

Hardness Optimization Using Takeuchi and ANOVA Method for Al₂O₃ Nano-Coating by PLD Systems

Ayman M. Hassan^a, Ali A. Alwahib^{b,*}, Abbas K. Hussein^c, Isaac S. Najim^b, Omar S. Dahham^d, Ahmed A. Al-Amiery^e, A. Mindilf, Makram A. Fakhri^{b**}

^aLaser and Optoelectronics Engineering Department, Dijla University College, Baghdad, Iraq

^bCollege of Laser and Optoelectronics, University of Technology-Iraq, Baghdad, Iraq

^cMaterials Engineering collage, University of Technology-Iraq, Baghdad, Iraq

^dDepartment of Chemical Engineering, Faculty of Engineering, University of Baghdad, Baghdad, Iraq

^eAl-Ayen Scientific Research Center, Al-Ayen Iraqi University, AUIQ, P.O. Box: 64004, An Nasiriyah, Thi Qar, Iraq

^fPhysics Department, Faculty of Science, University of Jeddah, Jeddah, Saudi Arabia

*Corresponding author. E-mail: Ali.A.Alwahib@uotechnology.edu.iq

**Corresponding author. E-mail: Makram.a.fakhri@uotechnology.edu.iq

ABSTRACT

In this work, the hardness of aluminum oxide thin film was optimized using the pulsed laser deposition technique. The optimization process involved designing experiments based on more than one variable, rather than using the traditional one-variable-at-a-time (OVAT) method, which is less reliable and requires more effort and time to achieve the required optimization, or may be highly susceptible to failure. Three levels of Taguchi were defined for two inputs that were changed simultaneously. These values were 700, 800, and 900 mJ for the laser energy and 100, 200, and 300 for the number of laser pulses. The nano-indentation technology of hardness test was examined nine experiments. The nano-indentation technology showed that the hardness of the thin film decreased from 38.34 GPa to 14.17 GPa with an increase in the laser pulse energy from (700 mJ, 100) to (900 mJ, 300). The optimum case for the greatest response appeared at 38.34 GPa. The design of the experiments based on the Taguchi method aims to reduce the discrepancy between the conditions that cannot be controlled and the conditions that are beyond our control and unknowable, thus giving a high-quality product. Analysis of variance (ANOVA) showed that the number of pulses has the most effective variable on the hardness of aluminum oxide thin film by 81.254%. The regression equation was found to show the relationship between hardness and the variables of aluminum oxide thin film prepared by PLD.

Keywords: PLD, Hardness, Al₂O₃ Nano-coating, Taguchi method, ANOVA, DOE, Regression equation

1. INTRODUCTION

Hardness is a material property that determines how much a material can resist the change made by permanent plastic deformation. The hardness of the material depends mainly on the structural properties and chemical composition. Thin films with high hardness, such as aluminum oxide (Al₂O₃) thin films, are helpful in many technological applications because they extend the life of the device and protect it from external influences [1-3]. Al₂O₃ thin films are used in applications such as anti-reflection and passivation of solar cells and can withstand high temperatures [4-6]. Accordingly, improving the hardness of aluminum oxide is essential to obtain high-quality thin films [7-9].

Materials with high hardness, including Al₂O₃, are particularly useful in technological research and various industries because they are long-lasting. Solid materials can be used as a coating to protect the material, maintain the chemical stability of the material, withstand high temperatures, and provide protection from corrosion [10-12]. Therefore, the Al₂O₃ coating is of broad interest due to its high hardness. Hardness indicates a material's resistance to cuts, scratches, punctures, and corrosion [13-15]. In general, the hardness of the coating Al₂O₃ is measured in

various thicknesses by indentation. One important thing to consider is the type of substrate and deposition technique. The coating hardness depends on the coating's composition and consistency, especially for PLD. For example, in sol-gel technology, it is impossible to obtain a strong and cohesive layer [16-18]

There are many deposition techniques for preparing Al₂O₃ thin films, and the pulsed laser deposition (PLD) technique is the most famous. PLD is a type of physical vapor deposition (PVD) in which a laser having a narrow frequency bandwidth and high-power density is used as a source for the ablation of the desired material, as shown in Figure 1 [19-21]. PLD technology is highly flexible since the source (laser) is located outside the vacuum chamber. PLD technology can easily control the deposition process because the parameters can be easily controlled. The deposition parameters are divided into two parts, the laser parameters and the vacuum chamber parameters. The laser parameters are wavelength, number of pulses, laser energy, repetition rate, spot size, and pulse duration. In contrast, the parameters of the vacuum chamber are the substrate temperature, the pressure of the vacuum chamber, the distance between the target and the substrate, the angle

between the laser and the target, and the laser distance from the target [22-24].

