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Optimized deposition parameters for titanium nitride coatings: Enhancing mechanical properties of Al 6011 substrates via DC sputtering

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9 INDUSTRY, INNOVATION AND INFRASTRUCTURE
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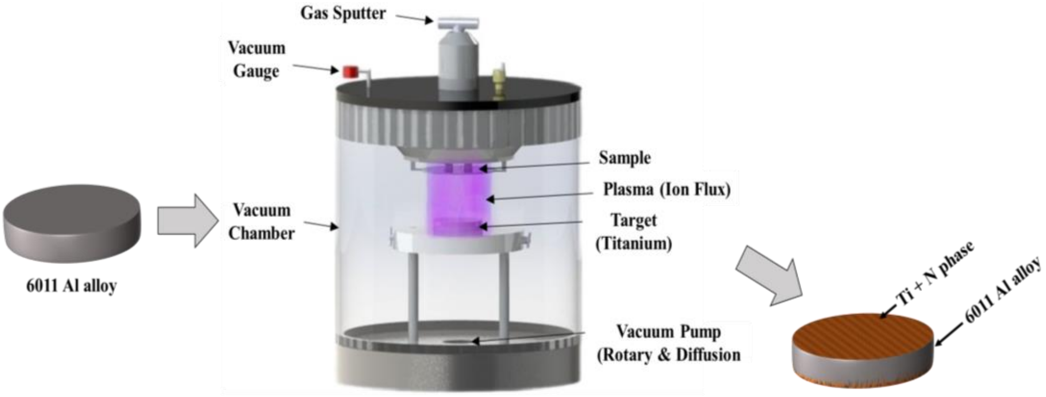

Highlights:

- The study identifies optimal sputtering parameters (70Ar:30N_2 gas ratio and 60-minute deposition) for TiN coatings on Al 6011 substrates, resulting in significant improvements.
- The optimized coatings achieve a 165% increase in surface hardness (88.92 HV) and a 54% reduction in wear rate compared to untreated samples.
- XRD and SEM analyses confirm the effectiveness of the enhancements, offering a cost-effective and scalable solution to enhance aluminum alloy performance for industrial use.

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Abstract

The growing demand for advanced coatings in industries such as aerospace and automotive necessitates materials with superior hardness, wear resistance, and thermal stability. Despite advancements in ternary coatings, research on binary Titanium Nitride (TiN) coatings remains limited, particularly in optimizing deposition parameters for lightweight aluminum substrates. This study aims to investigate the effects of sputtering parameters, specifically Ar:N_2 gas ratios and deposition durations, on the mechanical properties of TiN coatings on Al 6011 substrates. The optimized conditions (70Ar:30N_2 gas ratio and 60-minute deposition) yielded a 165% increase in surface hardness (88.92 HV) and a 54% reduction in wear rate compared to untreated samples. XRD and SEM analyses confirmed the dense microstructure and strong (200) phase orientation contributing to these enhancements. This research highlights a cost-effective and scalable approach to improving the performance of aluminum alloys, bridging the gap between fundamental studies and industrial applications.

Keywords: 6011 Al alloy; DC sputtering; TiN; Mechanical properties; Microstructure

1. Introduction

The demand for advanced surface coatings in modern industries such as aerospace, automotive, and machining has driven significant research into materials with high hardness, wear resistance, and thermal stability [1]–[4]. Titanium Nitride (TiN) coatings are widely recognized for their excellent mechanical and tribological properties, making them a preferred choice for enhancing the performance and lifespan of lightweight materials like aluminum alloys [5]–[8]. TiN coatings, deposited via methods such as DC sputtering, not only improve surface hardness but also reduce [9]wear, making them indispensable for applications requiring durability under extreme conditions [10]–[13].

