

STUDY OF INFLUENCE OF COLD ROLLING AND ANNEALING ON ULTIMATE TENSILE STRENGTH OF AA2024 ALLOY USING BOX-BEHNKEN EXPERIMENTAL DESIGN

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Abstract

This study investigates the effects of thermo-mechanical processing parameters, specifically cold rolling and annealing, on the ultimate tensile strength (UTS) of AA2024 alloy, employing a Box-Behnken experimental design. The deformation during cold rolling, the annealing temperature, and the time annealing were varied to evaluate their individual and combined effects on the UTS. The results demonstrated that the UTS increased with deformation from 50% to 60%, but decreased with higher annealing temperatures (460°C - 500°C) and extended annealing times (10 to 30 minutes). Among the parameters, annealing time exhibited the most significant influence on UTS, followed by deformation level and annealing temperature. The optimized parameters - 60% deformation, 460°C annealing temperature, and 10 minutes time annealing - predicted a UTS of 628 MPa, with experimental verification confirming a 3% deviation. This research underscores the critical importance of controlling thermo-mechanical parameters to optimize the mechanical properties of AA2024 alloy, offering valuable insights for industrial applications.

Keywords: Aluminum alloy 2024; Box-Behnken experimental design; cold rolling.

1. Introduction

Recently, aluminum alloys, with their superior strength, technical properties, and load-bearing capabilities, have become more widely utilized than pure aluminum. Aluminum can alloy with elements such as Cu, Mg, Si, Mn, Zn, and Li to form solid solutions that are sensitive to heat treatment [1, 2]. Among these alloys, AA2024 alloy is one of the most commonly used across various industries.

AA2024 alloy, commonly referred to as an "aviation material", is highly valued for its high strength, excellent load-bearing capacity, corrosion resistance, and low specific gravity. It is widely applied in industries such as aerospace, automotive, electronics, and the military sector [3, 4]. Due to the unique operational requirements in military applications, components such as bullet bodies, thermobaric warheads, nozzles, and wind

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cones, manufactured from AA2024 alloy, require superior mechanical properties compared to standard parts. To ensure that AA2024 alloy meets these stringent specifications, it must undergo alloying, heat treatment, deformation, or a combination of these processes [5].

It has been shown that alloying, heat treatment, and thermo-mechanical processing significantly influence the mechanical properties of AA2024 alloy. A. Albiter *et al.* [6-9] observed that the alloy's maximum strength increased from 379 MPa to 480 MPa when alloyed with TiC and SiC. Similarly, K. G. Sagar *et al.* [10] found that the addition of Beryllium improved the UTS by up to 10.7%. Additionally, F. E. Garchani *et al.* [11-13] demonstrated that aging treatments could elevate the UTS of AA2024 alloy to 512 MPa. The combination of heat treatment and deformation has also been explored as an effective method to further enhance the strength of AA2024 alloy. Z. Zhu *et al.* [14] investigated the effects of continuous extrusion speed, with varying spindle speeds from 4 to 8 rpm, and heat treatment conditions on the mechanical properties of AA2024 alloy. Their results showed that elongation reached 12.93% and the tensile strength increased to 497.6 MPa. R. Luciana *et al.* successfully determined the superplastic behavior of AA2024 alloy, particularly under high-temperature conditions of 460°C and appropriate strain rates (ranging from $8 \cdot 10^{-4}$ to $1.5 \cdot 10^{-3} \text{ s}^{-1}$), achieving elongation to failure exceeding 200% and an average grain size of 5 - 8 μm . The superplastic properties of the AA2024 alloy samples were developed through a thermo-mechanical processing route, which included homogenization at 500°C for 8 hours, hot rolling with a 68% thickness reduction followed by water quenching, cold rolling with a 57% thickness reduction, rapid heating to 480°C for 15 minutes followed by water quenching, and stabilization annealing at 350°C for 30 minutes [15]. The combination of deformation and heat treatment in thermo-mechanical processing is widely recognized as an effective approach for enhancing both the tensile strength and ductility of AA2024 alloy. However, the complexity of these processing schemes requires precise control over numerous process parameters, making the evaluation and optimization of these parameters under specific thermo-mechanical conditions a challenging task.

