

Statistical Analysis of High-Power Impulse Magnetron Sputtering Parameters on the Growth and Composition of AlN Thin Films

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Abstract

This study presents an experimental investigation of Aluminium Nitride (AlN) thin film deposition using High-Power Impulse Magnetron Sputtering (HiPIMS). The main goal is to investigate the significant effect of HiPIMS sputtering parameters on the growth and chemical composition of the AlN thin film. A Design of Experiments (DoE) approach is used to conduct interaction studies among the common sputtering parameters, such as sputtering power, working pressure, and the Argon (Ar) to Nitrogen (N₂) ratio. The 2^k factorial design is specifically used in this case. The main plot effect demonstrates that the sputtering power was the most significant factor in AlN thin film growth, whereas the growth was inversely proportional to working pressure. The working pressure had a significant impact on the oxygen concentration of the AlN thin films. It was discovered that lower working pressure and higher HiPIMS power were chosen to obtain a balanced stoichiometry with less oxygen concentration. The strong preferred (002) plane orientation of AlN on Silicon, Si (111) substrate produced a with the lowest oxygen content of 5.4% by weight. The results of this work will aid in understanding HiPIMS capabilities and in streamlining the deposition procedure to produce AlN thin films of better quality for usage in piezoelectric and optoelectronic applications.

1. Introduction

Due to their broad band gaps, III-V materials have become increasingly popular for use in optical semiconductor devices [1]. Aluminium nitride (AlN), one of the III-V materials, has a high energy band gap of about 6 eV and can be employed in optical device applications that operate in the deep UV range [2]. For deep UV applications, 210 nm has been reported as the lowest wavelength using an AlN emitter [3]. In addition, AlN is a reliable buffer layer between the substrate and gallium nitride (GaN), reducing lattice mismatch and the possibility of crack, both of which diminish the lifespan of GaN-based devices [4]. AlN also has exceptional piezoelectric characteristics, as well as high heat conductivity (285 Wm⁻¹K⁻¹), and high resistivity (1x10¹⁶ Ω.cm) [5]. Metal organic chemical vapour deposition (MOCVD), molecular beam epitaxy (MBE), plasma enhanced chemical vapour deposition (PECVD), plasma enhanced atomic layer deposition (PEALD), and magnetron sputtering are popular techniques used in the creation of AlN thin films [6] – [9]. Among them, magnetron sputtering has additional benefits include the ability to fabricate thin films at lower process temperatures, ease of operation, and adaptability due to the ability to work with various power sources [10] [11].

The two conventional main power sources used in magnetron sputtering are direct current (DC) and radio frequency (RF). RF is known to provide a smoother surface, while DC is considered to have a faster rate of growth. However, DC suffers from target poisoning and arching phenomena that might harm the target and needs more power to achieve crystalline structure [12] [13]. In the manufacturing of AlN thin films, it is said that power is typically employed in the range of 1-2 kW for DC and 300–500W for RF [10]. RF sputtering, on the other hand, is recognized to have an excessively large coating area and a substantially slower development rate [12]. High-power impulse magnetron sputtering (HiPIMS), a different kind of magnetron sputtering that employs pulse power plasma discharge, was first developed in 1991 [13]. HiPIMS are known to generate a high plasma density because of the high level of ionization [13]. As results, a thick and smooth thin-film surface was created by the high degree of ionization in contrast to DC and RF reactive sputtering. Another study found that this method also improved thin film adhesion [11].

One of the most common experimental designs and a crucial component of Design of Experiment (DoE) are factorial designs. They permit the methodical analysis of how numerous factors interact and what effects they have on the response variable. In magnetron sputtering, specifically for AlN deposition, only a small number of researchers have reported on optimizing the deposition process using design experiments and statistical techniques [14], [15] [16] [17]. With the exception of one study by C.-T. Chang et al., which used the HiPIMS deposition technique utilizing the Taguchi method, the majority of research either used DC or RF magnetron sputtering. However, they concentrated on finding the ideal variable for mechanical coating application with high optical characteristics, and one important variable to be evaluated was the deposition temperature up to 200°C [14]. On the other hand, this study concentrated on important common factors including sputtering power, working pressure, and the Argon to Nitrogen ratio and how it affected the compositional and growth of AlN thin films using the HiPIMS technology at room temperature. The 2k factorial design was selected as the preferable approach because of its ease of use and capacity to reduce the number of experimental runs. In order to examine common factors and evaluate HiPIMS's capabilities without interference from outside sources, the deposition procedure did not use external heating aid as mentioned.