Despite significant advancements in surface coating technologies, including the development of ternary and quaternary coatings like TiCN [14], [15], TiSiN [16], [17], and TiAlN [14], [18], which exhibit enhanced hardness, wear resistance, and thermal stability [19], [20], TiN remains a crucial area of investigation due to its industrial relevance and cost-efficiency [21]. Existing studies extensively explore the effects of deposition parameters, such as Ar:N_2 gas ratios and sputtering times, on TiN coatings [22]–[27]. However, many of these investigations focus on substrates with minimal relevance to lightweight applications, or on general parameter trends without offering practical optimization for specific materials like Al 6011. This aluminum alloy is critical in industries such as aerospace and automotive [28]–[30], yet it is inherently prone to wear and requires targeted surface treatments to enhance its durability [31]–[36]. Furthermore, most research highlights the superior performance of ternary coatings but often overlooks the practicality of binary coatings like TiN [37], [38], which provide a balanced solution for applications that do not demand extreme thermal or corrosion resistance [39]–[41].

To address these gaps, this study introduces a systematic exploration of TiN coatings specifically tailored for Al 6011 substrates, focusing on optimizing sputtering parameters to maximize performance. The proposed approach emphasizes a process-property relationship analysis, systematically varying Ar:N_2 gas ratios and deposition durations to study their effects on the microstructure, hardness, and wear resistance of TiN films. Unlike previous studies, this research integrates advanced characterization techniques such as XRD, SEM, and EDS to establish a comprehensive understanding of how sputtering parameters influence mechanical properties. This targeted optimization not only fills the gap in practical applications of TiN coatings for lightweight alloys but also offers a scalable, cost-effective solution that bridges fundamental research with industrial needs. By focusing on Al 6011, the study directly addresses the material's performance challenges, providing actionable insights for industries seeking to enhance wear resistance while maintaining cost-effectiveness.

This study is to investigate the influence of sputtering parameters, specifically the Ar:N_2 gas ratio and deposition duration, on the mechanical and microstructural properties of TiN coatings on Al 6011 substrates. Through systematic testing of hardness, wear resistance, and microstructural changes, this research seeks to optimize sputtering conditions for achieving superior surface performance. The findings will address current gaps in the literature while reinforcing the practicality and industrial relevance of TiN coatings as a cost-effective surface treatment solution.

2. Material and Experimental Work

2.1. Materials and Coating process

This study investigated the deposition and properties of TiN thin films on aluminum alloy 6011 substrates. The aluminum alloy samples (cylindrical shaped with a diameter of 10 mm and a thickness of 3 mm) were prepared by sanding with silicon carbide paper ranging from 200 to 3000 grits, followed by polishing with Autosol and ultrasonic cleaning in alcohol for 15 minutes. **Table 1** summarizes the chemical composition of the 6011 Al alloy used in this study.

Table 1. Chemical composition of 6011 Al alloy (wt.%)

Fe	Si	Zn	Mg	Ni	Zr	Mn	Cu	Al
0.93	0.86	0.58	0.36	0.32	0.20	0.16	0.15	96.06

TiN thin films were deposited on the prepared substrates using DC sputtering. **Figure 1** illustrates the experimental setup for the deposition process. The Ti target used had a purity of 99.99%, a diameter of 50 mm, and a thickness of 3 mm. The deposition was carried out in a vacuum chamber with a base pressure of 2.5×10^{-4} mbar, maintaining a constant sputtering working pressure of 2 × 10^{-2} mbar. The source voltage and current were set at 3 kV and 10 mA, respectively, with a target-to-substrate distance of 2.5 cm.

The deposition process consisted of two stages:

Gas Ratio Variation: TiN thin films were deposited under varying argon (Ar) and nitrogen (N_2) gas ratios of 90:10, 80:20, 70:30, and 60:40%, coded as R9, R8, R7, and R6, respectively. Each deposition lasted for 60 minutes. The Vickers hardness of the samples was measured to identify the gas ratio yielding the highest hardness value, indicating the optimal Ar:N_2.

Deposition Time Variation: Using the optimal Ar:N_2 gas ratio determined in Stage 1, TiN films were deposited with sputtering times of 30, 60, 90, and 120 min corresponding to samples coded T3, T6, T9, and T12, respectively.