This study aims to investigate the impact of thermo-mechanical processes on the strength of AA2024 alloy, with particular emphasis on the effects of key process variables such as cold deformation, annealing temperature, and annealing time on the alloy's UTS. Additionally, the Box-Behnken experimental design [16] has been used in this study to develop a mathematical model that facilitates a comprehensive assessment of both the individual and interactive effects of the process factors on UTS. The analysis of the obtained model provides valuable insights into the influence of cold rolling deformation,

annealing temperature, and annealing time on UTS, thereby enhancing the understanding of how each parameter contributes to the overall strength of the alloy. The model has been further validated for accuracy through comparison with experimental data under the predicted optimal conditions.

2. Methodology

2.1. Material

The AA2024 alloy rods, with a diameter of 40 mm, were produced at the X59 plant of the Z127 factory using the casting-extrusion process. The chemical composition of the alloy under investigation is shown in Table 1, while the mechanical properties are provided in Table 2.

Table 1. Chemical composition of experimental AA2024 alloy

AA2024	Chemical composition								
	Si	Fe	Cu	Mg	Mn	Cr	Zn	Impurities	Al
Sample	0.06	0.10	4.23	1.50	0.49	0.01	0.01	0.02	93.40

Table 2. Mechanical properties of AA2024 alloy before thermo-mechanical process

Alloy	Ultimate tensile strength (MPa)	Elongation (%)
AA2024	255	5

2.2. Thermo-mechanical processing

The thermo-mechanical processing route described previously in [15] enhances the superplasticity of AA2024 alloy by promoting the formation of a fine-grained microstructure. The processes of homogenization at 500°C for 8 hours and hot rolling with a 68% thickness reduction were implemented to ensure uniform distribution of alloying elements and reduce grain size. The subsequent stages of cold rolling with a 57% thickness reduction, rapid heating to 480°C, soaking for 15 minutes, water quenching, and stabilization annealing at 350°C for 30 minutes increased dislocation density, strengthening the material and refining the grain structure, thereby improving elongation and ductility of the AA2024 alloy.

However, to further improve the UTS and ductility of AA2024 alloy, we have proposed a modified thermo-mechanical processing route based on the approach in [15], as shown in Fig. 1. In this process, cold rolling was performed with a deformation level ranging from 50% to 60%, followed by annealing at temperatures ranging from 460°C to 500°C, soaking for 10 to 30 minutes, and water quenching.

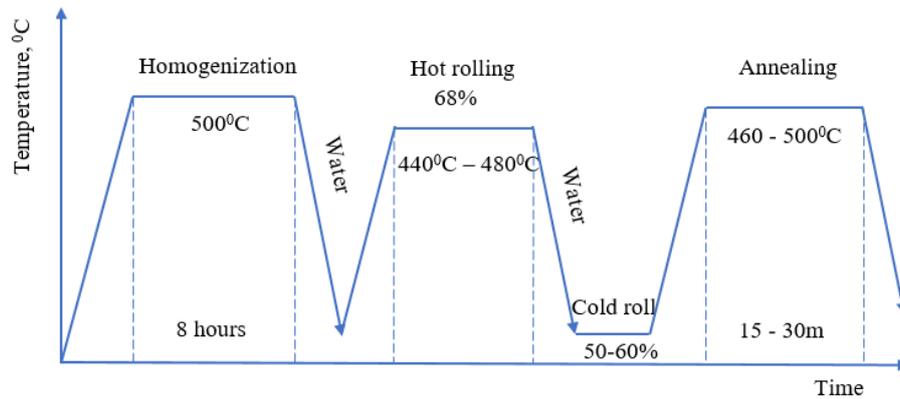


Fig. 1. Schematic diagram of thermo-mechanical processing of AA2024 alloy.

2.3. Experimental design

To investigate the effects of thermo-mechanical process parameters on the UTS of AA2024 alloy, an experimental design approach was employed. The Box-Behnken design (BBD) [16], a response surface methodology, was chosen for its efficiency in optimizing multiple factors while minimizing the number of experiments required.

