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Technology for Preparing Nanocrystalline Diamond Coatings by Hot-Filament CVD Method

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Abstract: This paper systematically investigates the core technical system for preparing nanocrystalline diamond coatings (NDC) by hot-filament chemical vapor deposition (HFCVD), so as to provide theoretical and process references for the industrial development and cross-field applications of this technology. Nanocrystalline diamond coatings have a grain size of 5–100 nm, combining ultra-high hardness, exceptional surface finish, excellent thermal conductivity and strong chemical inertness. With the merits of low equipment cost, large deposition area and high controllability of process parameters, HFCVD has become the core method for the industrial preparation of NDC. This paper reviews the development history of HFCVD technology and NDC, elaborates on the core processes in preparation including substrate selection, gas source design, hot-filament matching and pretreatment, analyzes the working principle of the equipment, the preparation flow and the optimization mechanism of key process parameters, summarizes the application status of the coatings in multiple fields, clarifies the industrialization bottlenecks of the current technology, and proposes key research directions for future efforts.

Keywords: Hot-filament Chemical Vapor Deposition (HFCVD); Nanocrystalline Diamond Coating (NDC); Preparation Process; Superhard Coating
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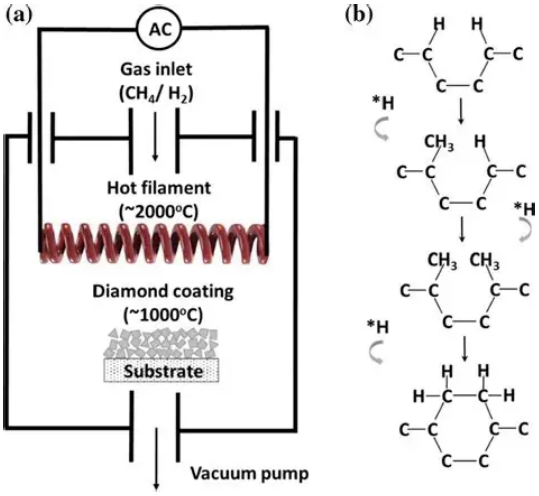
Introduction

With the rapid development of high-end manufacturing towards high precision, long service life and low energy consumption, superhard functional coating technology has become a key breakthrough for improving the service performance of core components. Among them, nanocrystalline diamond coating specifically refers to a diamond-phase thin film with a grain size of 5-100 nm and sp^3-bonded carbon atoms as the main component. Compared with traditional micro-scale diamond coatings, it achieves both ultra-hard properties and high surface finish by virtue of the high-density grain boundaries formed by nano-grains, and also retains the intrinsic ultra-high thermal conductivity and chemical inertness of diamond materials. It has shown irreplaceable application value in fields such as precision machining, aerospace seals, biomedical implants, and high-end electronic devices.

Hot-Filament Chemical Vapor Deposition (HFCVD) is the mainstream industrial process for preparing nanocrystalline diamond coatings. Its core principle is to crack the mixed reaction gas of CH_4/H_2 through high-melting-point metal wires electrically heated to 2000-2200 °C, initiate gas-phase chemical reactions on the substrate surface several millimeters away from the hot filaments, and ultimately achieve the oriented deposition of carbon atoms in the diamond phase on the substrate.

Compared with other CVD technologies, HFCVD technology has the advantages of simple equipment structure, low investment cost, capability of batch deposition of large-size workpieces, and flexible regulation of process parameters, which can adapt to different substrate materials and coating performance requirements. It is currently the preferred technology for the industrial preparation of nanocrystalline diamond coatings.

Figure 1. Schematic Diagram of HFCVD



Currently, the industrialization process of nanocrystalline diamond coatings still faces multiple technical bottlenecks: on the one hand, HFCVD technology has inherent problems such as weak substrate adhesion, low deposition rate (usually <1 μm/h), and poor uniformity in large-area deposition; on the other hand, the standardized evaluation system for coating performance has not yet been improved, and the lack of cross-industry application standards restricts the transformation efficiency of technological achievements. Against this background, major industrial countries around the world have incorporated advanced coating technology into their new material development strategies. China's "14th Five-Year Plan" clearly lists "high-performance coating materials" as a key research field for cutting-edge new materials, and the European Union and the United States have also successively invested special resources to support the innovation of surface engineering technology. Policy orientation has promoted the research and development focus to shift from single process optimization to the whole-chain innovation of "material-equipment-application", and at the same time driven the evolution of HFCVD technology towards green manufacturing with low temperature, low pressure and low carbon.

This paper, by systematically sorting out the technical system for preparing nanocrystalline diamond coatings by HFCVD, integrates the core contents of material selection, process regulation, application scenarios and technical bottlenecks, aiming to provide a systematic reference for the breakthrough in basic research and the promotion of industrial application of this technology.