The results indicated that the gas ratio and deposition time significantly influenced the properties of the TiN films. The optimal Ar:N_2 gas ratio resulted in films with the highest hardness, which can be attributed to the densification of the TiN structure and improved adhesion to the substrate. Increasing the sputtering time further enhanced the film thickness and mechanical properties, with a clear trend observed in the hardness measurements. These findings highlight the critical role of process parameters in tailoring the characteristics of TiN thin films.

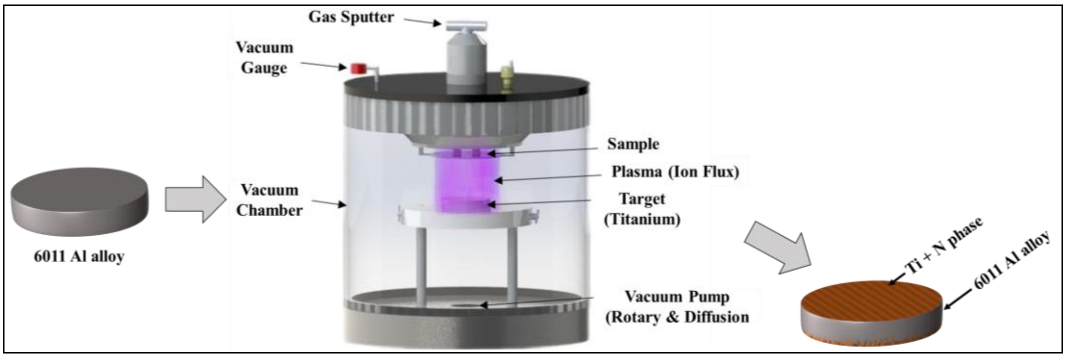

Figure 1. Schematic of the TiN thin film deposition process

2.2. Structure Characterization

The X-ray diffraction technique (XRD, Rigaku Miniflex 600) using Cu-Kα radiation (λ=0.15406 nm) was used to identify the phases in the obtained surface layers. The working voltage and current are 40 kV and 15 mA, respectively.

2.3. SEM Analisys

Scanning electron microscope (SEM, Brand Zeiss type Evo 10) was used to observe coating surface and morphology. With energy dispersive X-ray spectrometer (EDS) was also utilized for chemical composition analysis.

2.4. Mechanical Characterization

The coated samples' surface hardness was tested under 10 gf for 10 s using a Vickers microhardness tester (Matsuzawa MMT-X7). Dry sliding wear tests on cylindrical 6011 Al alloy plates were performed using an Ogoshi High Speed Universal Wear Testing Machine (Type OAT-U) at 1430 rpm. The usual contact load was 2.12 kg and 60 s. Furthermore, the wear rate (W_r) was obtained from the wear volume V, the test load W, and the sliding distance L according to the following, Eq. (1) [42], [43].

Additionally, after the wear test was completed, the friction surface was observed using a scanning electron microscope. This was done to elucidate the microstructural alterations resulting from the wear test.

$$W_r = \frac{V}{WL}\ [mm^2/kg] \tag{1}$$

3. Results

3.1. Structural Analysis of TiN Coating using XRD

Figure 2 shows the XRD pattern of the sample coated with TiN through the use of DC sputtering. Peaks located at angles 42.26° with planes (200) were identified to form the TiN phase.

In addition, growth peaks of titanium aluminide phase (TiAl) at angles 74.51° and 78.34° with planes (201) and (211), and AlN at angles 39.34° and 46.55° with planes (111, 200) were identified. This indicates that the intensive Ti-N reaction can cause diffusion to form Ti-Al and AlN. There is also a TiO_2 phase growth peak at an angle of 44.68° with plane (120).