Table 3. The Box-Behnken design matrix

No. Exp.	Encoding variable			Real variable		
	x_1	x_2	x_3	ϵ , %	T , °C	t , min
1	-	-	0	50	460	20
2	+	-	0	60	460	20
3	-	+	0	50	500	20
4	+	+	0	60	500	20
5	-	0	-	50	480	10
6	+	0	-	60	480	10
7	-	0	+	50	480	30
8	+	0	+	60	480	30
9	0	-	-	55	460	10
10	0	+	-	55	500	10
11	0	-	+	55	460	30
12	0	+	+	55	500	30
13	0	0	0	55	480	20
14	0	0	0	55	480	20
15	0	0	0	55	480	20

In this study, three key process variables were selected and coded as follows: Degree of deformation during cold rolling (coded as x_1), ranging from 50% to 60%; Annealing temperature (coded as x_2), ranging from 460°C to 500°C; Annealing time (coded as x_3), ranging from 10 to 30 minutes.

These variables were normalized and coded within the range of [-1, 1]. These variables were varied systematically according to the Box-Behnken design matrix, which includes 15 experimental runs. The design matrix, shown in Table 3, outlines the combination of factor levels for each experiment, ensuring an effective evaluation of the individual and interactive effects of the variables on the UTS.

The experimental results were analyzed using Modde 5.0 software, with non-significant coefficients (p -value > 0.05) excluded from the model. Regression analysis was employed to develop a mathematical model representing the relationship between the process parameters and the UTS. This model facilitates the optimization of process parameters to maximize tensile strength.

2.4. Sample preparation

The experimental samples for the thermo-mechanical processing were prepared as flat plates with dimensions of 4.5 mm in thickness, 15 mm in width, and 60 mm in length. These samples were cut from a cylindrical aluminum alloy bar with a diameter of 40 mm using a wire-cutting method. The 15 initial experimental samples, prepared according to the thermo-mechanical processing route, are presented in Fig. 2a.

Following the proposed thermo-mechanical processing sequence, the samples were first homogenized at 500°C for 8 hours, followed by water quenching. Subsequently, the hot rolling process was conducted in a unidirectional manner, achieving a 68% reduction in thickness, resulting in a 1.44 mm thick strip, which was also water quenched. The processed samples, as depicted in Fig. 2b, were then utilized for the Box-Behnken experimental design.

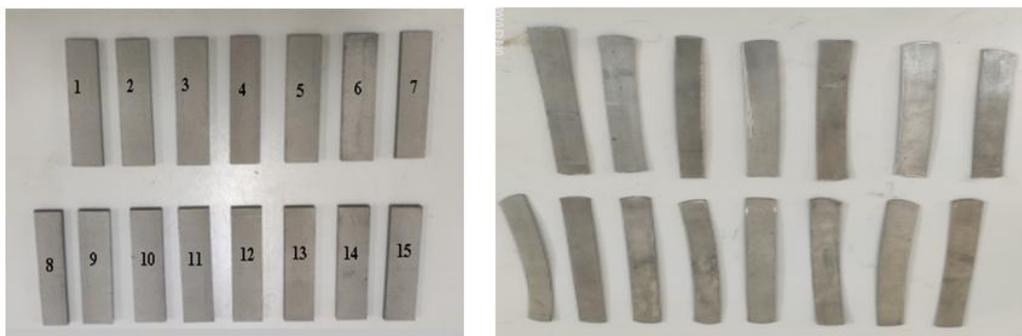


Fig. 2. Initial samples (a) hot rolled samples (b).

The tensile test specimens were prepared according to TCVN 197-1:2014 using a wire cutting method, with the specimen length aligned with the rolling direction. The dimensions of the tensile specimens are presented in Fig. 3.

