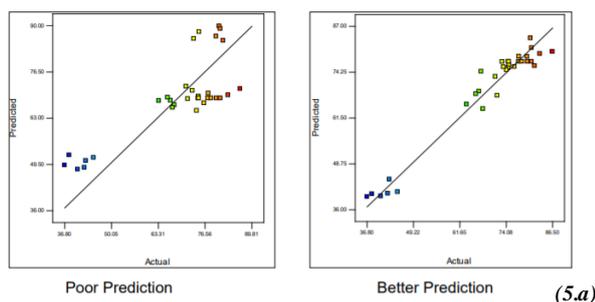
Fig. 5 *Predicted vs. Actual: a) from the DX software b) from our experiment*

The test points should be randomly distributed around the straight line at an angle of 45°, if clusters of points above or below the straight line are observed this indicates high or poor predictability (Fig. 5.a). By carefully observing the graphs it is concluded that in the experiment the predictability from the mathematical model is satisfactory (Fig. 5.b).

5.5 Box-Cox plot for power transformations.
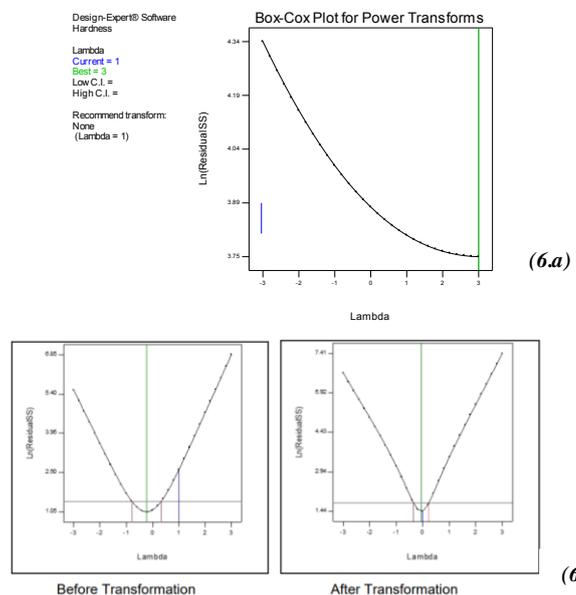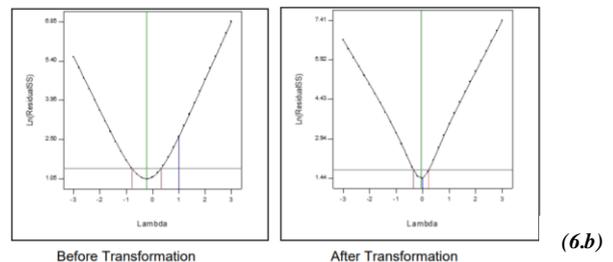
Fig. 6 *Box-Cox plot for power transformations a) from our experiment b) from the DX software*

When the ratio $y_{max}/y_{min} > 10$ usually indicates a transformation is required. For raport $y_{max}/y_{min} < 3$ the power transforms have little effect (Fig.6.a).

By carefully observing our experiment (Fig.6.b) the ratio is 1.4099 that means it is not necessary the power transform: $y^{(\lambda=1)}$.

6. Results

Below we are shown the data generated from the DX software:
   a. Mathematical model equations
   b. Response Surface Method - RSM
   c. Isocontours of the Indicator of Process (hardness, HBN)
   d. Effects of interaction between factors (temperature-time)

a) Mathematical model equations for our experiment

Mathematical model equations: is a quadratic polynomial which expresses the process indicator (hardness HBN) depending from the factors and the interaction between them.

Final Equation in Terms of Coded Factors it is:

$$Hardness = +92.20 + 3.57*A + 5.84*B - 6.75*A*B - 4.66*A^2 \; 6.66*B^2$$

Final Equation in Terms of Actual Factors it is:

$$Hardness = -945.98637 + 9.10519* A \; Temperature + 41.54021*B \; Time - 0.15000* A \; Temperature * B \; Time - 0.020722* A \; Temperature^2 - 0.74028* BTime^2$$

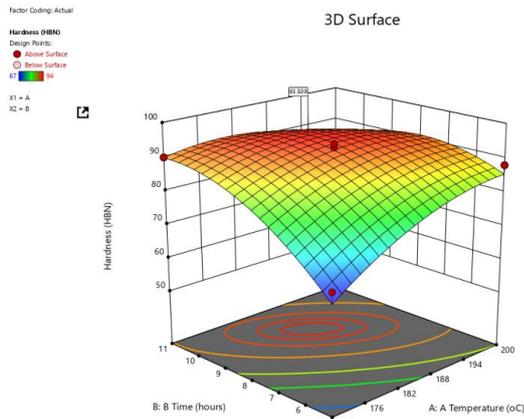*b) Response surface method (RSM)*

Fig. 7 *The response surface (3D) plot of Hardness vs Time-Temperature*

c) Isocontours of the Indicator of Process (hardness, HBN)
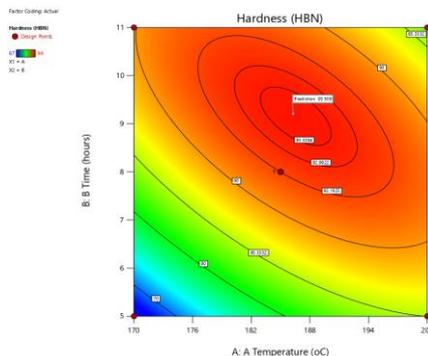

Fig. *The contour plot of Hardness (HBN) vs Time-Temperature*

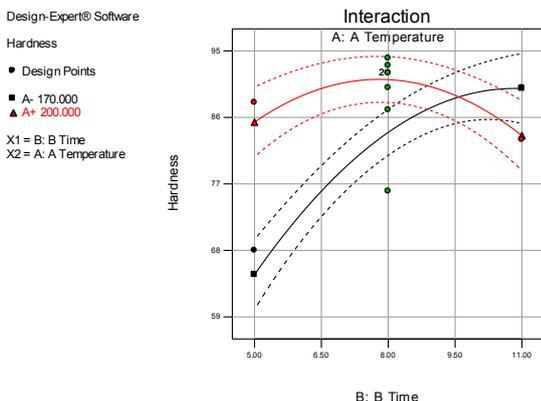d) Effects of interaction between factors *(temperature-time)*

Fig. 9 *Interaction between factors*

7. Conclusions

1. In this paper we have presented the methodology for the planning and analysis of the experiment for the case RSM-CCD applied in artificial aging of aluminum alloys.

2. Careful planning of the experiment ensures that the results are obtained with the right expectation, within low probability errors (p-values), with a minimum number of tests.

3. The RSM-CCD method provides in addition to the 3D graphical representation of the response surface also:

- *Mathematical model* of the dependence of the process indicator on the factors taken in the study
- The *effect of the interaction between the factors* and what is more important:
- *Isocontours* of the indicator depending on the factors within the studied limits.
- We consider the latter especially important for industrial operators as it serves them as a **technological card** for process management.
- We hope that this paper will be useful for various researchers who aim to optimize the parameters of a process, as we are convinced that it will serve us in future work to optimize the parameters of artificial aging of various connections of aluminium.

8. Acknowledgment

9. Conflict of interests

The author would like to confirm that there is no conflict of interests associated with this publication and there is no financial fund for this work that can affect the research outcomes.

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