When preparing Al₂O₃ thin films, researchers often use a standard method called (OVAT) to prepare the thin films. OVAT means that one variable change and the rest of the variables remain constant. This approach relies on luck, guesswork, and experience for its success [25-27]. In addition, this approach consumes time, material, and cost to reach the required goals. However, DOE based on more than one variable plays a vital role in influencing the performance of the process. Through this type of experiment, it is possible to understand the effect of variables. The impact of some variables on the outputs may be strong, some may have an average effect, and some may be weak or ineffective. Therefore, the goal of such a carefully designed experiment is to understand any variables that significantly affect the

performance and then determine the best variables to obtain the desired functional performance of the product. The importance of the DOE can be summarized as follows [28-30]: it reduces time, cost, and material consumption, and the relationship between process input and output can be understood. The best variables affecting the process can be identified. It is possible to obtain a stable product with good specifications. A mathematical relationship can be found between the inputs and the outputs. The process is defined as converting inputs into outputs, as shown in Figure 1. Inputs are parameters or process variables such as laser energy, number of pulses, or substrate temperature. While the results are characteristics such as hardness and are always referred to as responses. The controllable variables (X) can be easily changed during the experiment, and then select the optimal inputs for X to.

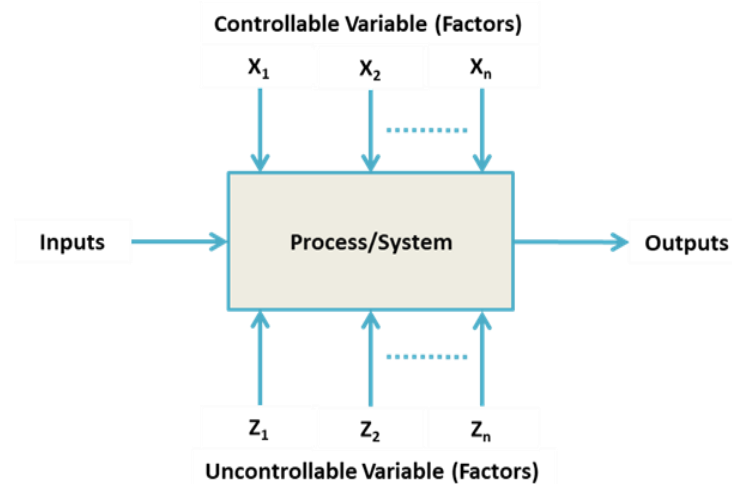


Figure 1. The general design of the process.

Table 1 indicates that the highest hardness reached about 28.8 GPa after C. Cibert et al. used the PLD technique to prepare Al₂O₃ thin films at 800 °C. It can also be noted that the highest hardness values are achieved when using the

PLD technique compared with other techniques. Also, all the literature surveys relied mainly on the standard OVAT method for blocking Al₂O₃ thin film. reduce the effects of uncontrollable variables(Z).

Table 1 Literature survey that studied the hardness of Al₂O₃ thin film

Authors	Technique	OVAT	Hardness
C. Cibert et al.[31]	PLD	Substrate temperature: 30°C and 800°C	6.4GPa and 28.8GPa
X. Wang et al.[32]	PLD	Laser Energy Range: 700mJ- 900mJ	Range: 18GPa- 24GPa
G. Balakrishnan et al. [33]	PLD	Substrate temperature Range: 30°C- 700°C	Range: 20.8GPa- 24.7GPa
Taivo J. et al.[34]	Atomic layer deposited (ALD)	Deposition scheme (by cycles) range: 1000 × Al ₂ O ₃ - 6 × (10 × Al ₂ O ₃ +200 × ZrO ₂)	Range: 11GPa–15 GPa
Lauri A. et al.[35]	ALD	temperature Range: 30°C- 700°C	Range: 13GPa–18GPa
Maria L. et al.[36]	sputter	Room temperature and Annealed temperature	Room temperature: 4.7GPa Annealed temperature: 10.5GPa
Kadhim R. et al.[37]	PLD	Substrate temperature range: 300°C- 500°C	Range: 22.7GPa- 6.164GPa
Z. Fu[38]	ALD	Temperature range: 150 °C- 700°C	Range: 9.1GPa- 17.1GPa