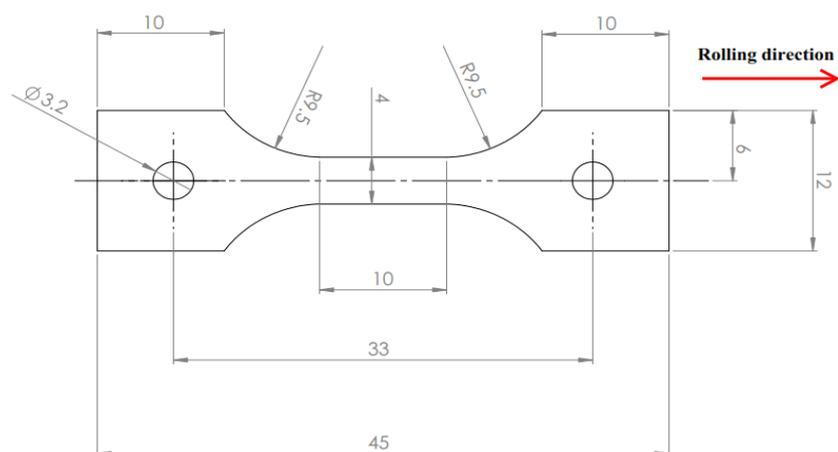


Fig 3. The tensile test specimens.

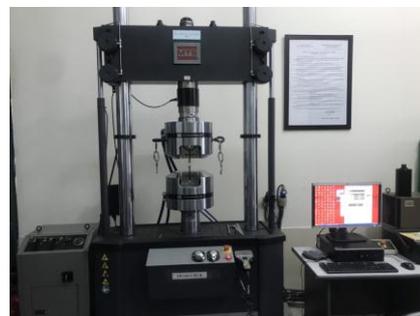
2.5. Laboratory equipment

A locally manufactured plate rolling mill with a roller diameter of 200 mm was used to perform the cold rolling process. All samples were cold rolled in multiple steps to achieve a total thickness reduction of 50%, from which 4 samples were selected for experiments No. 1, No. 3, No. 5, and No. 7. The remaining samples were further rolled to achieve a total thickness reduction of 55%, and 7 samples were selected for experiments No. 9 to No. 15. Finally, the remaining 4 samples were rolled to a total thickness reduction of 60% and were selected for experiments No. 2, No. 4, No. 6, and No. 8. The image of the rolling mill is shown in Fig. 4a.

A Nabertherm LH 120/13 chamber furnace (1300°C, 120L), manufactured in Germany, was utilized for the annealing process. The furnace chamber has internal dimensions of 500 × 500 × 500 mm.



a)



b)

Fig. 4. The sheet rolling mill (a) and Tensile testing MTS (b).

The tensile strength tests were conducted on an MTS 810 testing machine, manufactured in the USA, with a maximum force capacity of 500 kN. The image of the testing machine is shown in Fig. 4b. The tensile specimens were clamped into the machine's grips using a fixture, secured with a pin through a 3.2 mm diameter hole. The grips moved at a quasi-static test speed of 1.0 mm/min, continuing until the tensile specimens failed.

3. Results and discussion

The samples processed through rolling and annealing, based on the Box-Behnken experimental design, are presented in Fig. 5.



Fig. 5. Samples resulting from thermo-mechanical processing based on the Box-Behnken design.

The specimens before and after the uniaxial tensile test, along with the stress-strain graph of the tensile specimen from Experiment No. 1, are presented in Fig. 6.

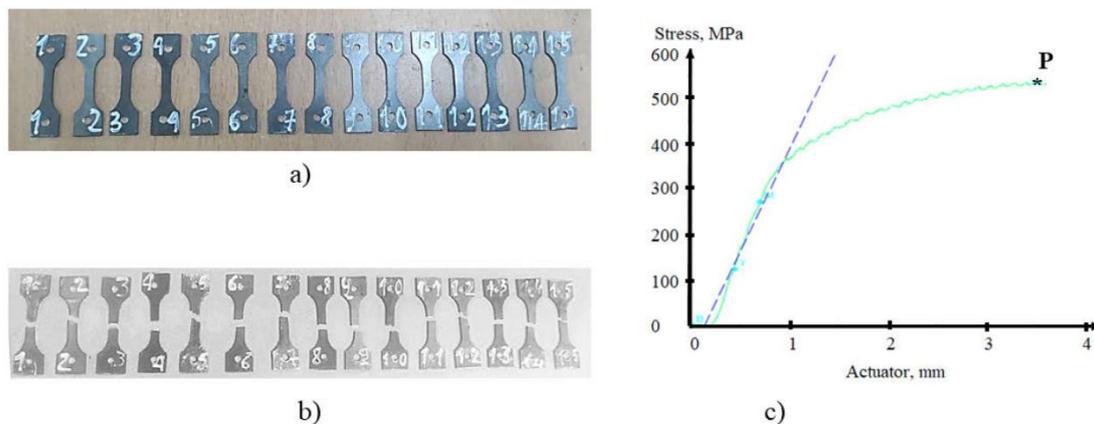


Fig. 6. Specimens before (a) and after (b) the tensile test, and the stress-stroke graph for experiment number 1 from the Box-Behnken experimental design (c).