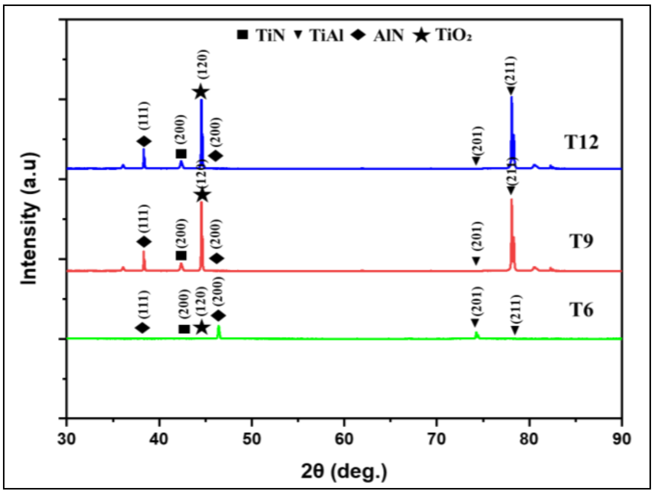

Figure 2. XRD pattern of TiN thin film of samples T6, T9 and T12

3.2. Microstructure of TiN-coated

Grain growth occurs in localized areas (**Figure 3**), forming white islands that grow into a continuous layer. The T6 layer showed dense morphology on the surface (**Figure 3b**). With the increase in deposition time, the surface of the T9 and T12 layers became denser and smoother (**Figure 3c** to **Figure 3d**).

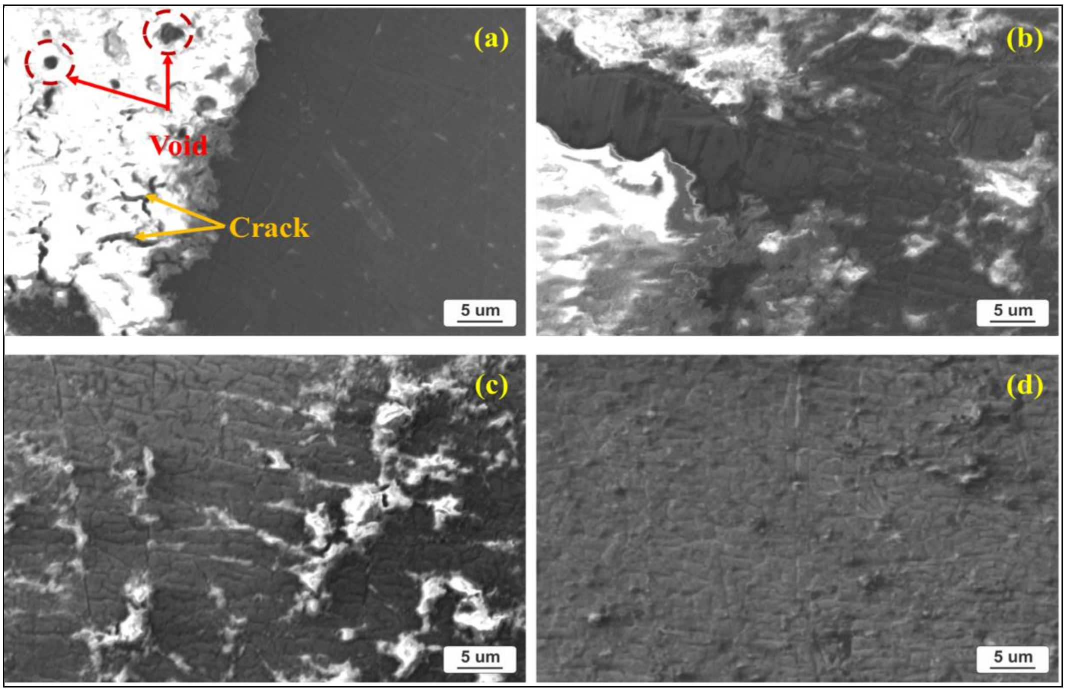

Figure 3. Surface morphology of TiN-coated at different times with Ar:N_2 gas ratio:
a) T3;
b) T6;
c) T9;
d) T12

Figure 3 shown that deposited TiN thin films grow through islands using the Volmer-Weber growth mechanism, forming TiN via in-situ chemical reactions between Ti adatoms and N_2 ions chemically adsorbed on the substrate surface [44].