Sometimes a variable is used, for example, changing the temperature of a substrate to prepare thin films and fixing other variables such as laser energy and wavelength. But in fact, the effect of the substrate temperature may be weak on the preparation of thin films. Hence, the researcher resorts to changing another variable to reach the required results, which leads to the consumption of time, effort, and cost. To avoid problems with OVAT, it is necessary to conduct a design experiment (DOE) based on more than one variable. The importance of DOE lies in reducing material consumption, time, and cost to reach the required goals. It is also possible through DOE to understand the relationship between parameters (variables) and product characteristics. There are many methods for designing experiments, and the Taguchi method is the most common[39-42].

For the robust design of experiments, Taguchi's method is one of the leading methods in this field. The Taguchi method is a statistical method developed by the Japanese scientist Genichi Taguchi to improve the quality of products. The Taguchi method aims to eliminate the causes of poor quality and make product performance insensitive to variation in uncontrolled conditions[43-46]. The Taguchi method is based on the idea of reducing the variance between controllable and uncontrollable conditions and moving quality characteristics closer to the goal target. A target can

be described as achieving a product's or process's expected quality standards. If the product deviates from the target value, it will lead to quality losses [47-49].

In this research, for first-time aluminum oxide (Al₂O₃), nano-coating was designed based on the Taguchi and ANOVA method. Al₂O₃ was prepared using pulsed laser deposition technique. The design of the experiment and hardness results analysis based on the nano-indentation technology were discussed thoroughly in this paper.