The UTS results corresponding to the Box-Behnken design matrix are presented in Table 4.

Table 4. Tensile strength results from Box-Behnken design

No. Exp.	1	2	3	4	5	6	7	8
UTS, MPa	537.8	616.9	498.2	539.3	586.4	604.5	452.5	547.3
No. Exp.	9	10	11	12	13	14	15	
UTS, MPa	586.5	562.9	498.9	414.9	526.9	519.4	534.4	

The results of processing the experimental data from the Box-Behnken design using Modde 5.0 software are presented in Table 5. The full model achieved an R² value of 0.991, indicating that 99.1% of the variability in the response variable (UTS) is explained by the model. The adjusted R² (R² adj) was 0.975, reflecting a high degree of model fit even after accounting for the number of predictors. Three interaction terms $x_2 \cdot x_2$, $x_3 \cdot x_3$, and $x_1 \cdot x_2$ were excluded from the model due to their non-significance (p-value > 0.05), indicating that these terms did not have a statistically significant impact on tensile strength. The Q² value was 0.893, demonstrating the model's strong predictive power, with a confidence level set at 0.95. These results indicate that the model, after the removal of insignificant coefficients (Eq. (1)), maintains high accuracy and reliability in predicting the effects of process parameters on the tensile strength of AA2024 alloy.

Table 5. Results of the regression analysis

No.	1	2	3	4	5
1	Tensile strength	Coeff. SC	Std. Err.	P	Conf. int
2	Constant	526.900	5.02137	1.49057e-009	12.9079
3	x_1	29.1375	3.07495	0.000221187	7.90443
4	x_2	-28.1000	3.07495	0.000262905	7.90443
5	x_3	53.3375	3.07495	1.16678e-005	7.90443
6	$x_1 \cdot x_1$	26.5124	4.52620	0.00205517	11.6350
7	$x_2 \cdot x_2$	-5.36253	4.52620	0.28934700	11.6350
8	$x_3 \cdot x_3$	-5.73750	4.52620	0.26075900	11.6350
9	$x_1 \cdot x_2$	-9.50006	4.34863	0.08064290	11.1785
10	$x_1 \cdot x_3$	19.17500	4.34863	0.00696024	11.1785
11	$x_2 \cdot x_3$	-15.10000	4.34863	0.01780430	11.1785
12					
13	N = 15	Q ² = 0.893		Cond. No = 4.2385	
14	DF = 5	R ² = 0.991		Y-miss = 0	
15		R ² Adj. = 0.975		RSD = 8.6973	
16				Conf. lev = 0.95	

The obtained model has the form:

$$y = 526.9 + 29.14x_1 - 28.1x_2 - 53.34x_3 + 19.18x_1x_3 - 15.1x_2x_3 + 26.51x_1^2 \quad (1)$$

3.1. Effect degree of deformation on UTS

As observed in Fig. 7 and Fig. 8, the effect of the degree of deformation (ranging from 50% to 60%) on UTS follows a parabolic trend. Initially, as the degree of deformation increases from 50% to 54%, there is a slight decrease in UTS. However, as the deformation level further increases from 54% to 60%, UTS rises significantly. This behavior is a direct consequence of the work-hardening introduced during cold rolling, where dislocations accumulate and strengthen the material. The reversal of the trend, with UTS increasing beyond 54% deformation, indicates that the work-hardening effect becomes dominant over the softening caused by annealing, leading to enhanced strength.