2. Experimental Details

2.1 Materials Preparation

HiPIMS techniques were used in this experiment to deposit AlN thin films on silicon substrates at room temperature. The silicon wafer with (111) orientation, Si (111) substrates were cleaned with isopropyl alcohol solution (IPA) to remove any contamination on the surface. The substrate then rinsed with deionized (DI) water, and then drying with nitrogen flow. Subsequently, the setup's load lock mechanism was used to load the substrates into the sputtering chamber (SNTTEK PSP 5004, Korea). An aluminium target (ITASCO, Korea) with a diameter of 3 inches and a purity of 5N was sputtered in this experiment. The target was positioned 120 mm above the substrate inside the chamber. The sputtering system is equipped with two power sources, a High-Power Impulse power source (STARFIRE, USA) and a DC power source (PSPLASMA SDC1024A, Korea), both of which can generate DC pulses with maximum 2 kHz repetition rates and pulse widths up to 400µs. Fig. 1 shows the detail schematic diagram of sputtering chamber with HiPIMS power supply and load lock system.

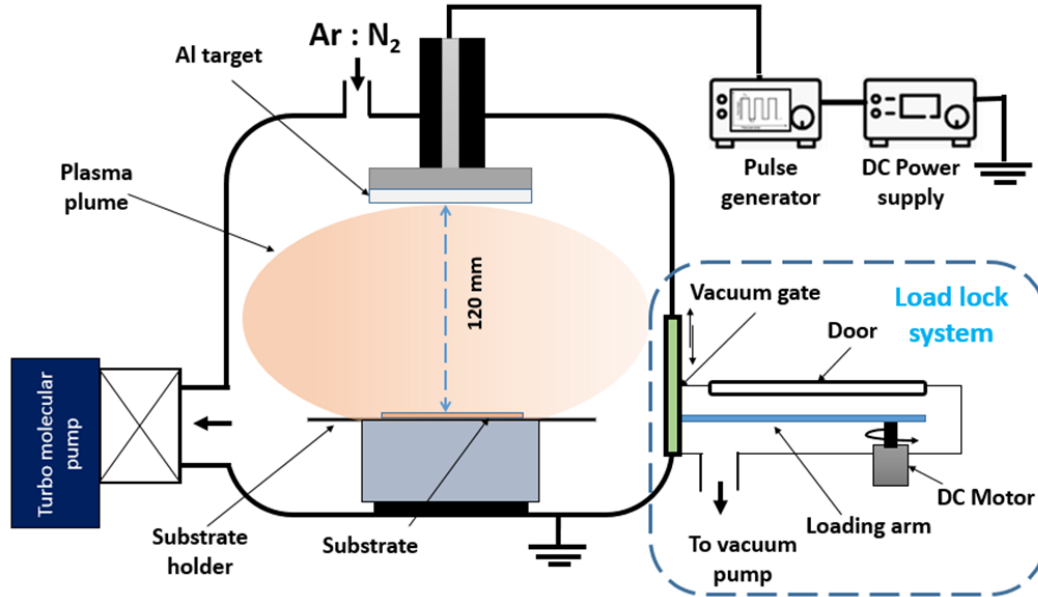


Fig. 1 Schematic diagram of HiPIMS system for AlN deposition

2.2 Sputtering Parameters Condition

The 2^k factorial design, a widely employed method at the preliminary stages of experimentation, facilitates a straightforward and simple approach. In this particular investigation, the factors under study included sputtering power, working pressure, and the Ar to N_2 flow ratio. Consequently, the factorial design comprised three factors, where each factor having two quantitative level of high and low. Table 1 shows the sputtering parameters condition as factors investigated and its respective levels. The table also showing that this design approach having k is equal to three where A, B, and C correspond to the three factors studied. The sputtering power that delivered by HiPIMS to the Al sputter target was set up at minimum of 100 W and maximum of 300 W. The capability of in-house DC supply to the HiPIMS is at 500 W maximum. Therefore, the HiPIMS power was limited to below 500 W range. The working pressure was set at minimum of 3 mTorr and maximum of 10 mTorr. Notably, the high and low level of working pressure chosen was known as the typical range in deposition of AlN thin film by magnetron sputtering. As for the flow ratio of Ar to N_2 , the minimum ratio was set to 50/50 sccm which correspond to 1 and 100/50 sccm that correspond to 2 as a result of total Ar flow divided to total N_2 flow fed to the chamber in standard cubic centimetre unit. By percentage, the N_2 content was reduced at flow ratio of 2 at 30% while at 50% for flow ratio of 1.

Table 1 Factors investigated and levels

Symbol	Sputtering Parameters (Factors)	Level - Low (-1)	Level - High (1)
A	Sputtering Power (W)	100	300
B	Working Pressure (mTorr)	3	10
C	Ar to N_2 flow ratio	1	2