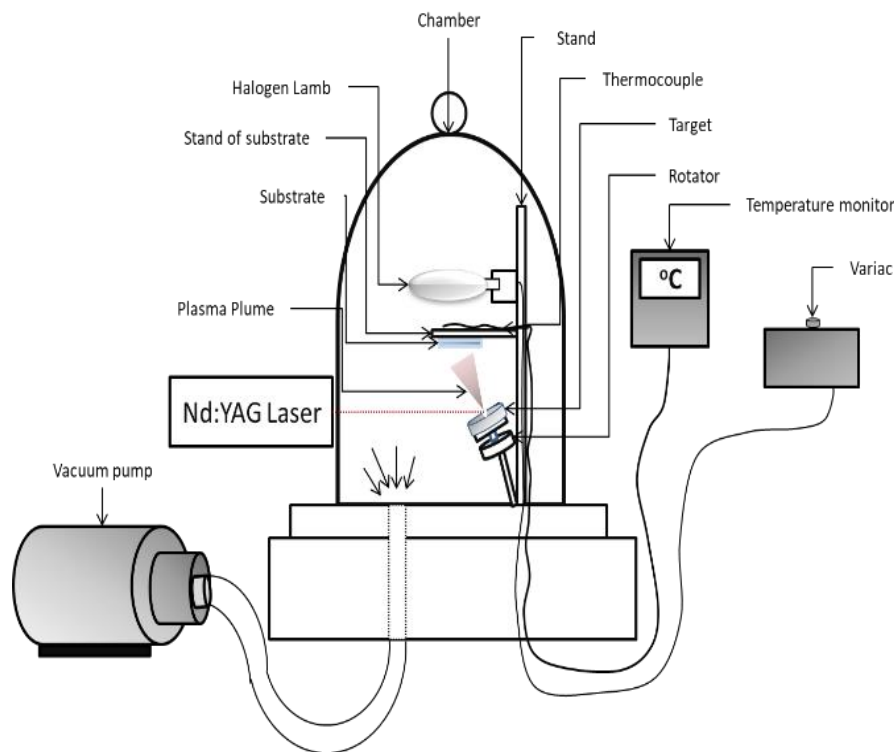
2. EXPERIMENTAL WORK

2.1 Al₂O₃ Deposition

The process of preparing the alumina thin film starts from Al₂O₃ Nano powder (particle size less than 40 nm and 99.9% purity) pressed by A hydraulic press device to form a solid target. Sixteen tons were applied to press 4g of Al₂O₃ powder in 3 minutes. A circular Al₂O₃ target with a diameter of 2 cm and 0.5 cm thickness is ready for PLD. PLD system consists of a group of important components, each component performing a specific function [(Table 2)]. These components are grouped together in one system called a PLD system [(Figure 2)].

Table 2 The function of the components of the PLD system

Components	Function
Q-switching Nd:YAG laser	It used to Ablate the target.
Variac	It used to control the temperature of the halogen lamp.
Halogen lamb	It is used to generate the required temperature of the substrate.
Thermocouple	It is used in substrate temperature sensing.
Temperature monitor	It is used to measure temperature (NO gas was insert inside the chamber)
Rotator	It is Used to rotate the target to improve the quality of the ablation.
Vacuum pump	It is used to empty the vacuum chamber.

**Figure 2.** Diagram of component of PLD System.

In this work, the target was placed in a vacuum chamber at 3 cm distance from the substrate, the pressure inside the chamber was 10^{-3} mbar. The deposition method was done using pulsed laser placed at 10 cm from the target and the angle of incidence between the target and the laser was 45° . Laser wavelength is 1064 nm (Nd-YAG), 1.8 mm spot diameter, 3Hz repetition rate, and 10 ns pulse duration. Various number of pulses, laser energy at temperature of substrate of 200°C were used to prepare the thin films samples. Laser power 700, 800, and 900 mJ, and the number of pulses 100, 200, and 300 were used.

2.2 Nano-Indentation Technology

Indentation Nano-indentation technology has been used for testing mechanical properties. In terms of working principle, coating hardness can be measured by increasing the force of the applied load on the thin film to reach a specific penetration depth and then unloading the applied load leaving an effective area on the coating surface as shown in Figure 3.

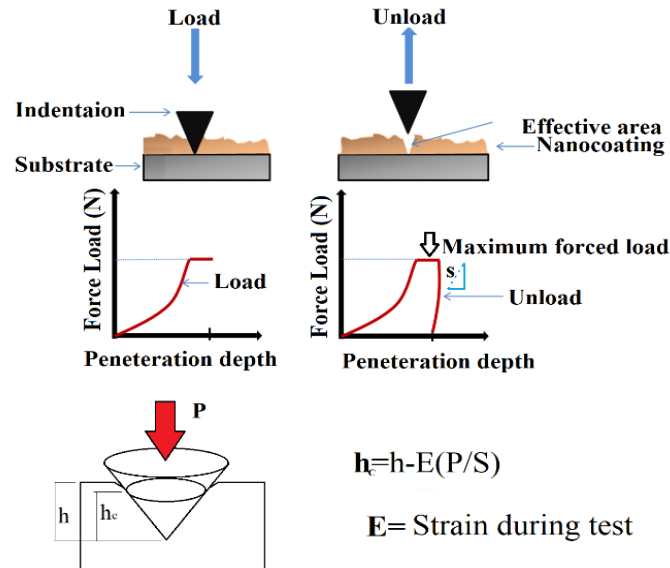


Figure 3. Principle work of Nano-indentation Technique.

The penetration depth was determined by a depth equal to the thickness of the thin film. The general hardness of the Al_2O_3 thin film was calculated by using following formula [50-53]:

$$H = \frac{F}{A} \quad (1)$$

2.3 Design of Experiment (DOE)

The experiments of this work were designed based on Taguchi method design. Laser energy, and the number of pulses were applied as an inputs in three levels [(Table 3)]. By using Minitab 19 software, analysis of variance (ANOVA) was performed to show the effect of each input on the hardness. The linear regression equations of the Al_2O_3 thin film hardness was found.