Figure 4 displays the EDS results on the surface of the TiN layer. The nitrogen content on the surface of the T3 layer is about 0.03%, then increases to 0.96% for the surface of the T6 layer. However, in the T9 and T12 layers, the nitrogen content decreased, 0.6% and 0.06%, respectively. In addition, on various coating surfaces, there are Oxide (O) contents of 44.34%, 48.99%, 56.76%, and 55.17%, as shown in **Table 2**.

3.3. Mechanical Properties

3.3.1. Hardness

Figure 5a shows the microhardness profile on the effect of gas ratio Ar: N_2 gas ratio and sputtering time. The TiN coated samples have higher hardness than the untreated samples. The hardness value of the untreated sample is 33.58 HV. The hardness of the R9-R7 coating increases to, 49.06, 50.58, and 88.92 HV. Meanwhile, the R6 layer experienced a decrease in hardness of 40.56 HV. Thus, the highest hardness is obtained in the R7 layer about 165% compared to the untreated sample.

Figure 5b illustrates the hardness measurement of TiN thin films on substrates for various deposition times at optimum Ar:N_2 sputter gas flow conditions. It was found that as the deposition time increased, sample T6 had the highest hardness of 88.92 HV. Furthermore, samples T9 and T12 experienced a decrease in hardness of 42.84 and 42.00 HV, respectively.

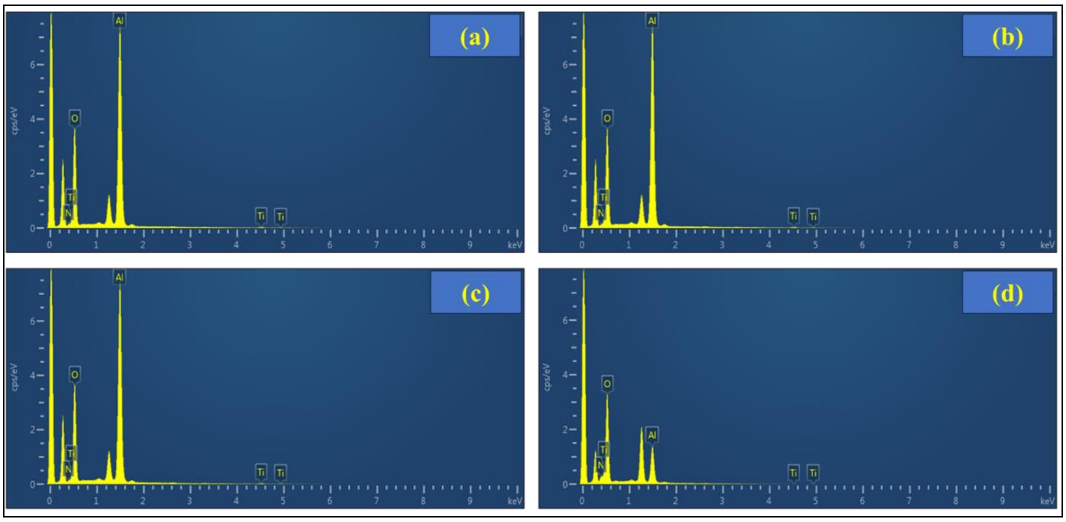

Figure 4. EDS analysis of the surface of TiN-coated at different times with Ar:N_2 gas ratio: a) T3; b) T6; c) T9; d) T12

Table 2. EDS analysis of the surface of TiN-coated at different times with Ar:N_2 gas ratio

Sample	Atomic (%)			
	Al	Ti	N	O
T3	26.70	28.93	0.03	44.34
T6	16.09	33.96	0.96	48.99
T9	5.84	36.80	0.6	56.76
T12	6.89	37.88	0.06	55.17