1. Core Materials and Pretreatment Technology for the Preparation of Nanocrystalline Diamond Coatings

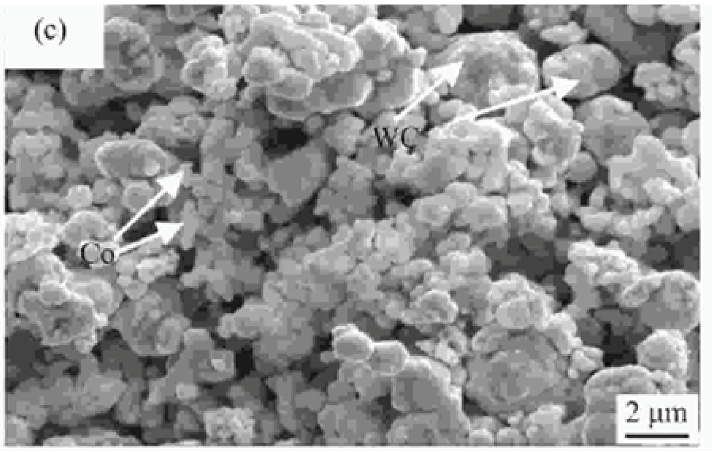
The selection and pretreatment of preparation materials are the foundation for determining the final quality of nanocrystalline diamond coatings, which directly affect the nucleation efficiency, crystalline purity, interface adhesion and service performance of the coatings. It mainly includes three core components: substrate materials, gas source system and hot-filament materials, as well as the supporting substrate pretreatment process.

1.1 Selection and Compatibility of Substrate Materials

The substrate is not only a physical support for coating deposition, but its thermal expansion coefficient, crystal structure, and high-temperature chemical stability directly affect the nucleation and growth of the coating, the internal stress level, and the interface bonding effect. In the HFCVD process, the substrate must withstand a deposition temperature of 300-900 °C and form a stable bonding interface with the diamond material to avoid coating cracking and peeling caused by thermal mismatch. The characteristics and suitable scenarios of commonly used substrate material classifications are as follows:

Cemented Carbide: Centered on WC-Co cemented carbide, its hardness can reach HV 1500-1800, with excellent mechanical strength, which can form performance synergy with diamond coatings, making it the preferred substrate material for cutting tools and wire drawing dies. Its core defect is that the surface Co binder phase will catalyze the graphitization of diamond at high temperatures, damaging the bonding between the coating and the substrate. Therefore, the surface Co phase must be removed through pretreatment. Among them, the WC-Co cemented carbide with a cobalt content ≤6 wt% can have a bonding strength with the coating increased to more than 50 MPa after pretreatment.

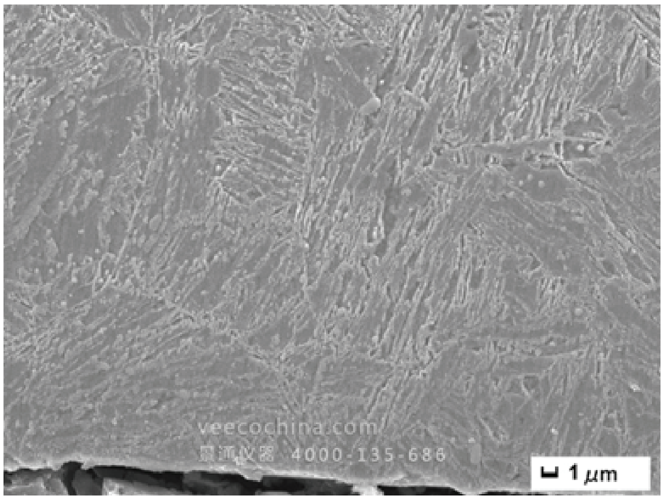
Figure 2. Typical SEM Image of WC-Co Powder



Silicon Materials: Including monocrystalline silicon and polycrystalline silicon, they have a regular lattice structure, a diamond nucleation density of up to 10^9 cm^{-2}, good compatibility with microelectronic processes, and a thermal expansion coefficient slightly different from that of diamond, which can reduce thermal stress cracks during the deposition process. They are the core substrate materials for coatings of microelectronic devices and micro sensors. Their disadvantages are low mechanical strength and high brittleness, which make them unable to withstand heavy-load working conditions.

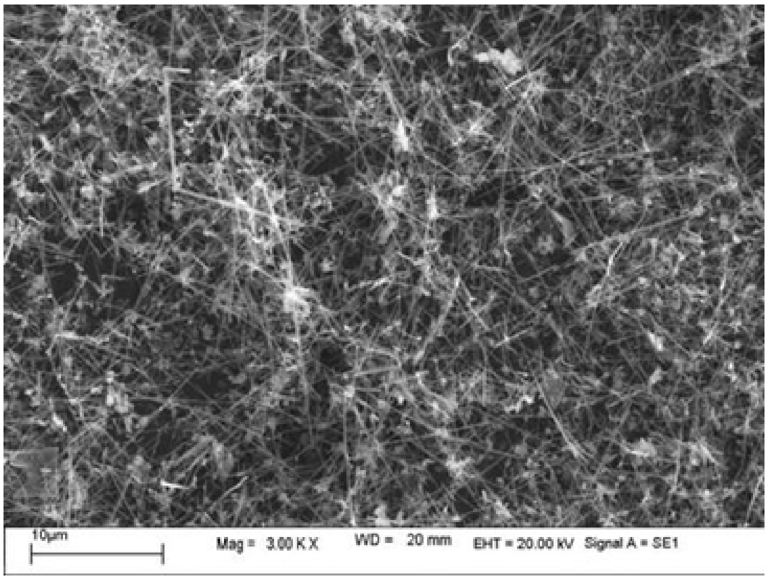
Metal Materials: Including titanium alloys, stainless steel, molybdenum, etc. Titanium alloys have strong corrosion resistance, stainless steel has low cost, and molybdenum has a high melting point, which can adapt to different working condition requirements. The core problem of this type of material is that their thermal expansion coefficient is greatly different from that of diamond, which is prone to generate huge internal stress after deposition, leading to coating peeling. It is necessary to prepare a transition layer to alleviate the problem of thermal mismatch, and they are mainly used in the fields of aerospace structural components, corrosion-resistant components, and medical surgical instruments.