From Table 1, the test matrix can be derived and summarized as shown in Table 2 where an L8 orthogonal array for three factors, each with two levels, was used. An L8 orthogonal array is a specific type of orthogonal array that consists of 8 experimental runs. It is well-suited for studying the main effects of three factors, each with two levels, as well as any two-factor interactions. In an L8 orthogonal array, each factor is assigned to two levels (usually denoted as -1 for low or minimum and 1 for high or maximum). The levels represent the different settings or conditions of the factors being studied. The array is structured in a way that ensures the levels of each factor appear an equal number of times across the eight runs, satisfying the orthogonality property. The Minitab 21 software has been used to generate statistical analysis through factorial design approach. Based on L8 orthogonal test matrix, factorial design with three number of factors and full factorial was selected with full resolution.

During the deposition process, the chamber was first evacuated to a base pressure of approximately 10^{-6} Torr to ensure a high vacuum condition. The Al sputter target's surface was then cleaned using a 15-minute pre-sputtering step under Ar ambient with an RF power of 50 W. The deposition was carried out using the HiPIMS

technique, and a total of eight runs were performed. The HiPIMS parameters included a pulse width of 30 μ s at a repetition rate of 2000 Hz. To achieve uniform thin film deposition, the substrate holder was continuously rotated at a constant speed of 10 rpm throughout the 120-minute deposition time. During the deposition, the sputtering power was controlled by adjusting the target voltage using a DC power supply, while the working pressure was regulated by adjusting the throttle valve position. Additionally, the concentrations of Ar and N₂ inside the chamber were controlled by adjusting their respective mass flow rates using flow controllers. The main purpose of this study was to investigate the influence and interaction of common sputtering parameters on the deposition of AlN thin films using HiPIMS. Therefore, no external heating was applied to the substrate. This approach was adopted to assess the fundamental capability of HiPIMS in producing crystalline AlN structures on substrates at room temperature

Table 2 *L8 orthogonal array table for AlN deposition*

No. of Runs	Sample Designation	A	B	C
1	N1	-1	-1	-1
2	N2	1	-1	-1
3	N3	-1	1	-1
4	N4	1	1	-1
5	N5	-1	-1	1
6	N6	1	-1	1
7	N7	-1	1	1
8	N8	1	1	1

2.3 Thin Films Characterization Method

The crystal structure of AlN thin films was examined using X-ray diffraction (XRD; Panalytical, UK) and a Cu-K radiation source (1.5406 nm). The diffraction pattern across the 20° to 80° range was collected using the 1/2 divergence slit and 0.5° incidence angle. An Energy Dispersive X-Ray Spectrometer, or EDS (Oxford Instruments, UK), linked to the FESEM (JOEL-JSM1763, Japan), was then used to analyse the elemental composition. The ellipsometry technique with ellipsometer (Filmetric F-20, US) was used to measure the thickness of AlN thin films.