Table 3 Inputs and levels

Input	Symbol	Unit	Level 1	Level 2	Level 3
Energy laser	E	mJ	700	800	900
Number of pulses	P	-	100	200	300

The design relied on making interactions between the inputs to obtain the greatest diversity in the experiments to

prepare the Al_2O_3 thin films. Each level of laser energy input (700, 800, 900 mJ) interacts with all other levels of the number of pulses (100, 200, 300) [(Table 4)].

Table 4 DOE is based on Taguchi method to prepared of Al_2O_3 thin films

No.	E (mJ)	P (pulses)
1	700	100
2	700	200
3	700	300
4	800	100
5	800	200
6	800	300
7	900	100
8	900	200
9	900	300

The hardness test of the Al₂O₃ thin films on quartz substrates was carried out by using the nano-indentation technique. Quartz substrates have high hardness, therefore, they give high accuracy for testing the hardness of Al₂O₃ thin films compared to brittle or flexible substrates [54-57]. In this work, the maximum depth of indentation equal to the thickness of the Al₂O₃ thin film was determined. The thickness of the thin film can be described as the layer of Al₂O₃ deposited on quartz substrates using the PLD technique. The thickness of the Al₂O₃ thin film was measured using a Fizeau interferometer technique. The thickness of the Al₂O₃ thin film ranged between 40 nm and 162 nm.

3. RESULTS AND DISCUSSION

3.1 Hardness of Al₂O₃ Thin Film

Figure 4 shows that the maximum load applied to the Al₂O₃ thin films does not exceed 200 μ N, as the maximum load that was applied ranged between 198.9 μ N and 190.1 μ N. In Figure 4-i, the maximum load was achieved in a narrow range between load and unload. This indicates that the film is highly susceptible to wear, and this event is also repeated in Figure 4-c,e. On the contrary, it appears in Figure 4-a that the maximum load is large between load and unloads, and this indicates that the Al₂O₃ thin film is wear-resistant well. In Figure 4-b, a kind of stability in the load strength was achieved at a penetration depth ranging between 35 and 45 nm, and then the load begins to gradually increase. On the contrary, when the load was shed with increasing force and rapidly during the test, it will be similar to the load in Figure 4-c. This indicates a slow increase in the force of the applied load during the hardness test.