Figure 3. Typical SEM Image of Martensitic Stainless Steel



Ceramic materials: Including SiC, Al_2O_3, Si_3N_4, etc. They have excellent high-temperature resistance. The long-term service temperature of SiC can reach 1600℃, with strong chemical stability, and the thermal expansion coefficient is closer to that of diamond. The internal stress level of the coating is low, which can effectively reduce the risk of coating cracking. The disadvantages of this type of material are high brittleness and high difficulty in processing complex shapes. They are mainly used in extreme working conditions such as high-temperature wear-resistant components and insulating devices.

Figure 4. Typical SEM Image of SiC Powder



1.2 Design of Gas Source System

The gas source system of the HFCVD process is the material foundation for the growth of diamond coatings, which directly determines the carbon source supply, phase purity and grain structure of the coatings. It is mainly divided into three categories: carbon source gas, hydrogen and doping gas. The functions and ratio design of various gases are as follows:

Carbon source gases: Commonly used gases include methane (CH_4), acetylene (C_2H_2), acetone (C_3H_6O), etc. Methane is the most commonly used carbon source, with a simple molecular structure and stable reaction. It decomposes under the high temperature of the hot wire to generate carbon-containing active groups, which is suitable for the controllable growth of nanodiamond coatings. Acetylene has a high carbon content and can achieve a higher deposition rate, but the reaction process is difficult to control; acetone is more suitable for the uniform deposition on substrates with complex shapes.

Hydrogen (H_2): Hydrogen is indispensable in the process of preparing nanodiamond coatings by hot filament CVD. On the one hand, it is used to dilute the carbon source gas, regulate the carbon concentration of the reaction system, and prevent the excessive formation of non-diamond phases; on the other hand, it decomposes at high temperatures to generate hydrogen atoms, which preferentially etch sp^2-hybridized non-diamond carbon, promote the dominant growth of sp^3-hybridized diamond phases, and ensure the phase purity of the coating.

Doping Gases: Commonly used ones include nitrogen (N_2) and borane (B_2H_6). Doping gases can specifically regulate the grain size, mechanical properties and electrical properties of the coating. For example, the N atoms formed by the decomposition of nitrogen can accelerate the hydrogen extraction step in the reaction process, increase the secondary nucleation rate, refine the diamond grains, and improve the hardness of the coating; borane doping can realize the p-type semiconductor modification of the diamond coating, expanding its application in the field of electronic devices.

Table 1. Gas Sources and Reaction Gases for HFCVD

Gas Types	Commonly Used Gases	Functions and Roles	Typical Ratios
Carbon Source Gases	Methne (CH_4)[8, 10],Acetylene (C_2H_2), Acetone (C_3H_6O)	Provide carbon atoms required for deposition	1-5 vol%
Carrier Gas / Diluent Gas	Hydrogen (H_2)[6-9], Argon (Ar)	Dilute the carbon source and promote the formation of sp^3 bonds	95-99 vol%
Doping Gases	Nitrogen (N_2)[7], Borane (B_2H_6)	Regulate electrical properties (n-type / p-type)	100-5000 ppm

1.3 Hot Filament Material Selection

The hot filament is the core component of an HFCVD system and serves as the energy source for the cracking of reaction gases. Its performance directly determines the process stability and coating quality. It must meet four core requirements: high melting point, good high-temperature mechanical strength, low vapor pressure, resistance to carburization, and stable resistance-temperature characteristics.

In HFCVD processes, the hot filament must be heated to 1800–2400 °C to achieve efficient cracking of hydrogen and carbon-source gases. For this reason, the hot filament material must have a melting point above 3000 °C to avoid melting or deformation at high temperatures. Meanwhile, it should maintain good high-temperature mechanical strength to prevent bending and sagging of the suspended filament under high heat, which would cause uneven filament–substrate spacing and lead to variations in coating thickness and quality. Low vapor pressure and carburization resistance reduce coating contamination caused by high-temperature evaporation of the filament, avoid corrosion of the filament by carbides, and extend its service life. Stable resistance-temperature characteristics enable precise control of filament temperature, ensuring consistency in batch processing.