3. Results and Discussion

3.1 Structure of AlN Thin Films

Fig. 2 shows the AlN crystal structure for all eight samples (N1 to N8) as seen by the XRD spectrum. For the hexagonal close-packed structure of AlN, the spectrum produced by High Score Plus was compared to the ICSD database using reference code 98-003-1169. According to the spectra, N6 and N2 looked to be predominate, with the AlN crystal structure having a (002) plane orientated, as the maximum intensity (002) peak occurred at the 2 θ position of 36°. However, the (103) plane also significantly observed for sample N2 at 2 θ position around 66° indicating that the influenced of higher N₂ concentration based on Ar to N₂ ratio at minimum. At the top, N8 was more likely to be a polycrystalline AlN crystal structure with nearly equal intensity counts for different peaks, while N5 has a dominating (100) plane orientation. In this study, the crystal plane orientation of AlN can be tuned by simply varies these three common factors. The preferred (002) orientation plane which also refer to c-axis AlN can be achieved as nearly single crystal plane shown by sample N6.

The c-axis orientation of AlN crystals is highly desirable for various applications, especially in piezoelectric and optical devices, due to its unique properties. However, producing c-axis-oriented AlN thin films at room temperature has been challenging [18]. The results show the effectiveness of HiPIMS in overcoming this challenge. HiPIMS is known for its unique characteristic of producing highly energetic sputter particles, which can significantly influence the growth of AlN crystals. This study demonstrates that HiPIMS enables the successful growth of c-axis-oriented AlN thin films even at room temperature with considerably longer target to substrate distance of 120 mm. The study further reveals that with average power levels below 500 W, HiPIMS is capable of producing high-quality crystalline AlN thin films on Si substrate. This finding is significant because it indicates that the HiPIMS technique can achieve the desired c-axis orientation of AlN even at relatively low power settings, making the process more energy-efficient and cost-effective.

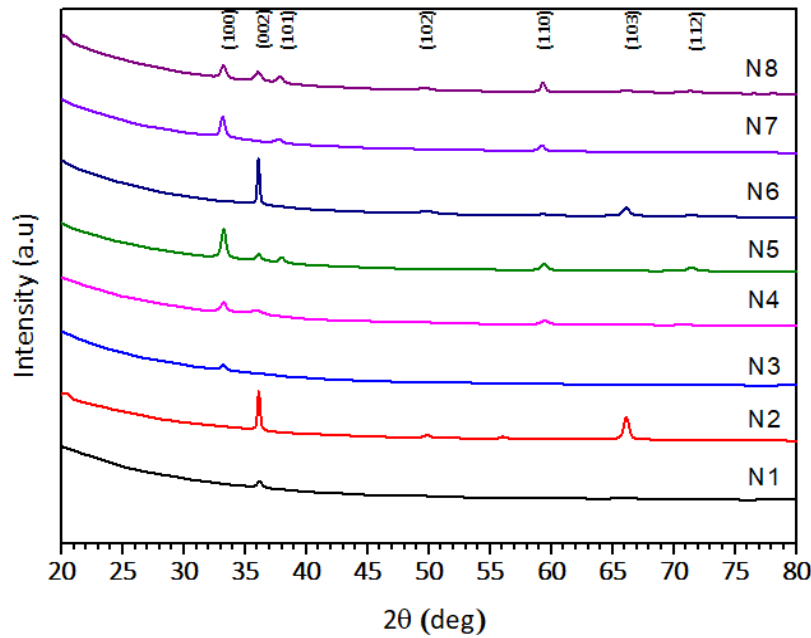


Fig. 2 XRD spectrum of all samples (N1 to N8)