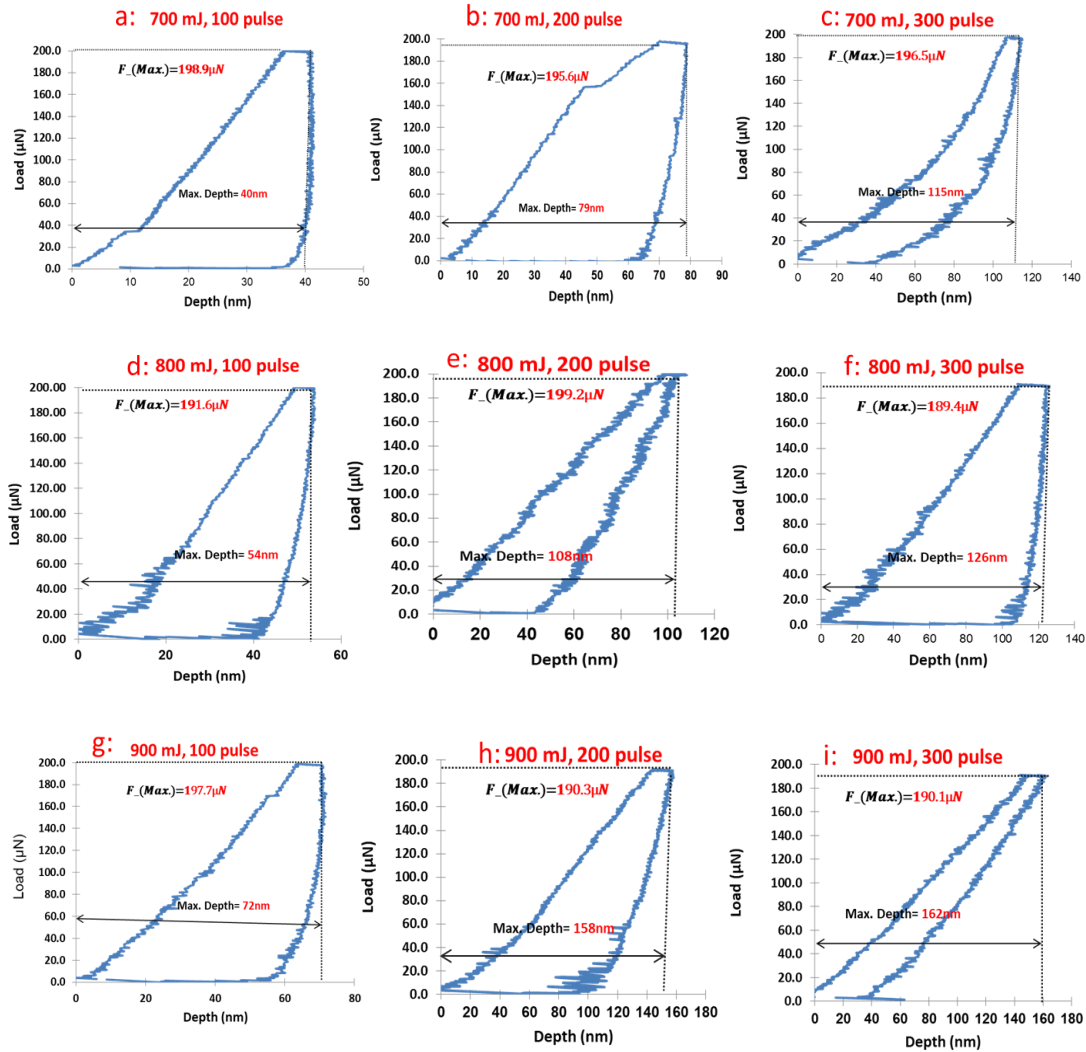


Figure 4. Hardness test-based nano-indentation technology of Al₂O₃ thin films deposited by PLD.

The indentation load was applied to the thin film of Al_2O_3 vertically to reach a selected depth. when indention unloads was left behind a trace on the surface of the film, the area of which is estimated at (A). The surface area (A) the Al_2O_3 thin film is negatively proportional to the hardness. From Figure 5, with the increased thickness of the Al_2O_3 thin film (maximum load), the area (A) formed on the surface increases. It is noted that at 40 nm, the lowest area (A) formed on the surface of the thin film of Al_2O_3 was about

5187.8 nm^2 , while the highest area (A) of about 13415.7 nm^2 was achieved at 162 nm. Often, when the thickness of the thin film increases, the defects of the film increase, including reduces its resistance to wear, as the thin film is less film tension [35, and 54-57]. Accordingly, the area (A) affected by indentation can easily shift in the case of large thickness thin film, and this explains why the area increases significantly at 162 nm compared to 40 nm.

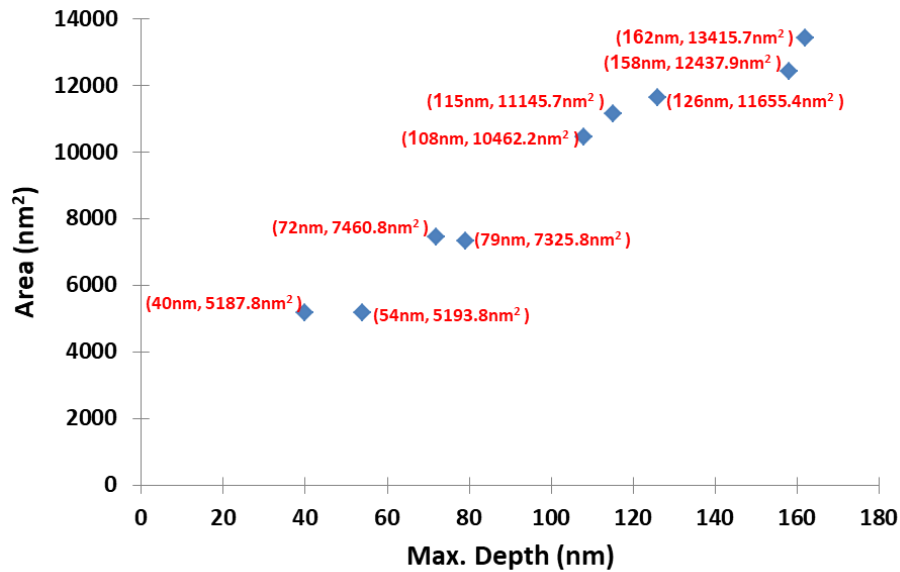


Figure 5. Relationship between Area (A) and Max. Depth.