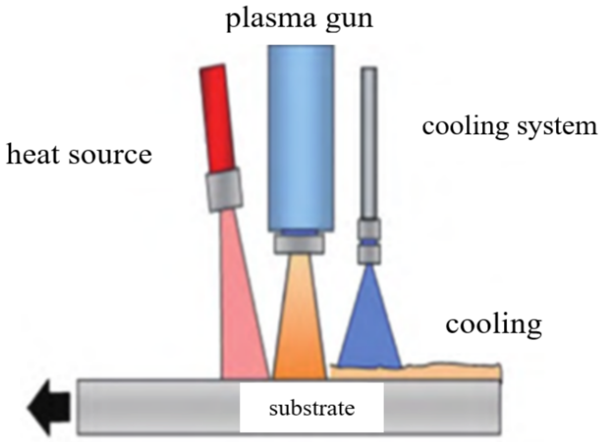
Commonly used hot filament materials at present include tantalum (Ta), tungsten (W), rhenium (Re) and their alloys. The filament diameter is usually 0.1–0.5 mm, mostly arranged in a parallel array with a spacing of 5–15 mm. Among them, tungsten filaments [7, 8] are the most widely used. With a melting point of 3422 °C, they can operate stably for a long time at 2000–2300 °C, and feature good linearity in resistance-temperature characteristics and high temperature control accuracy. Tantalum filaments exhibit excellent high-temperature strength and corrosion resistance, making them suitable for long-period continuous deposition processes. Rhenium alloys have the strongest carburization resistance, with a service life significantly better than that of tungsten and tantalum, but at a higher cost.

1.4 Key Processes of Substrate Pretreatment

Substrate pretreatment is a key step to improve coating-substrate adhesion and diamond nucleation density. It mainly includes three core processes: surface roughening, chemical etching and interlayer design, together with supporting pre-deposition activation treatments such as hydrogen baking and filament baking.

Surface roughening: Its core function is to form a mechanical anchoring effect by roughening the substrate surface, increase the actual contact area between the coating and the substrate, and improve the interfacial bonding strength. The standard process adopts sandblasting: using 20–50 μm Al_2O_3 abrasives as the medium, the substrate surface is blasted at an incident angle of 60° under a pressure of 0.5 MPa to obtain an optimal roughness of Ra=0.8–1.2 μm, which can increase the coating bonding strength by 40%–60%. Excessive roughening will cause stress concentration in the coating and lead to early spalling, so the upper limit of roughness must be strictly controlled.

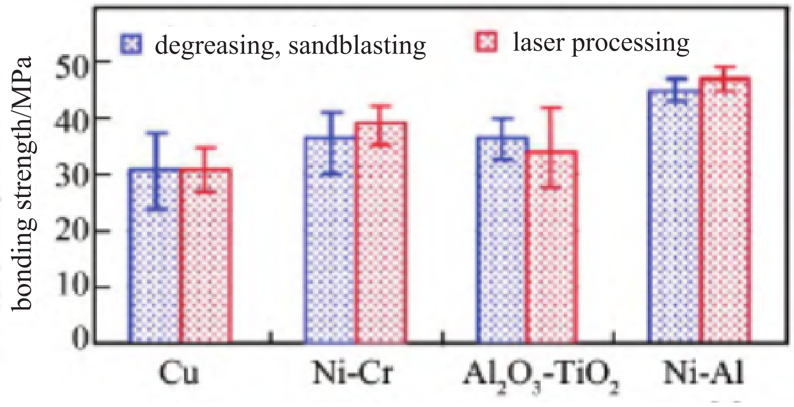
Figure 5. Schematic diagram of synchronous laser thermal spraying [3]



Researchers integrated laser cleaning equipment with thermal spraying equipment (including plasma gun, heat source and cooling system) to develop a synchronous laser thermal spraying process, which realizes simultaneous laser cleaning of the substrate and coating deposition during thermal spraying, as shown in Figure 5. The plasma gun is the key component, which sprays molten or semi-molten particles such as Ni-Al and Al_2O_3 onto the substrate surface under compressed gas flow. The heat source supplies energy to the plasma gun to ensure the sprayed materials reach the required state for film formation. The cooling system cools the equipment and controls the substrate temperature (≤200°C) to avoid high-temperature damage and ensure mechanical bonding between the coating and the substrate. Synchronous laser cleaning removes the micron-scale oxide layer on the substrate to provide a clean deposition interface, and continuous cooling ensures stable operation of the equipment and substrate. This process breaks through the limitation of separate traditional pretreatment and spraying, and provides a technical solution for improving coating bonding performance, extending service life and optimizing production efficiency.