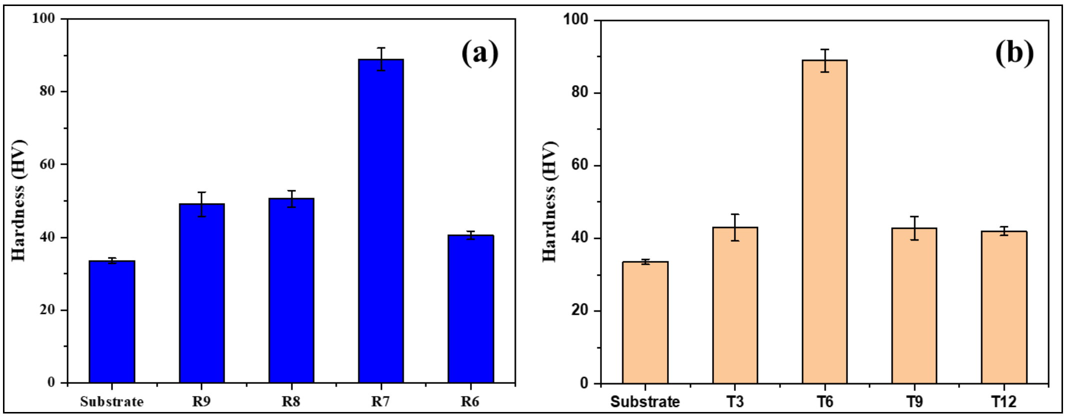

Figure 5. Surface hardness of substrate and TiN-coated: (a) stage 1; (b) stage 2 process deposition

3.3.2. Wear Behavior

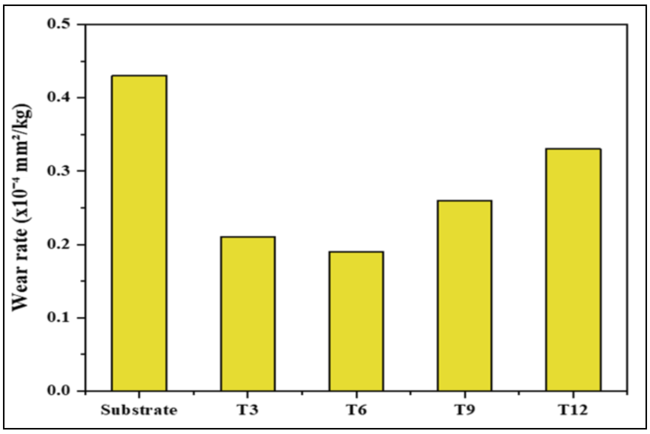

Figure 6. Wear rate of substrate and TiN-coated

Figure 6 shows the wear rate of untreated and TiN-coated samples. The wear rate of TiN-coated samples decreased drastically with increasing deposition time, especially for sample T6, with a wear rate of 0.19 x 10^{-4} mm^2/kg, compared to the untreated substrate of 0.43 x 10^{-4} mm^2/kg. Furthermore, samples T9 and T12 showed a decrease in wear resistance of 0.26 x 10^{-4} mm^2/kg and 0.33 x 10^{-4} mm^2/kg, respectively.

4. Discussion

This study demonstrates that the Ar:N_2 gas ratio and deposition duration significantly influence the microstructural and mechanical properties of TiN coatings deposited on Al 6011 substrates. XRD analysis revealed that the increase in hardness is primarily due to the strong (200) preferred orientation of the TiN phase, which enhances the coating's structural integrity (**Figure 2**). Nitride phase formation occurs when nitrogen atoms react with titanium atoms in the Ar:N_2 mixture, and the optimized gas ratio of 70Ar:30N_2 facilitated the efficient formation of TiN phases with reduced defects, achieving the highest hardness of 88.92 HV. In contrast, higher nitrogen flow ratios (e.g., 60:40) caused over-saturation, leading to voids and weaker grain boundaries, while lower ratios (e.g., 90:10 and 80:20) limited the formation of the nitride phase, reducing hardness. These findings align with prior studies emphasizing the need for precise nitrogen flow to achieve optimal stoichiometry and mechanical properties.

