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Effects of Target-Substrate Distance on Growth and Mechanical Characteristics of Nanodiamond Composite Coatings Fabricated by Coaxial Arc Plasma Deposition on WC-Co Substrate

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Abstract: *Nanodiamond composite (NDC) coatings fabricated by coaxial arc plasma deposition (CAPD) exhibit exceptional structural and mechanical properties, making them promising candidates for advanced cutting tool applications. However, the influence of certain key factors, such as the target-substrate distance (TSD), on the growth and characteristics of NDC films remains unclear. TSD affects the substrate ion current and ion bombardment energy, intimately linked to the structure and tribo-mechanical properties of the protective coatings. Therefore, NDC films were fabricated at diverse TSD values ranging from 5 to 50 mm, and their adhesion, hardness, and modulus of elasticity were investigated. The results revealed that growth and adhesion were significantly affected by TSD, whereas hardness and elastic modulus remained constant (from 10 to 50 mm TSD). The film's thickness declined from 21.88 to 1.8 µm with increasing TSD, and the film quality improved due to macroparticle elimination. Notably, the sample prepared at a distance of 10 mm exhibited the best adhesion resistance, together with a high thickness of 11.45 µm with a hardness of around 50 GPa. The target-substrate distance was revealed to have a crucial potential for determining the structural and mechanical characteristics of NDC films deposited by CAPD.*

Keywords: Hard coatings; Nanodiamond; Cutting tools; CAPD; Deposition parameters

1. INTRODUCTION

Cemented carbide, commonly known as WC-Co, is a sintered composite material consisting of tungsten carbide particles embedded in a cobalt metal matrix [1, 2]. Due to its high fracture toughness, WC-Co has been widely used in metal cutting applications include inserts, drills, and end mills [3-5]. Towards enhancing the performance and service life of cemented carbide tools, they are often coated with protective hard coatings like polycrystalline diamond [6], amorphous carbon [7], TiN [8], TiC, TiCN [9], and TiAlN [10]. Among these coatings, diamond stands out as the most promising material due to its extreme hardness in its natural state.

Diamond coatings have been deposited via chemical vapor deposition (CVD), such as hot filament CVD [11, 12] and microwave plasma-assisted CVD [13, 14]. However, the CVD process faces challenges, including the need for heat-resistant cutting tool materials because of the high substrate temperatures (800 °C to 1000 °C) during diamond deposition, the requirement for seeding diamond powder on the substrate surface in order to facilitate diamond growth, and the time-consuming deposition process exceeds 10 hours owing to low deposition rates of diamond films. Moreover, the catalytic influences of Co on the cemented carbide substrate can lead to poor adhesion in the interface between diamond films and the cemented substrate, resulting in graphitization at the interface and poor adhesion [15-19].

Hard amorphous carbon [20-24], known as diamond-like carbon (DLC), has gained popularity as an alternative to diamond coatings. DLC is cost effective and exhibits lower coefficient of friction compared with diamond coatings. Filtered cathodic vacuum arc technique (FCVA) [25] have been employed to fabricate hard DLC known as tetrahedral amorphous carbon (ta-C) at lower substrate temperature with comparable hardness to diamond films. However, the cobalt catalytic effects on WC-Co can still degrade the hardness and adhesion of a-C coatings, especially at elevated substrate temperatures. Moreover, the film thickness is limited to a few nanometers due to the high internal stress within the film. DLC films formed using CVD or sputtering show degraded hardness compared to ta-C films fabricated by FCVA method.

The demand for a new alternative hard carbon coating that combines the high hardness and thickness of diamond films with the low surface roughness and cost-effectiveness of hard DLC is crucial to enhance the performance of cutting tools while remaining eco-friendly [25, 26]. Nanostructured NDC films, which consist of countless diamond crystals with diameters below 10 nm encapsulated in a hard a-C matrix, present a promising solution [27-37]. These NDC films can be deposited using PVD coaxial arc plasma deposition (CAPD), resulting in films with high hardness (≥ 50 GPa), significant thickness (≥ 7 µm), and low internal stress (≤4.5 GPa) with significant deposition rate (0.65 nm/s) and sp^3 fraction (70 %) [38-41]. It's important to note that the hardness of carbon materials is influenced by the sp^3 content in the films, originating from either diamond or hard DLC unique features. The diamond growth mechanism in CAPD differs significantly from that of

CVD, and the features of the NDC coatings are highly depending on the preparation conditions of CAPD. Studies using optical emission spectroscopy have revealed that the dense arrival of high-energetic carbon species, notably C^+ ions, at the substrate is essential for the deposition of NDC coatings, possibly creating a supersaturated state for diamond growth and rich sp^3 amorphous carbon matrix [42, 43].