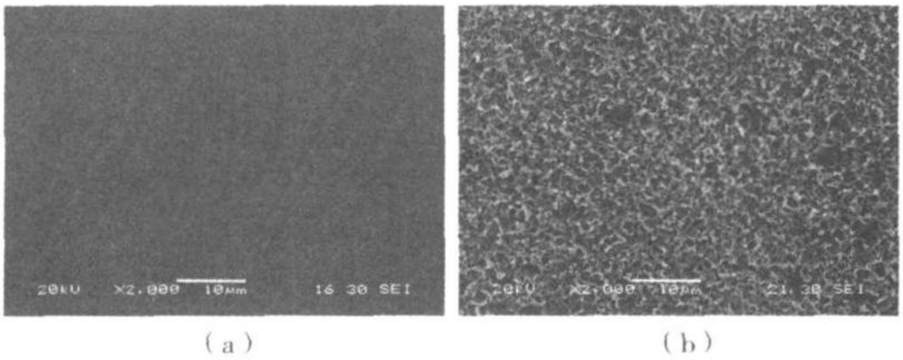
L Qin, L Ping et al. [4] compared the effects of "degreasing + sandblasting" and laser pretreatment on the bonding strength of Cu, Ni-Cr, Al_2O_3-TiO_2 and Ni-Al coatings. The results in Figure 6 show that after laser cleaning, the bonding strength of Cu, Ni-Cr and Ni-Al coatings is equivalent to or higher than that of the sandblasted group (the interfacial toughness of Ni-Al coating increases from 0.39 $MPa\cdot m^{1/2}$ to 2 $MPa\cdot m^{1/2}$). Only the high-melting-point Al_2O_3-TiO_2 coating exhibits increased interfacial porosity and lower bonding strength after laser cleaning, due to the semi-molten state of most particles and the smooth surface (Ra<1μm). The results quantitatively compare different pretreatment methods and verify the suitability of laser cleaning for Cu and Ni-based coatings, but do not analyze the regulation of laser parameters. It provides experimental support for the selection of pretreatment methods, and suggests that high-melting-point ceramic coatings need laser etching to optimize surface roughness for improved bonding strength.

Figure 6. Effect of laser cleaning on coating bonding strength[4]



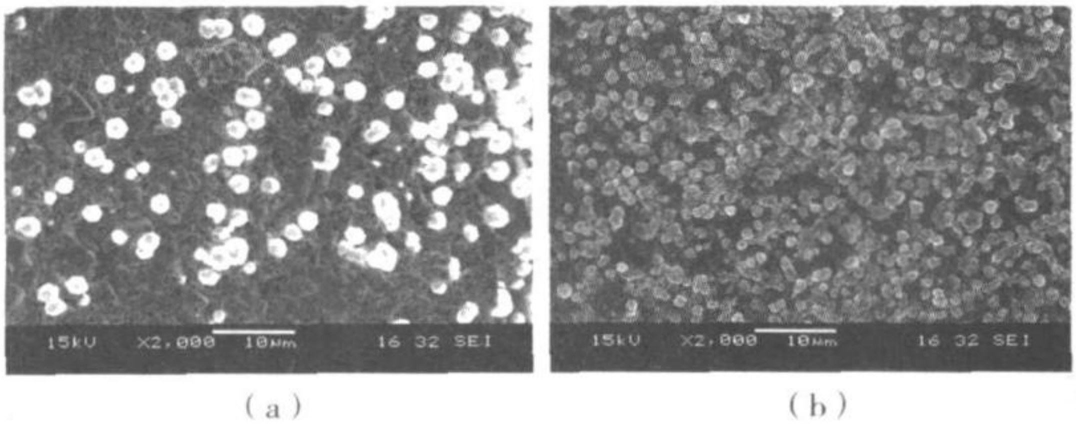
Chemical etching: It is mainly applied to WCCo cemented carbide substrates to remove the surface Co binder phase and eliminate its catalytic graphitization interference on diamond growth. The mainstream process is the "alcoholalkali twostep method" [5]. The substrate is first ultrasonically cleaned in methanol solution for 20 minutes to remove surface oil contamination, followed by ultrasonic etching in a $KOHK_3Fe(CN)_6H_2O$ mixture. This reduces the surface Co content from 6% to 0.4%–0.83%, increases surface roughness from 94.5 nm to 366 nm, and achieves a diamond nucleation density up to 10^9 cm^{-2} — far higher than the conventional acid pickling and grinding process. It is also suitable for pretreatment of complexshaped substrates such as drills and dies.

Figure 7. Comparison of surface morphologies of the substrate before and after two-step treatment



(a) (b)

Figure 8. Nucleation density on the substrate surface under different treatment conditions



(a) (b)

Interlayer design: Its core function is to alleviate the thermal expansion coefficient mismatch between diamond and the substrate, eliminate interfacial internal stress, and prevent high-temperature diffusion of elements such as Co from the substrate, ensuring high-quality growth of the diamond coating. The mainstream scheme adopts a Ti/TiC/TiN graded interlayer system, following four principles: gradual matching of thermal expansion coefficients, strong interfacial adhesion, stable chemical properties, and suppression of element diffusion. The Ti layer (0.3–0.5 μm) acts as an activation layer to enhance surface activity of the substrate; the TiC layer (1–2 μm) serves as a thermal matching layer with a thermal expansion coefficient between that of the substrate and diamond, smoothly transferring interfacial stress; the TiN layer (0.5–1 μm) functions as a diffusion barrier layer, which can react with Co to form a stable Co_3TiN phase and reduce the Co diffusion depth from 500 nm to below 80 nm. This gradient system reduces the interfacial residual stress from 8 GPa to 3.2 GPa, significantly improving the spalling resistance of the coating.

Pre-deposition activation treatment: includes filament carburization treatment (“filament baking”) and substrate hydrogen treatment (“hydrogen baking”).”Filament baking” involves heating tungsten filaments to 2300 °C and holding for 0.5 h to complete carburization and ensure stable working performance of the filaments. “Hydrogen baking” uses bombardment by high-temperature hydrogen atoms and ions on the substrate surface in a hydrogen atmosphere to remove surface impurities and oxide layers, providing a clean and active surface for diamond nucleation and further increasing nucleation density.