3.2 ANOVA

ANOVA is a statistical process that aims to show the most influential variables in the process. Through ANOVA, it is possible to understand the performance of the Al_2O_3 thin films preparation process, and thus the possibility of making future improvements. Through ANOVA analysis, it is possible to pay great attention to the factors that have the most influence and to neglect the factors of least influence or to replace them with other factors to reach an ideal quality of the product. In ANOVA analysis, Contribution refers to the ratio of the sum of squares (SS) of each variable to the sum of squares (SS_{TOTAL}) as shown in the following formula [58-61]:

$$\text{Contribution (\%)} = \frac{SS}{SS_{\text{TOTAL}}} \quad (2)$$

In Table 6, it is noted that the sum of squares (SS) for the number of pulses is about 522.88, which is greater than the sum of squares SS for the laser energy, which is estimated at about 120.63. SS_{TOTAL} represents the sum of squares (SS) obtained for the variables of pulse number and laser energy. The number of pulses appears to be the factor that most affects the hardness of the Al_2O_3 thin film, with an estimated rate of about 81.254% compared to the laser energy, which is estimated at about 18.648%, which means that changing the number of pulses contributed greatly to improving the hardness in a very large way.

Table 6 ANOVA

Inputs	SS	Contribution%
Laser energy (E)	120.63	18.648
Number of pulses (P)	522.88	81.254
SS_{TOTAL}	643.51	

3.3. Regression Equation

Regression analysis is a statistical tool that builds a statistical model to estimate the relationship between variables (inputs) and a response (characteristic) so that a

statistical mathematical equation results. This equation can be used to predict future responses by using new variables. The deflection equations are divided into two parts, the simple regression equations, and the multiple regression equations.

This work used a multiple regression equation to use two variables represented by the number of pulses and laser energy. The multiple regression equation can be formulated according to the following formula [62-65]:

$$y_i = \beta_0 + \beta_1 x_1 + \beta_2 x_2 \quad (3)$$

Y_i refers to the response (hardness), while x_1 refers to the laser energy, and the variable x_2 refers to the number of pulses. β_0 expresses the regression constant while β_1 and β_2 express the deflection coefficient of the laser power variable and the number of pulses, respectively. The deviation equation can be found easily by using many statistical programs such as Minitab.

Using the Minitab 19 program, the hardness regression equation is calculated using the following formula [66-68]:

$$H = 76.9 - 0.0445E - 0.0895P \quad (4)$$

Through Equation 4, the regression constant appeared to be about 76.8, while the regression coefficient for the laser energy appeared to be 0.0445 and for the number of pulses to be 0.0895.

To ensure the accuracy of the prediction of the regression equation, an R-Square (R^2) was found. R^2 refers to the proportion of variance in the response that can be predicted by the variables [65, and 69-71]. The R^2 value ranges from 0 to 1. When the value of R^2 is equal to or close to 1, this indicates that the equation has high accuracy in predicting the response value. In this work, the value of R^2 of the deviation equation appeared at about 0.88, meaning that the probability of success in the prediction is relatively good since the inferred value is close to 1. The accuracy of the hardness regression equation prediction can be increased in the future by increasing the number of variables and their levels.

4. CONCLUSION

Taguchi's experimental design contributed to the optimization of the hardness of the aluminum oxide thin film prepared using the pulsed laser deposition technique. The hardness of the aluminum oxide thin film was greatly affected by the thickness of the thin film, as the increase in the thickness negatively affected the hardness of the thin film. At a thickness of 40 nm, the hardness appeared to be about 38.34 GPa, while the hardness decreased to about 14.17 GPa with the thickness increased to about 162 nm. Analysis of variance (ANOVA) aims to show the most influential variable and it turns out that the number of pulses has the most effect on the hardness of the aluminum oxide thin film by 81.254%. The regression equation plays a role in showing the relationship between the hardness and the variables represented by the number of pulses and the laser energy. It can also be used to predict new values of hardness by using new levels of the variables.

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