CAPD stands out among physical vapor deposition techniques for producing thick protective coatings with high quality and extreme properties, as depicted in Figure 1. Various deposition parameters, including arc discharge repetition rate, base pressure, substrate temperature, deposition power, target types, gas types, and substrate type, affect the structural, mechanical, optical, and tribological characteristics of NDC coatings. Despite extensive research on NDC films, there remains a lack of comprehensive and fundamental investigations into the interaction between deposition key factors and film properties. The rational design of novel advanced NDC films for practical applications can be achieved by understanding the impacts of all deposition parameters in CAPD.

Several researchers have explored the effects of key CAPD parameters on NDC properties. Naragino et al. [34, 35] investigated the impact of arc discharge repetition rate and substrate temperature on the mechanical characteristics of NDC coatings fabricated on cemented carbide substrates. Low repetition rates (1 Hz) and low substrate temperatures (room temperature) yielded NDC films with hardness of 51 GPa, Modulus of 520 GPa, and a film thickness of approximately 3 µm. Furthermore, Yoshida et al. [44] investigated the impact of arc discharge repetition rate (R.R) in the hydrogen presence on the mechanical properties of hydrogenated NDC films. The increase from 5 to 20 Hz in the repetition rate decreased the nanodiamond grain size from 2.6 to 1.9 nm and the hardness from 23 GPa to 11 GPa, due to suppressed successive growth after nucleation caused by short time intervals and large effective deposition rates. Ali et al. [33] examined the impacts of applying negative bias on the mechanical behaviors of NDC films. Applying negative bias to WC−Co substrates significantly improved the deposition rate of the films, releasing internal stresses and enabling thicker film deposition. On the other hand, negative bias at various frequencies in the range of 40-80 kHz degraded hardness due to increased sp^2 bonds within the films.

Researchers have also investigated the impact of different dopants in the carbon target. For instance, Egiza et al. [37] examined the doping effect of Silicon and Chromium blended graphite targets on the mechanical properties of doped films at numerous dopant concentrations. The doping with Cr and Si resulted in a reduction of film hardness. However, in a separate study, Egiza et al. [32] specifically focused on 1 at.% Si-doped targets to suppress Co diffusion and enhance the sp^3 bonds formation (C−C), leading to an increase in hardness. Furthermore, the implications of Boron dopant on the structural and tribo-mechanical characteristics of NDC fabricated on WC−Co substrates by CAPD were

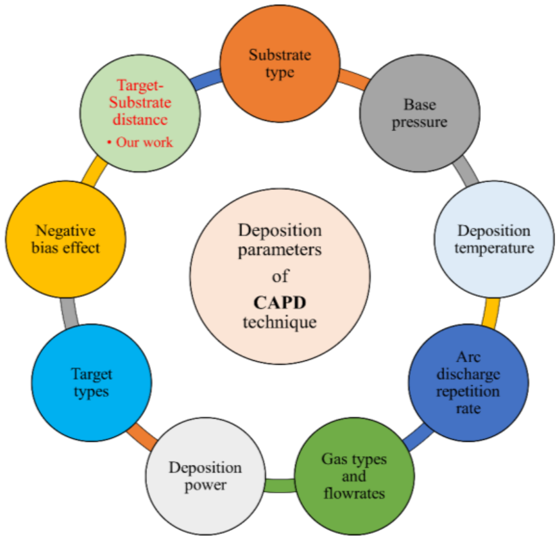

Figure 1. Schematic illustration for key parameters of preparation device (CAPD).

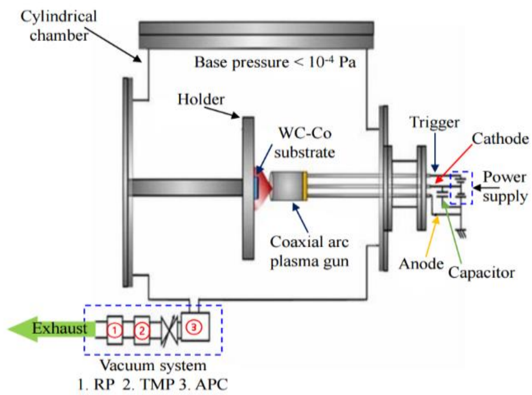

Figure 2. Schematic diagram of preparation apparatus (CAPD).

investigated by Egiza et al. [45]. They found that 1 at. % B doping significantly improved the film's hardness to 60 GPa, particularly when a buffer layer was used to mitigate the Co catalytic effects at the substrate-film interface.