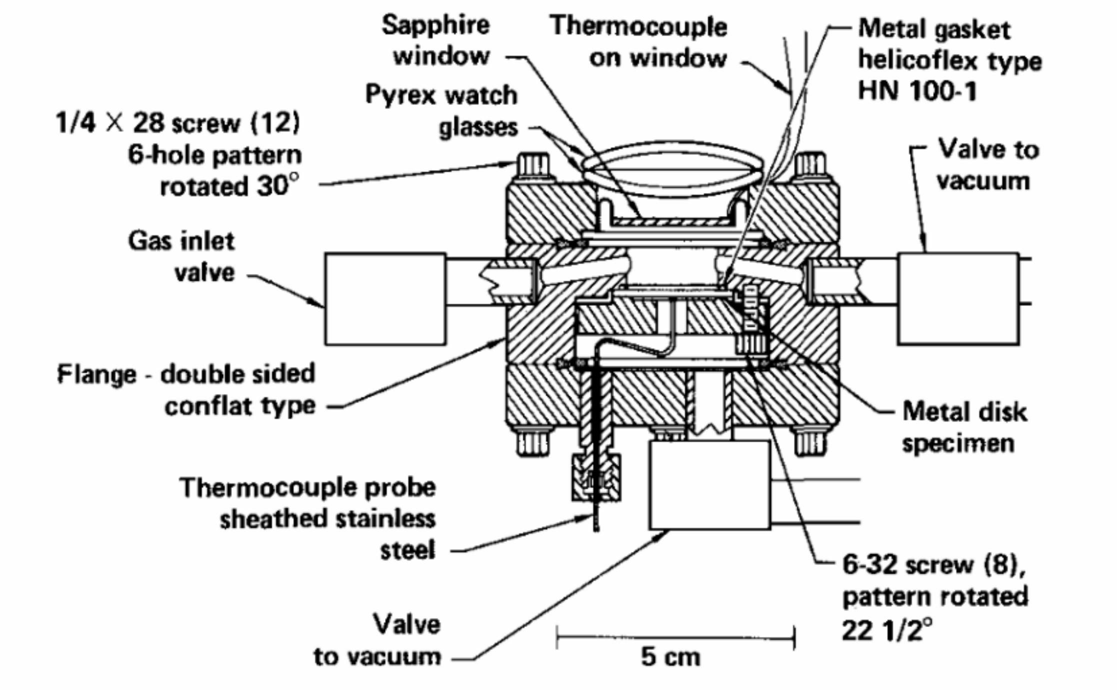
2 Process Principle and Parameter Control of Hot Filament CVD Preparation

2.1 Technical Principle and Equipment Structure

The basic principle of HFCVD technology is to heat a high-melting-point metal hot filament to 1500–2300 °C by electric current, so that the CH_4/H_2 mixed reaction gas introduced into the reaction chamber undergoes high-temperature pyrolysis on and near the filament surface, producing various active species such as atomic hydrogen and methyl radicals. Driven by concentration and thermal gradients, these active species migrate to the substrate surface maintained at 700–1000 °C. Through a series of complex surface reactions including adsorption, desorption and surface diffusion, diamond nucleation and continuous growth are completed, eventually forming a continuous nanodiamond coating on the substrate surface. Throughout the process, the selective etching effect of atomic hydrogen is critical. It preferentially removes sp^2-hybridized non-diamond carbon formed on the substrate surface, ensuring the dominant growth of the sp^3-hybridized diamond phase, which is the key to obtaining high-purity diamond coatings.

Vacuum Reaction Chamber[11]: It is the core space for coating deposition, usually made of 304L stainless steel. A high vacuum level above 10^{-5} Torr is required to isolate contamination from air impurities during coating growth. It is also equipped with a sapphire observation window and heating and temperature control components to realize real-time monitoring of the deposition process and precise temperature control, eliminating uneven coating thickness and composition caused by edge effects.

Figure 9. Cross-section of a cylindrical vacuum chamber for edge-effect-free studies of gas–metal reactions [11]