3.2 The Growth of AlN Thin Films

Minitab 21 was used to generate output for investigating the interaction between sputtering parameters affecting the thin film thickness of AlN deposited through the HiPIMS technique. In Fig. 3, graphical analysis tools for the standardized effect are displayed. The normal plot in Fig. 3(a) indicates that the AlN thin film thickness is notably influenced by discharge power (A), Ar to N₂ ratio (C), and the combination of both A and C. According to the findings, the sputtering discharge power (A) significantly contributes to the growth rate of the AlN thin film, as discussed by G.F. Iriarte et al. [19]. In the normal plot, factors that are located closer to the line are generally considered to have less significance in influencing the thickness of the thin film, while factors that are far from the line are more influential. Specifically, in the context of the experiment, factors that fall far behind the line are indirectly proportional to the thickness of the AlN thin film. One such factor is B, which represents the working pressure.

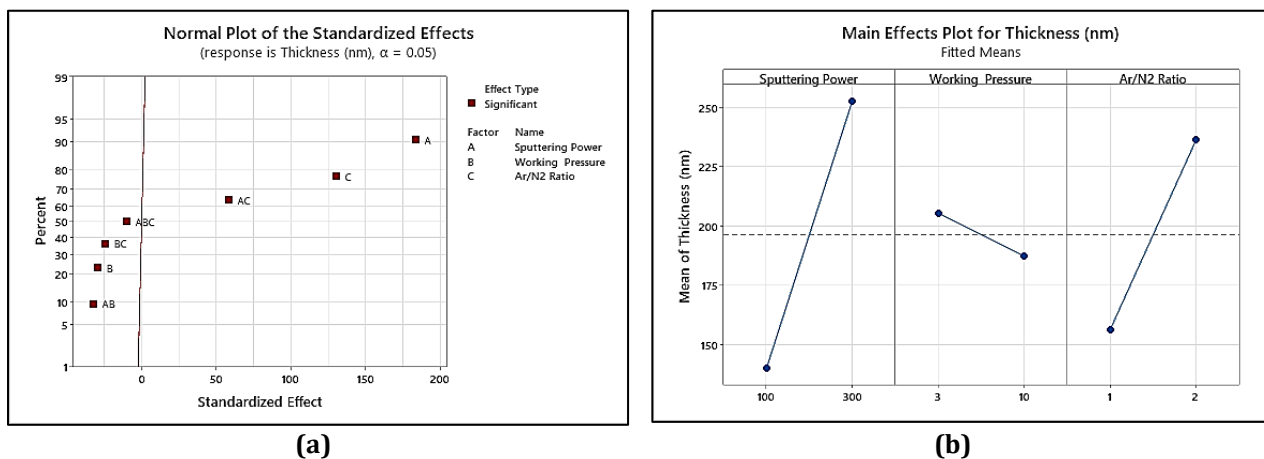


Fig. 3 Graphical analysis for AlN thin film thickness (a) normal plot of the standardized effects; (b) main effects plot

From the normal plot, it was observed that the data point for working pressure is slightly far behind the line. This suggests that the growth rates of AlN decrease as the working pressure increases. In other words, higher working pressure leads to a reduction in the thickness of the AlN thin film. This observation is further supported by the main effect plot in Fig. 3(b), where the impact of individual factors on the thin film thickness is depicted.

The main effect plot clearly illustrates that only working pressure shows an inverse relationship with the thickness measurement at higher pressure levels. This means that as the working pressure increases, the thin film's thickness decreases, which is in line with the findings from the normal plot.

The contour plot is used to understand the interaction between two independent factors and their impact on a response variable. In this specific case, the contour plot is employed to study the growth of AlN thin films, and it typically displays two factors, sputtering power, and working pressure, with each varying along different axes. Additionally, the Ar to N₂ ratio is also being studied as an independent factor. Fig. 4 in the study presents individual contour plots, each providing insights into the interaction between these factors and the resulting growth of the AlN thin film. By varying these three factors, we may estimate the thickness of the thin film under different conditions. For example, the contour plot reveals areas where the film's thickness becomes significantly thicker, which is represented by the dark green regions. To achieve this desired thickness, it is observed that lowering the working pressure while simultaneously increasing the sputtering power is the most effective approach. These findings are valuable for optimizing the thin film deposition process. By analysing the contour plot, one can identify the specific combinations of sputtering power, working pressure, and Ar to N₂ ratio that led to the desired film thickness. This allows for better control over the deposition process and enables the production of AlN thin films with the desired properties for specific applications.