SEM and EDS analyses further revealed the role of deposition time in influencing the coatings' performance. Shorter deposition times (30 min) resulted in incomplete layers with voids, while extended times (90 and 120 min) led to grain coarsening and saturation, which adversely affected hardness (**Figure 3** and **Figure 4**). The optimal deposition time of 60 min yielded dense, uniform coatings with superior wear resistance [45], [46]. The observed reduction in wear rate by 54% compared to untreated samples underscores the durability of TiN coatings under optimized conditions. The abrasive wear mechanism (**Figure 7**), characterized by grooves and cracks on the worn surface, was indicative of the coatings' high hardness, which minimizes adhesive wear and enhances tribological performance [47], [48].

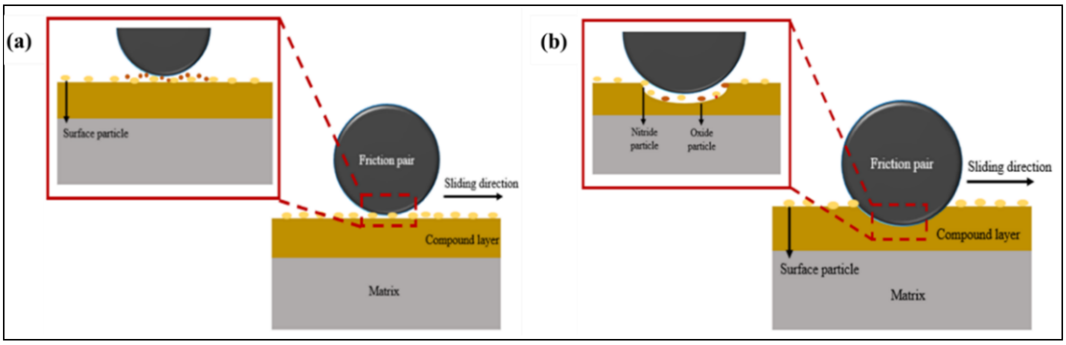

Figure 7. Ti–N coating sample wear mechanism schematic: (a) abrasive wear; (b) adhesive wear

The formation of microstructural features such as granules and the chemical interaction between titanium and nitrogen were evident in SEM images (**Figure 8**). The nitrogen diffusion into the substrate matrix, facilitated by the optimized gas ratio and deposition time, contributed to the coating's integrity. However, elemental analysis indicated a reduction in nitrogen content in coatings with extended deposition times, likely due to desorption or reduced nitrogen adsorption efficiency. Additionally, residual oxygen in the chamber resulted in TiO_2 formation, affecting the coating's morphology and hardness (**Figure 8**).

Compared to existing research on ternary coatings like TiCN [15], [49], [50] and TiSiN [51]–[53], this study highlights the practical advantages of binary TiN coatings for lightweight aluminum alloys. The cost-effective, scalable sputtering process used here bridges the gap between theoretical optimization and industrial application. By leveraging advanced characterization techniques, this study provides novel insights into optimizing TiN coatings, addressing gaps in understanding the process-property relationship for enhancing the wear resistance and hardness of Al 6011 substrates.

The microstructural and mechanical properties of TiN coatings on Al 6011 substrates are significantly affected by the ratio of Ar_2 gas and the duration of deposition. Studies have shown that both these factors play a crucial role in determining the hardness, wear resistance, and overall quality of the coating, all of which are vital for enhancing the performance of aluminum components.

The influence of the Ar:N_2 gas ratio on the microstructure of TiN coatings is notable. Research indicates that a gas ratio of 70:30 optimizes hardness and wear resistance for aluminum substrates [54]. Higher ratios of Ar:N_2 have been associated with improved mechanical properties, resulting in coatings that exhibit greater hardness and better elastic recovery when subjected to load [55].

Deposition duration is also a critical factor; optimal hardness is typically achieved within a time frame of 60 to 120 min, depending on the specific gas ratio used [54]. Lengthening the deposition time can lead to increased coating thickness and enhanced wear resistance, often reflected in substantial improvements in hardness compared to initial values [54].

These findings reaffirm the importance of carefully controlled sputtering parameters and position binary TiN coatings as a practical solution for applications requiring improved surface performance without the complexity and cost of ternary systems.