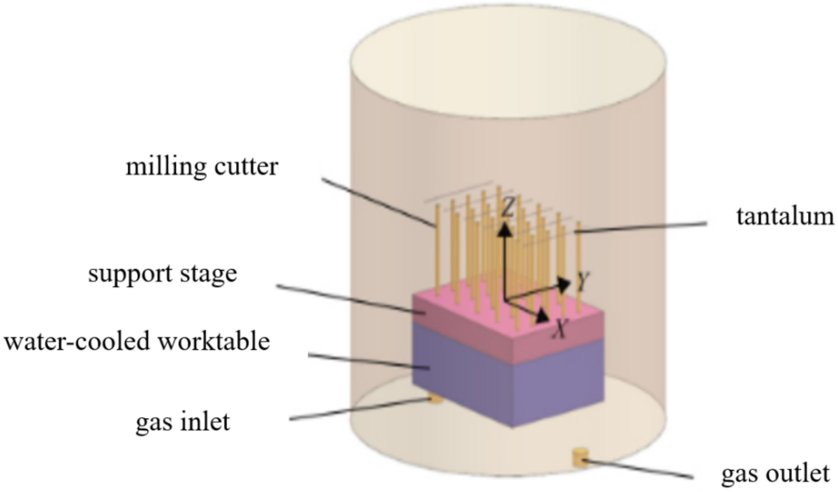
Gas Delivery System: It precisely controls the flow ratio of CH_4/H_2, the core gases for diamond growth (a CH_4 concentration of 3% ensures sp^3-bond-dominated growth), stabilizes the generation efficiency of carbon-active species, and avoids an increase in graphite phase caused by excessively high concentration or a decrease in nucleation density caused by excessively low concentration. Doping gases such as N_2/Ar allow precise tuning of grain size (e.g., N_2 doping achieves nanodiamond grains smaller than 30 nm). Through quantitative gas control, the system realizes precise regulation of coating composition and structure, ensures batch-to-batch consistency, and avoids performance drift caused by fluctuations in gas proportion.

Hot Filament Power Supply System: It maintains tungsten/tantalum filaments at a high temperature of 2000–2200 °C via stable electric current, serving as the core energy source for H_2 decomposition into H atoms for etching and purification and CH_4 decomposition into carbon-active groups for diamond growth in HFCVD. Current instability can cause temperature fluctuations of ±50 °C, reducing the H_2 decomposition rate from 90% to 75%, resulting in abnormal carbon source behavior or increased impurity carbon. The system ensures continuous and efficient energy supply from the hot filament through steady-current temperature control, avoiding deposition interruption or degraded coating quality.

Substrate Heating System: It provides precise temperature control for different substrates (600–900 °C for cemented carbide, $<$ 500 °C for polymers), avoiding substrate damage and optimizing coating adhesion. In the cemented carbide temperature range, it suppresses surface diffusion of Co, reduces graphitization interference, and improves interfacial bonding strength. In the low-temperature range for polymers, it preserves the structural integrity of the substrate. Through adaptive temperature control, the system protects the substrate while providing suitable surface energy for diamond nucleation, solving the compatibility problem between the substrate and the diamond coating.

In Figure 10, the components of the HFCVD apparatus work synergistically for precise control of the tool temperature field: tantalum filaments at 2000–2200 °C produce radiant heat to drive the decomposition of CH_4/H_2 into carbon-active species; the milling cutter must be kept within the 600–900 °C process window, whereas conventional structures easily suffer from thermal accumulation and overheating above 950 °C, which induces graphite phase formation. After flow channel optimization of the water-cooled worktable, the temperature gradient is reduced from 25 °C/mm to 8 °C/mm. The coppermolybdenum composite support stage balances thermal conductivity and service life, achieving a threefold lifetime extension over pure copper. The 45° oblique gas inlet nozzles generate a swirling flow, raising the deposition rate by 12%. Through the “heat generation–heat transfer–heat dissipation” chain, all components realize precise temperature control and avoid the risk of thermal runaway.

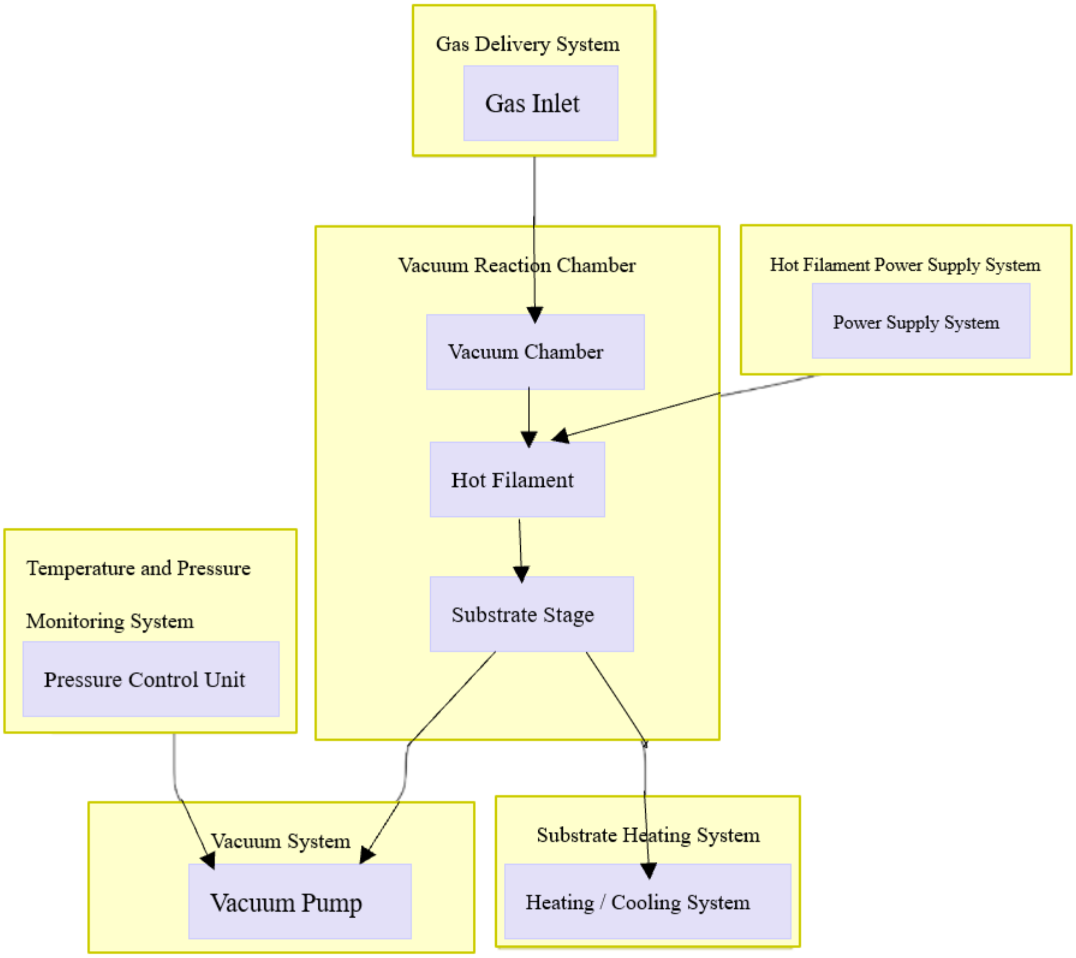
Figure 10. Simulation model of the HFCVD system [12]