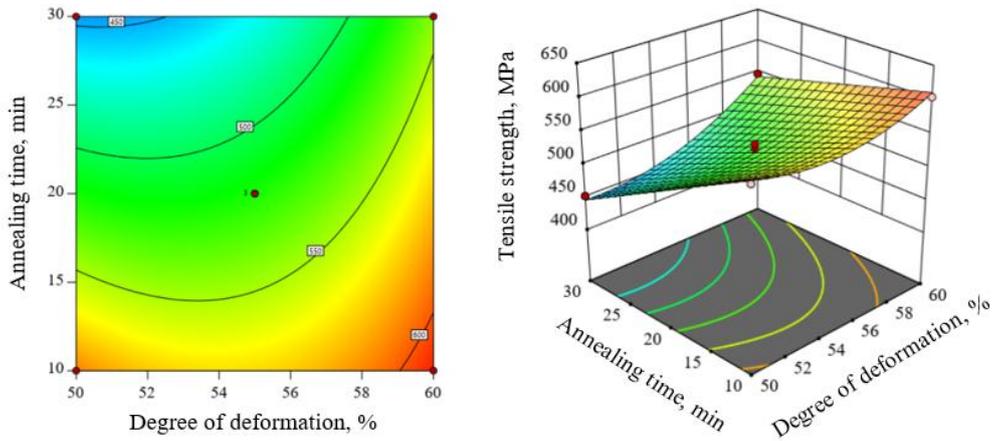


Fig. 7. Effects of the annealing time and degree of deformation on tensile strength of AA2024 alloy.

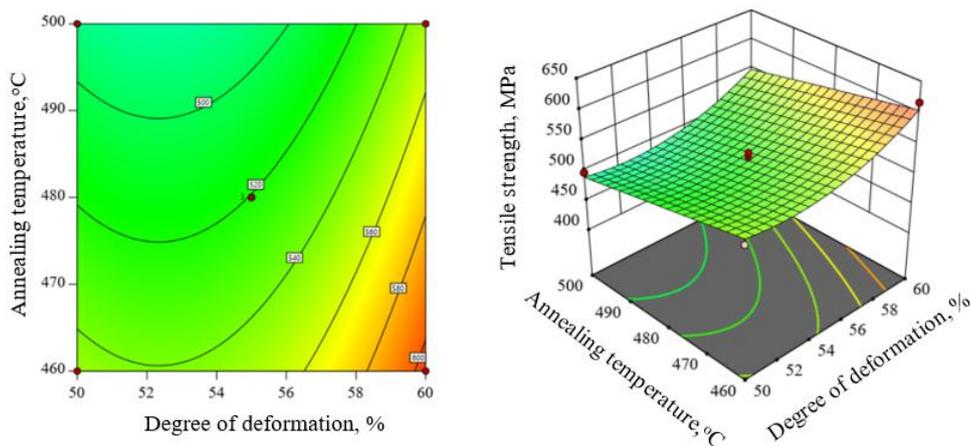


Fig. 8. Effects of the annealing temperature and degree of deformation on tensile strength of AA2024 alloy.

3.2. Effect of annealing temperature and time on UTS

Figure 9 demonstrates the combined influence of annealing temperature and annealing time on the UTS of the material. The data show that as the annealing temperature increases from 460°C to 500°C, there is a slight decrease in UTS, indicating that temperature alone has a relatively modest effect on the alloy's strength. However, this decrease becomes more pronounced when combined with longer annealing times, suggesting that time plays a more significant role in affecting UTS.

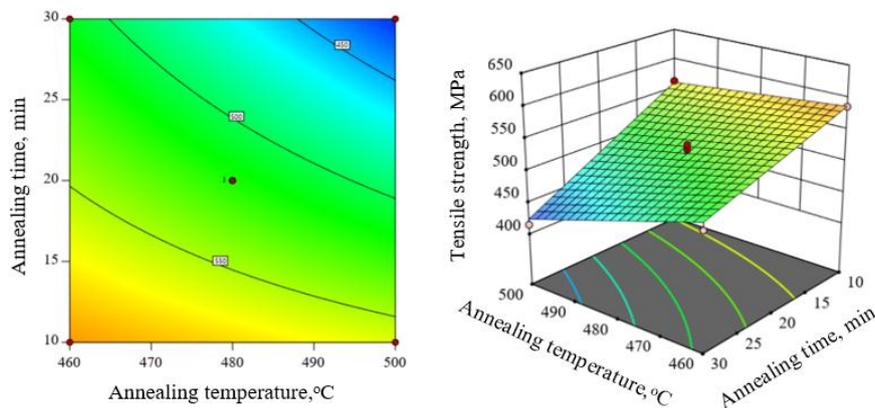


Fig. 9. Effects of the annealing time and annealing temperature on tensile strength of AA2024 alloy.