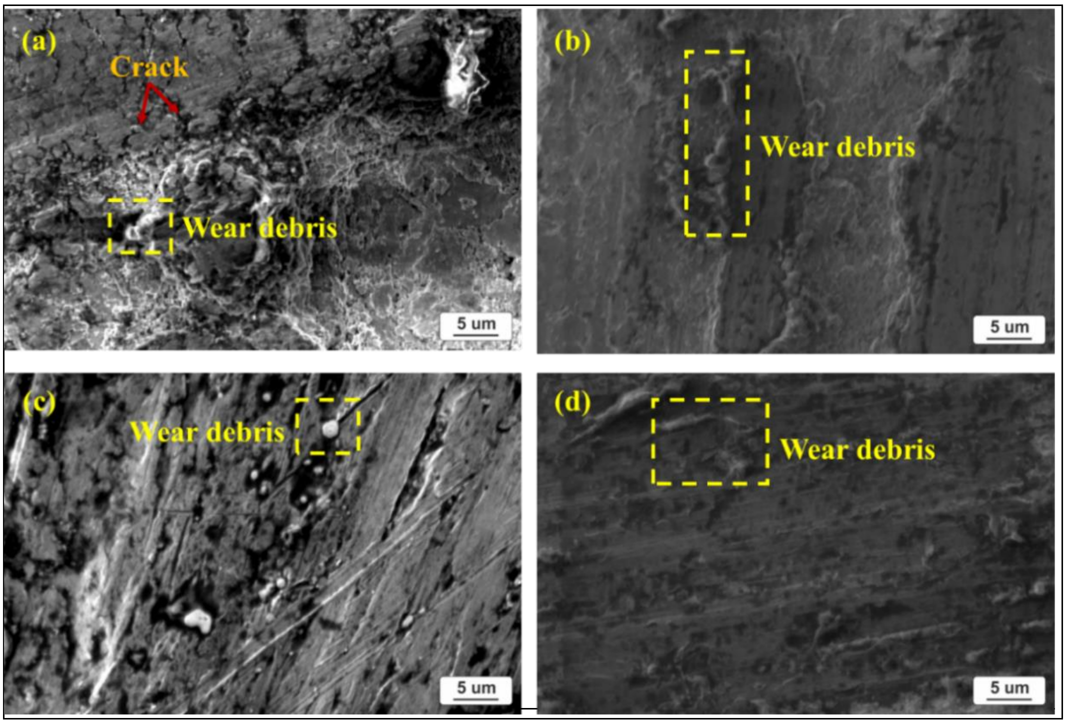

Figure 8. Ti–N coating sample wear mechanism schematic: (a) abrasive wear; (b) adhesive wear

5. Conclusion

This study evaluated the mechanical properties of TiN films deposited on Alloy 6011 aluminum substrates using DC sputtering, focusing on the effects of deposition parameters. The optimal conditions were identified as a gas ratio of 70Ar:30N_2 and a deposition duration of 60 minutes. XRD analysis revealed that the TiN film exhibited a strong texture in the (200) and (313) planes. SEM analysis showed the formation of a non-uniform but atomically dense TiN film. The TiN coating significantly increased the surface hardness of Alloy 6011 Al reaching a maximum of 88.92 HV. The wear rate of Alloy 6011 Al decreased with increasing deposition duration, with minimum values of 0.43 x 10^{-4} mm^2/kg and 0.19 x 10^{-4} mm^2/kg, respectively. Abrasive wear was identified as the predominant wear mechanism. These findings demonstrate the effectiveness of DC sputtering in improving the surface properties of aluminum alloys, highlighting the critical role of optimizing gas mixture ratios and deposition durations to achieve superior hardness and wear resistance in TiN coatings.

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Authors' Declaration

Authors' contributions and responsibilities - The authors made substantial contributions to the conception and design of the study. The authors took responsibility for data analysis, interpretation, and discussion of results. The authors read and approved the final manuscript.

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Availability of data and materials - All data is available from the authors.

Competing interests - The authors declare no competing interest.

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