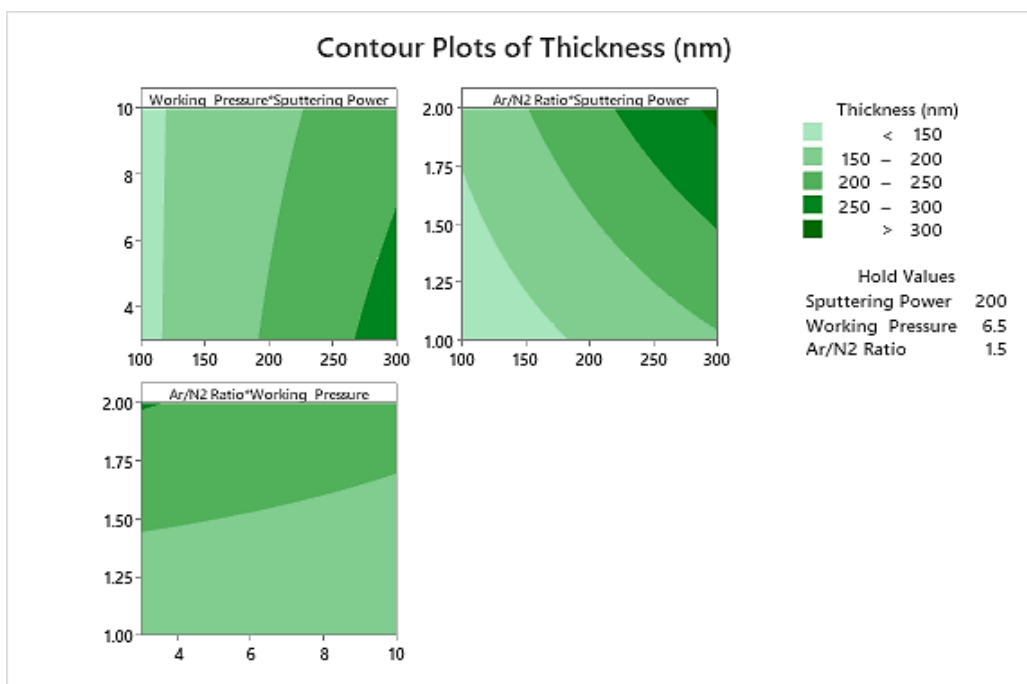


Fig. 4 Contour plot of AlN thin film thickness

3.3 Composition of AlN Thin Films

In an AlN thin film, the primary elements are Al and nitrogen N₂. The relative proportions of Al and N₂ in the film can be determined by analysing the peak intensities in the EDS spectrum. Additionally, the presence of other elements, like oxygen (O), can be detected in the EDS spectrum if they are present in the film. Fig. 5 presents the element composition in AlN thin films by EDS peaks based on intensity and weight percentage. AlN thin films with a stoichiometric composition, where the ratio of Al to N₂ is 1:1, are considered ideal and balanced. These films exhibit high thermal conductivity and high electrical resistivity, making them well-suited for applications in high-power electronic devices and high-temperature thermal management. The main effect plot in Fig. 6 provides valuable insights into the influence of different factors, on the composition of the AlN thin film. Specifically, the plot indicates that the oxygen content in the AlN film is significantly affected by the working pressure. In the context of achieving a balanced stoichiometry in the Al-N composition, it is desirable to have a lower oxygen content. The Main Effect plot shows that by adjusting the working pressure, one can effectively control the oxygen content in the AlN thin film.