While both temperature and time influence on UTS, the annealing time appears to have a stronger impact. As annealing time increases from 10 to 30 minutes, UTS exhibits a marked reduction. This behavior is attributed to the fact that prolonged annealing encourages recrystallization, which occurs more rapidly at higher temperatures. The process of recrystallization not only refines the grain structure but also eliminates dislocations that had been introduced during cold working. Dislocations are essential contributors to work-hardening, which enhances the strength of the material. By removing these dislocations, recrystallization reduces the work-hardened state of the alloy, leading to a decline in UTS. In this study, it is evident that the accelerated recrystallization process under elevated temperatures and prolonged annealing leads to a reduction in dislocation density, which further softens the material.

Furthermore, grain coarsening with prolonged annealing time has a detrimental effect on the material's mechanical properties. Fine grains are typically associated with higher strength and improved ductility because they provide more barriers to dislocation movement. In contrast, coarser grains allow for easier dislocation movement, which weakens the material and decreases its tensile strength. Therefore, while annealing is necessary to restore some of the ductility lost during cold working, care must be taken to avoid excessive grain growth that compromises the mechanical performance of the alloy.

The influence of the thermo-mechanical process parameters on UTS in this study can be quantified using the following formula [16]:

$$X_i^* = \frac{1}{4} \left| \sum x_i^+ \hat{y}_i^+ + \sum x_i^- \hat{y}_i^- \right| \tag{2}$$

where x_i ($i = 1, 2, 3$) represents the coded variables, with "+" indicating the high level ($x_i = 1$) and "-" indicating the low level ($x_i = -1$).

From the data presented in Table 4, the relative impact of the thermo-mechanical parameters on UTS can be determined

$$X_1^* = 58.3; X_2^* = 56.2; X_3^* = 106.7 \tag{3}$$

The results indicate that annealing time has the most significant influence on UTS, followed by the deformation level, and lastly, the annealing temperature.

The behavior observed in this study, particularly the balance between strength and ductility, is consistent with findings from several studies on the thermo-mechanical processing of AA2024 alloy [14, 15] and AA7075 alloy (equivalent to B95 GOST 4784-97) [17, 18]. While the results align with existing research, it is important to acknowledge that variations in alloy composition, initial microstructure, and processing conditions can result in different UTS outcomes. Nevertheless, the trends identified in this study highlight the critical importance of carefully controlling deformation, annealing temperature, and annealing time to optimize the mechanical properties of AA2024 alloy.

3.3. Optimal prediction and experimental verification

The optimal thermo-mechanical processing conditions was presented in Table 6. Based on the trends observed in this study, the maximum predicted UTS of 628 MPa is achieved under the following optimal conditions: degree of deformation 60%, annealing at a temperature of 460°C, and an annealing time of 30 minutes.

Table 6. Determination of the optimal thermo-mechanical processing parameters for maximum tensile strength

No.	1	2	3	4	5	6
	Degree of deformation	Annealing temperature	Annealing time	Tensile strength	iter	Log (D)
1	50	473.469	10	591.621	247	0.4531
2	50	477.605	10.0001	591.39	165	0.4592
3	60	460	10	628.112	0	-10
4	59.999	471.464	10.0002	619.587	150	-1.1887
5	59.4529	460	10.2561	619.987	286	-1.2613
6	60	460	15	614.787	17	-0.6021
7	50	473.379	10	591.621	5002	0.4531
8	59.9465	470.11	10.5405	618.333	32	-0.9935

The tensile test specimen, processed under the optimal conditions with a 60% strain level, had a final thickness of 0.58 mm. Experimental results, verified with these optimal parameters, indicated a UTS of 608.8 MPa, as shown in Fig. 10. When compared to the maximum predicted UTS from the model (Eq. (1)) derived using the Box-Behnken experimental design, the deviation was approximately 3%. This slight deviation demonstrates the reliability and accuracy of the developed model in predicting the UTS of AA2024 alloy when subjected to the proposed thermo-mechanical processing scheme.