Moreover, the choice of substrate type significantly influences the application of NDC films and the deposition process. Researchers have successfully deposited NDC films on various substrates, including WC−Co, Si, and Ti, using CAPD methods. For instance, Hiroshi et al. [35, 36] achieved the fabrication of NDC coatings on cemented carbide substrates for cutting tool applications, while Ali et al. [29, 30] investigated the capability of the CAPD apparatus to fabricate adherent NDC films on unheated silicon substrate. Furthermore, Osman et al. [28, 46] demonstrated the successful room temperature deposition of adherent NDC coatings on titanium substrates by employing an ion etching gun to remove the oxide layer at the interface before deposition.

Despite extensive research on the preparation and tribo-mechanical features of nanodiamond composite films, the influence of key factors remains incompletely understood. One such critical factor is the target-substrate distance (TSD), which can lead to a more homogenous distribution of ejected atoms but with diminished kinetic energy as they reach the substrate. These variations in energy may impact the characteristics of NDC coatings. The influence of TSD on film growth and mechanical features is one of particular interest to gain a comprehensive understanding of the deposition process and to optimize the fabrication of high-quality NDC films. Studies on various materials, including titanium aluminum nitride [47], SZO [48], and a-C coatings [49], have demonstrated the significant impact of TSD on microstructural, crystal quality, electrical properties, and hardness. In nanostructured ternary nitride coatings and a-C films, increasing TSD results in higher film thickness and a rise in the sp^3/sp^2 ratio, leading to increased hardness and electrical resistivity up to an optimum distance. Beyond this optimum distance, hardness and electrical resistivity start to decrease. The findings emphasize the importance of carefully selecting the target-substrate distance to achieve high-quality films with desired mechanical properties.

Based on the above literature, the authors focus on exploring the impact of target-substrate distance on the growth and mechanical characteristics of NDC coatings. Our investigation involves evaluating the hardness, Young's modulus, adhesion, and surface morphology of NDC films at various TSDs (ranging from 5 mm to 50 mm) while maintaining consistent deposition conditions. Through this research, we aim to gain valuable insights into the correlation between target-substrate distance and NDC characteristics.

2. MATERIALS AND METHODS

NDC films were fabricated on unheated WC-Co substrates using the coaxial arc plasma deposition (CAPD) technique (ULVAC, APG-1000) as shown in Figure 2. The TSD was varied from 5 to 50 mm. The CAPD setup included a high-purity graphite rod equipped in a coaxial arc plasma gun. Prior to deposition, the cylindrical chamber was evacuated by a set of rotary and turbomachinery pumps to a base pressure below 10^{-4} Pa. The arc plasma gun operated at 100 V with a repetition rate of 1 Hz and a 720 µF capacitor.

The mechanical properties of the fabricated samples were assessed using a nano-indenter (picodentorHM500), and surface morphologies were observed by scanning electron microscopy (SEM, JEOL JSM-6500F). Based on film thickness and deposition time, the deposition rate was estimated. Adhesion strength was assessed through a blasting adhesion test using silicon carbide particles. The blasting experiments were done using silicon carbide (SiC) particles ranging in size from 25 to 70 µm under dynamic blasting pressures of 0.35 MPa for 10 seconds. The adhesion assessment criterion is the time necessary to cause film separation in the blasting spot region. By observing the damaged regions and the volume removed by blasting with an optical microscope, the crucial blasting duration was determined.

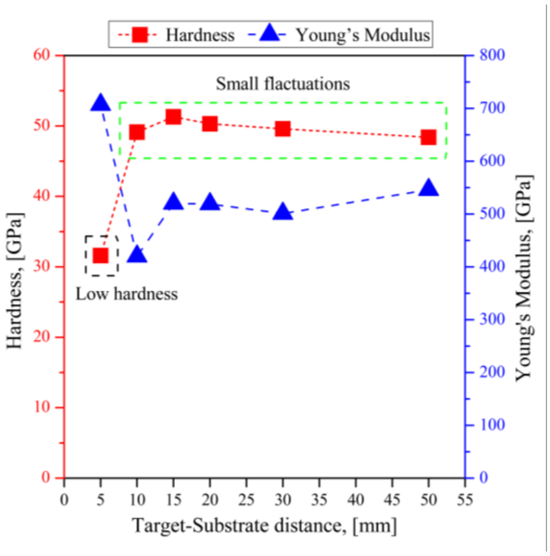

Figure 3. Nanoindentation measurements of NDC films at various distances between substrate and target.