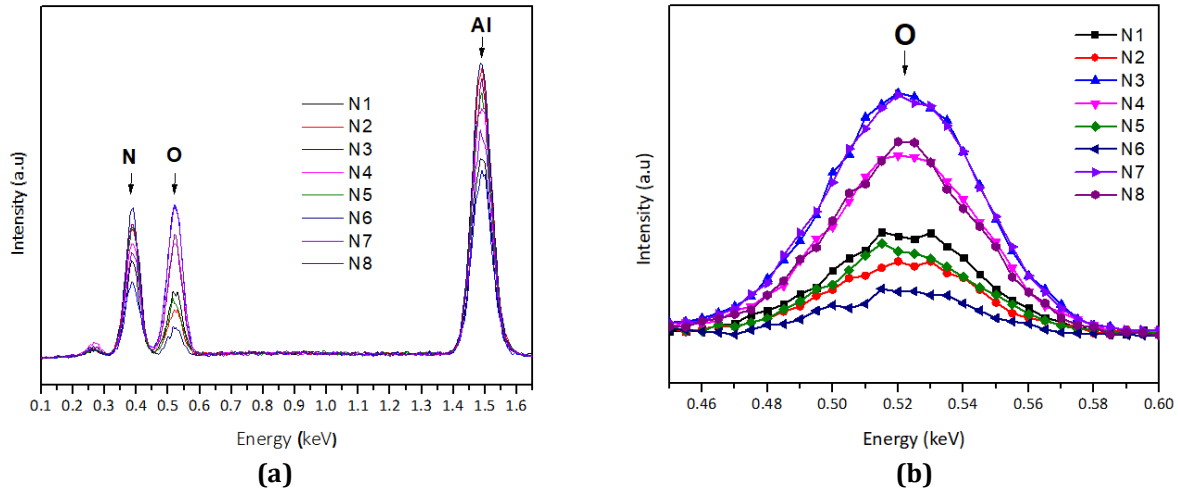


Fig. 5 Composition of AlN thin film by weight (a) EDS of all samples; (b) Oxygen content

Further analysis of all the samples reveals that a combination of higher HiPIMS power, an Ar to N₂ ratio of 2, and the lowest sputtering pressure leads to the production of AlN thin films with improved stoichiometry in terms of Al-N weight percentage. This specific combination of parameters yields Sample N6, which exhibits better stoichiometry compared to other samples. Interestingly, the sputtering parameter recipe that produces Sample N6 also leads to the lowest oxygen content, with only 5.4% oxygen found in the AlN thin film as clearly shown by EDS in Fig. 5(b). This finding reinforces the view that the working pressure plays a critical role in determining the oxygen content and achieving the desired stoichiometry in the AlN thin film.