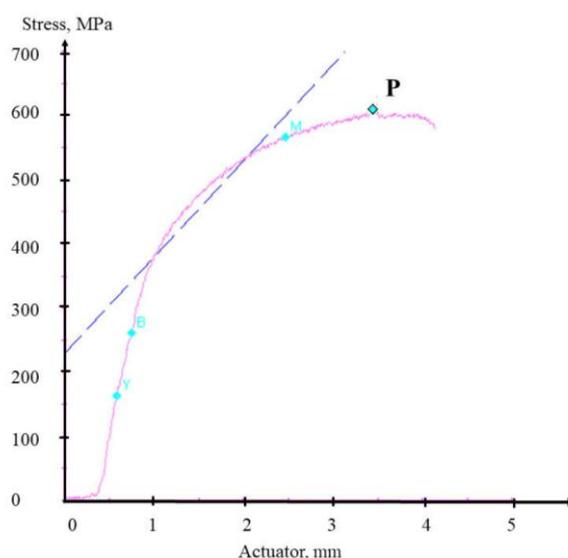


Fig. 10. Experimental verification of optimal thermo-mechanical processing parameters.

4. Conclusion

This study investigated the influence of thermo-mechanical processing parameters: degree of deformation, annealing temperature, and annealing time - on the UTS of AA2024 aluminum alloy. Using a systematic Box-Behnken experimental design, the optimal conditions were determined to be 60% deformation, annealing at 460°C, and a holding time of 10 minutes, which predicted a UTS of 628 MPa. Experimental verification resulted in a UTS of 608.8 MPa, with a deviation of only 3%, confirming the accuracy and reliability of the developed regression model.

The findings emphasize that annealing time exerts the most significant effect on UTS, followed by deformation level and annealing temperature. The model accurately predicts UTS across various thermo-mechanical conditions, underscoring the critical importance of precise control over processing parameters. These insights contribute valuable knowledge for optimizing the mechanical properties of AA2024 alloy, particularly in industries that require high-strength materials.

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NGHIÊN CỨU ẢNH HƯỞNG CỦA CÁN NGUỘI VÀ Ủ ĐẾN GIỚI HẠN BỀN KÉO CỦA HỢP KIM AA2024 SỬ DỤNG QUY HOẠCH THỰC NGHIỆM BOX-BEHNKEN

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Tóm tắt: Nghiên cứu này đánh giá ảnh hưởng của các thông số gia công nhiệt cơ, cụ thể là cán nguội và ủ, đến giới hạn bền kéo cực đại (UTS) của hợp kim nhôm AA2024, sử dụng quy hoạch thực nghiệm Box-Behnken. Mức độ biến dạng, nhiệt độ ủ và thời gian giữ nhiệt được thay đổi để đánh giá tác động riêng lẻ và kết hợp của chúng đến UTS. Kết quả chứng minh rằng việc tăng biến dạng từ 50% đến 60% làm tăng UTS, trong khi nhiệt độ ủ cao hơn (460°C đến 500°C) và thời gian ủ kéo dài (10 đến 30 phút) dẫn đến giảm UTS. Trong số các thông số, thời gian ủ thể hiện ảnh hưởng đáng kể nhất đến UTS, tiếp theo là mức độ biến dạng và nhiệt độ ủ. Các thông số được tối ưu hóa - biến dạng 60%, nhiệt độ ủ 460°C và thời gian giữ 10 phút - dự đoán UTS là 628 MPa, với một kiểm chứng thực nghiệm xác nhận độ lệch 3%. Nghiên cứu này nhấn mạnh tầm quan trọng của việc kiểm soát các thông số nhiệt cơ học để tối ưu hóa các tính chất cơ học của hợp kim AA2024, cung cấp thông tin chi tiết có giá trị cho các ứng dụng công nghiệp.

Từ khóa: Hợp kim nhôm 2024; quy hoạch thực nghiệm Box-Behnken; cán nguội.

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