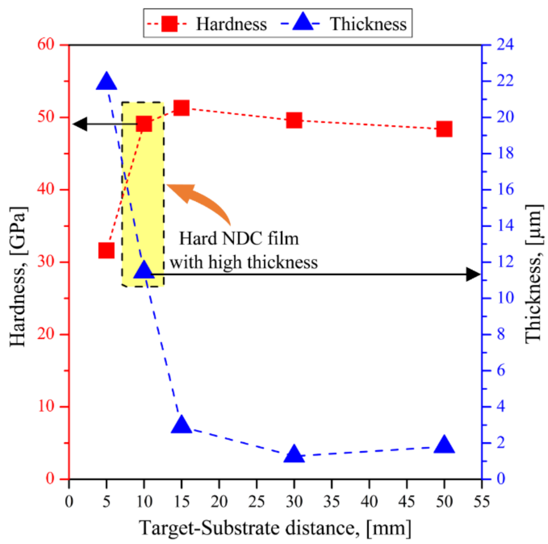

Figure 4. Nanoindentation measurements and film thickness of NDC coatings at different TSDs.

3. RESULTS AND DISCUSSION

3.1 Effects of target-substrate distance on the mechanical characteristics of NDC coatings

3.3.1 Hardness and Modulus of elasticity

Both of hardness and elastic modulus of the fabricated NDC coatings were evaluated using nanoindentation. Figure 3 demonstrates the hardness and modulus of elasticity variation as a function of target-substrate distance. An initial increase in hardness followed by stabilization was observed with increasing TSD. The higher hardness and larger modulus were attributed to effective suppression of undesirable graphitization owing to NDC fabrication at room temperature. This indicates

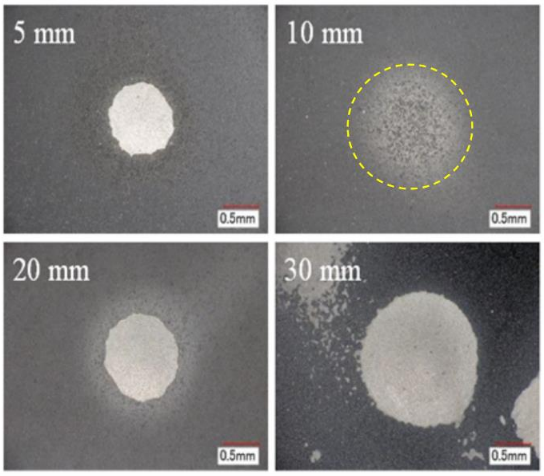

Figure 5. SEM images of blasting tests for NDC coatings utilizing SiC up to 10 s.

that hard NDC coatings showed a higher mechanical stability at a 10 mm-TSD (Figure 4), which also displayed good surface quality (Figure 8).

3.3.2 Adhesion

Apart from hardness and modulus of elasticity, the strength of coating adhesion is another crucial property of hard coatings. To evaluate the adhesion strength between the films and substrates, a blasting adhesion test was performed using silicon carbide particles (25–70 µm) (Figure 5). The film fabricated at a TSD of 10 mm showed excellent adhesion and was not affected by the blasting. On the other hand, there was a noticeable decrease in adhesion resistance as the TSD increased. Due to the dense arrival of high-energy atoms from the CAPD target, it seems that a stronger interaction was formed at the shorter TSD between the NDC film and the substrate interface. The coating deposited at 5 mm and room temperature did not exhibit improved adhesion with the decrease in distance, possibly due to

leading to a higher film thickness compared to other samples.

3.2 Effect of target-substrate distance on NDC growth and film properties

The plasma process of arc discharge and its impact on the growth of NDC coatings at several TSDs were investigated. NDC films were fabricated at TSDs ranging from 5 mm to 50 mm on WC-Co substrates, all at the same discharge energy (3.6 J/pulse). Analysis of the arc plasma emission showed that the proportion of C^+ ions in the plasma is remarkably high compared to C atoms and C_2 dimers [50, 51]. The energetic C^+ ions are believed to play a crucial role in diamond nucleation.