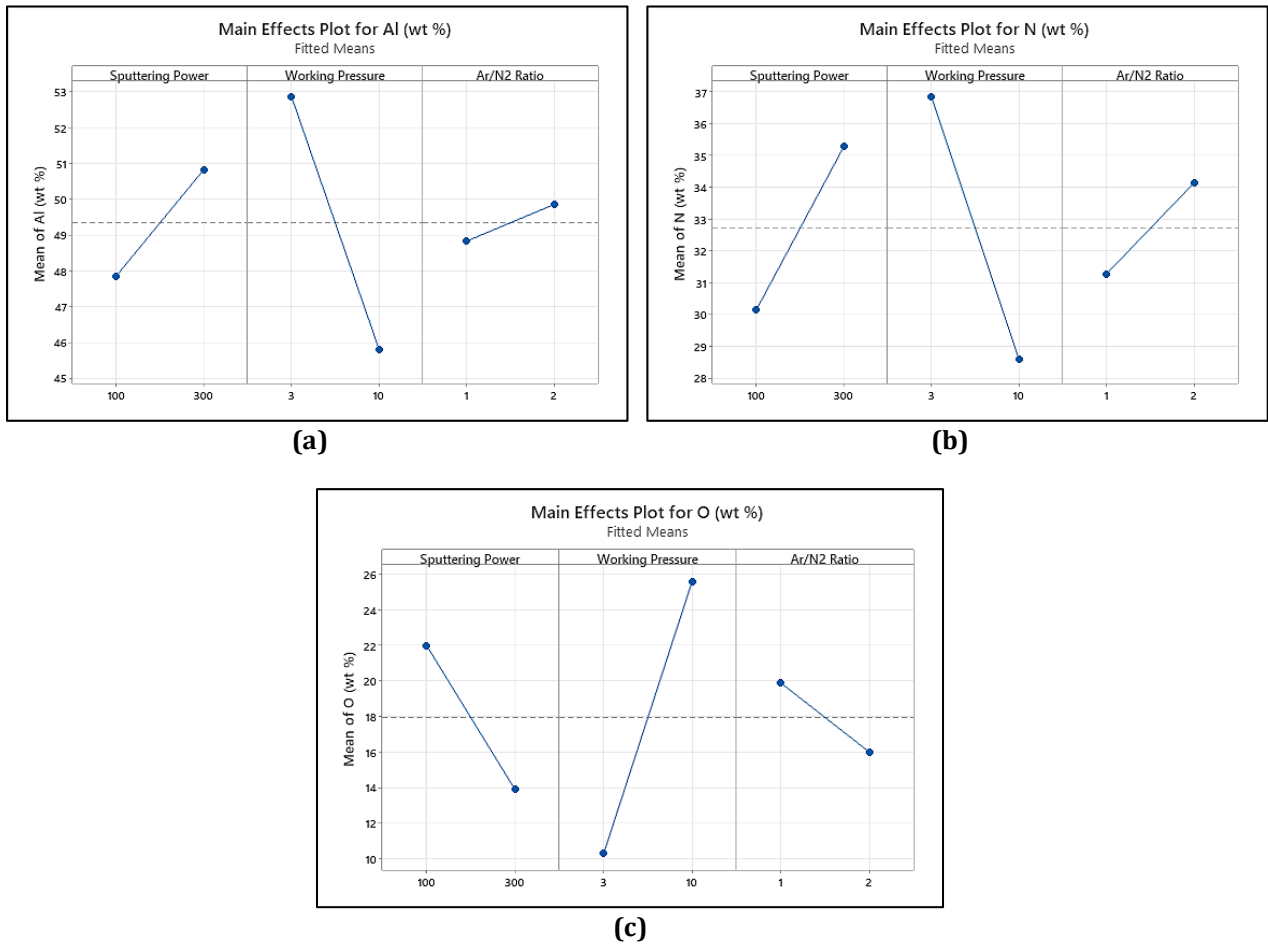


Fig. 6 Main effects plot (a) Al (wt%); (b) N (wt%); (c) O (wt%)

4. Conclusion

In conclusion, this study successfully demonstrated the deposition of AlN thin films on Si (111) substrates using the HiPIMS technique at low temperatures. Its main objective was to investigate the influence and interaction of different sputtering parameters on AlN thin film growth, aiming to achieve balanced stoichiometry at room temperature. This investigation was accomplished through a statistical approach. By carefully adjusting the common sputtering parameters, the study demonstrated the feasibility of depositing crystalline c-axis-oriented AlN thin films at low power levels, making the process energy-efficient and cost-effective and making it an attractive option for industrial applications. The implementation of a 2^k factorial design efficiently minimized the number of experimental runs while providing valuable insights into optimizing the deposition process of AlN thin films through HiPIMS. This approach enhances the understanding of the factors influencing film growth and covers the way for further advancements in thin film technology. The results of this research have significant implications, particularly in the fields of piezoelectric and optical devices, where high-quality AlN films are in demand. By offering new possibilities for producing advanced thin films with enhanced properties, this study contributes to the ongoing progress in thin film technology.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: **study conception and design:** Zulkifli Azman, Nafarizal Nayan, Mohd Rofei Mat Hussin, Mohd Yazid Ahmad; **data collection:** Zulkifli Azman; **analysis and interpretation of results:** Zulkifli Azman, Ahmad Shuhaimi Abu Bakar, Norain Sahari, Anis Suhaili Bakri; **draft manuscript preparation:** Zulkifli Azman, Nafarizal Nayan. All authors reviewed the results and approved the final version of the manuscript.

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