As the distance between the carbon target and the substrate increased (Figure 6 (3)), the number of C^+ ions reaching the substrate naturally decreased due to energy loss during travel. This change in TSD led to different supersaturated conditions, influencing subsequent diamond growth and films features. At the optimal TSD (Figure 6 (2)), a strong supersaturated state facilitated diamond grain growth, resulting in larger grains and increased film thickness. Conversely, at the lowest TSD (5 mm), the presence of macroparticles (Figure 6 (1)) negatively affected the supersaturated state, leading to degraded mechanical properties.

The deposition rate of NDC coatings was significantly affected by TSD (Figure 7). Deposition at 5 mm TSD yielded a rate of 2.19 nm/pulse, while at 50 mm TSD, it dropped to 0.19 nm/pulse [50, 51]. This decline in deposition rate can be revealed to the reduced efficiency of the coating process due to the deceleration of carbon species ejected from the graphite cathode with increasing TSD.

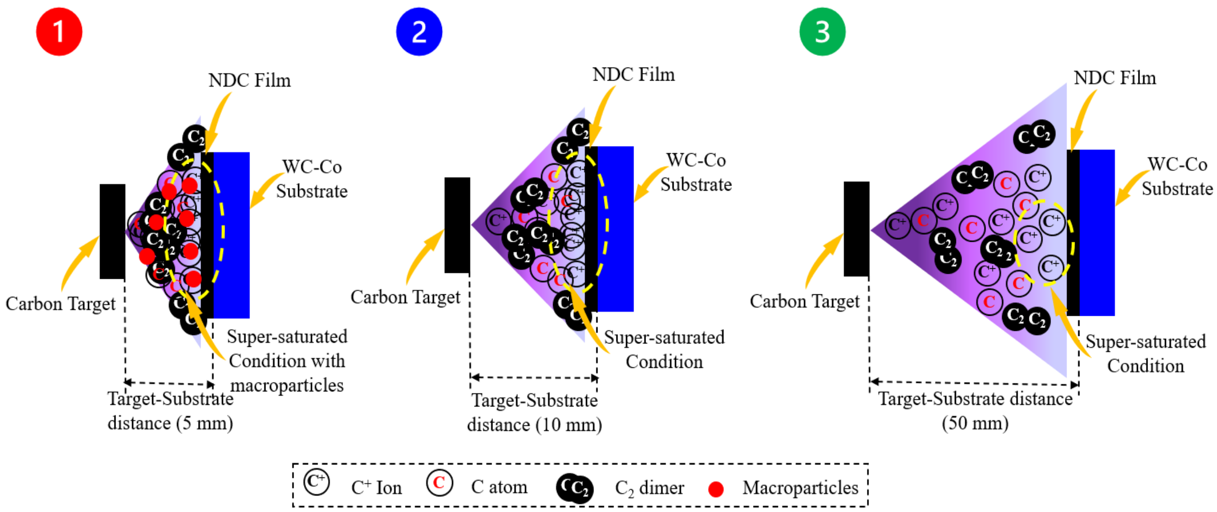

Figure 6. Illustration graph for the growth procedure of NDC film during deposition at different target-substrate distances: (1) 5 mm, (2) 10 mm, and (3) 50 mm.

residual stresses generated in this sample as a result of the high arrival of energetic atoms to the substrate surface,

Surface morphologies of the NDC films (Figure 8) showed that all substrates were completely coated with NDC films,

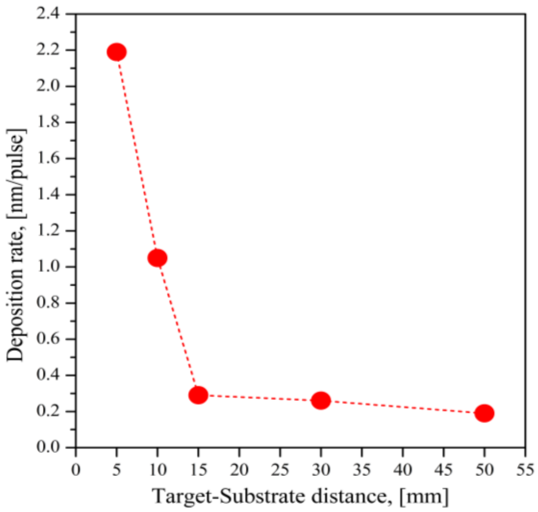

Figure 7. Deposition rate of NDC films at various distances between the substrate and target.

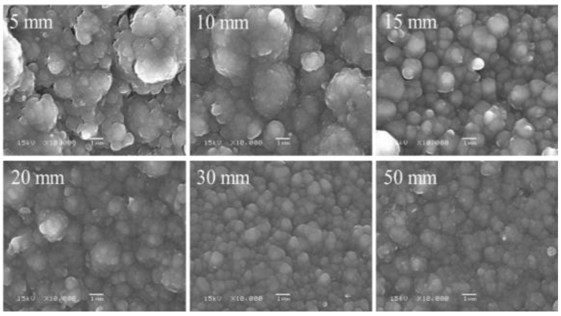

Figure 8. Top view SEM images of several NDC films.

exhibiting a homogeneous surface. Nanodiamond crystal clusters embedded in an a-C matrix were the significant cause of the protruding areas. These clusters became smaller and denser as the target-substrate distance increased. Additionally, TSD significantly influenced film thickness, decreasing sharply from 21.88 µm to 1.8 µm in the range of 5 mm to 50 mm [50, 51]. This reduction in film thickness might be associated with a decrease in film density, influenced by various factors such as residual stress, hardness, defect density, surface roughness, and crystallinity.

4. COMPARISON BETWEEN THE CURRENT AND PREVIOUS WORK

NDC films deposited without heating substrate exhibit higher hardness and Young's modulus compared to typical DLC films and are comparable to ta-C coatings as displayed in Figure 9. Furthermore, unlike ta-C films with thickness limitations in the range of a few hundred nanometers, NDC hard coatings can be fabricated to thicknesses of several micrometers (≥ 11 µm) comparable to diamond coatings. This unique characteristic sets NDC films apart from traditional DLC films and makes them highly attractive for various applications.

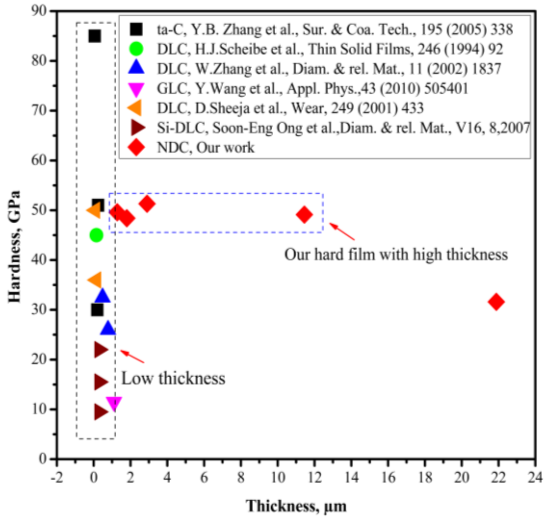

Figure 9. Comparison between NDC films and hard a-C films.

5. CONCLUSIONS

In this study, NDC films were synthesized using the CAPD method, and the impact of target-substrate distance on film growth and mechanical characteristics was thoroughly analyzed. The results indicated that as the TSD increased, the thickness of NDC film decreased, in addition to demonstrating a reduction in the density and size of nanodiamond clusters. In particular, the hardness and elastic modulus of the NDC coatings exhibited an initial increase followed by stabilization by increasing TSD. The NDC films fabricated at a target-substrate distance of 10 mm displayed superior adhesion resistance, high hardness (49.12 GPa), high modulus (420 GPa), and a substantial thickness (11.45 µm) with a uniform surface morphology. The study underscored the significant influence of target-substrate distance as a critical process parameter controlling the structural and physical properties of NDC films prepared by coaxial arc plasma deposition. These findings have practical implications for tailoring nanodiamond composite coatings with enhanced performance in various engineering applications.

Highlights

- Nanodiamond composite (NDC) films were successfully fabricated using the eco-friendly CAPD technique.
- The study explored the influence of target-substrate distance on the growth and mechanical characteristics of NDC films.
- The research resulted in the production of a thick and robust NDC film on WC-Co substrates without peeling or defects.
- The deposition rate exhibited a sharp decline with increasing target-substrate distance.
- Optimal mechanical properties and growth were achieved at a TSD of 10 mm.

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