Vacuum System: Composed of a mechanical pump, molecular pump and other components, it enables dynamic pressure control within 10^{-3}–10^{3} Pa. Before deposition, the chamber is pumped to high vacuum to remove residual impurity gases; during deposition, a stable reaction pressure is maintained to provide a suitable pressure environment for coating growth and ensure the steady diffusion and reaction of process gases.

Temperature and Pressure Monitoring System: It collects key process parameters such as hot filament temperature, substrate temperature and chamber pressure in real time. Automatic adjustment of parameters is realized through a closed-loop control system, eliminating parameter fluctuations caused by equipment aging, gas flow drift and other factors. This ensures the stability of each deposition batch and supports the industrial mass production of coatings.

Figure 11. Schematic Diagram of the Hot Filament CVD (HFCVD) Apparatus



2.2 Standard Fabrication Process Flow of HFCVD Diamond Coatings [8, 10, 13]

Reaction Chamber Preparation and Evacuation: The cleaned AISI 316 stainless steel substrate is placed at the designated position in the quartz tube reaction chamber, and the coiled tungsten filament is fixed in place. A precise and constant filament-to-substrate distance of 3–10 mm is maintained to avoid uneven heating. The chamber is then evacuated with a vacuum pump to a high vacuum of 10^{-5}–10^{-7} Torr to remove impurities such as oxygen and water vapor, preventing filament oxidation and non-diamond carbon formation, thus ensuring coating purity and interfacial adhesion strength.[7]

Heating of Hot Filaments and Introduction of Process Gases: The hot filament power supply is activated to heat the filaments to the set temperature (typical value: 2300 ℃).[8] The reactive gases such as H_2 and CH_4 are decomposed by high-temperature radiation to produce carbon active species required for diamond nucleation. Meanwhile, deposition gases are introduced in proportion, commonly hydrogen, methane and nitrogen, with the flow ratio of methane to hydrogen being 1:30-100 and the flow ratio of nitrogen to methane being 1:0.1-3.[1] Precise control of carbon source concentration can avoid graphitization or slow growth, and nitrogen doping can regulate grain size, providing core support for the regulation of coating structure and properties.

Precise control of the substrate temperature: is maintained within the suitable growth range of 700°C–1000°C for diamond coatings using a heating device. An appropriate temperature promotes the adsorption and diffusion of active species, avoids loose coatings at low temperatures, suppresses graphitization at high temperatures, and reduces thermal mismatch internal stress. Accurate temperature control is critical to the quality and growth rate of nanocrystalline diamond coatings.

Coating Growth Stage: Under suitable temperature and gas conditions, active species adsorb and react on the substrate surface to form nanodiamond nuclei and grow continuously. The nuclei gradually connect and merge into a continuous coating. The deposition time ranges from several hours to tens of hours, and the coating thickness can be adjusted on demand by controlling the time (e.g., 3–8 μm for cutting tool coatings, thinner for microelectronic coatings) to adapt to different application scenarios. A continuous, pore-free coating can improve wear resistance and protection, providing structural guarantee for its functional performance.

Cooling and Post-Treatment: After deposition, turn off the hot filament power supply and gas passages, and allow the reaction chamber to cool naturally to room temperature to avoid a sudden increase in thermal stress between the coating and the substrate caused by rapid cooling, prevent coating cracking and spalling, and ensure structural integrity. Targeted post-treatment can be performed according to application requirements [2]： The coating on medical scalpels forms micro-nano structures through high-temperature oxidation to achieve super hydrophobicity and anti-blood adhesion; coatings for cutting tools and microelectronic devices are ground and polished to reduce surface roughness (e.g., Ra < 5 nm) [1] , to meet high-precision and flatness requirements and make coating properties more suitable for practical applications.

Table 2. Typical Process Steps of HFCVD

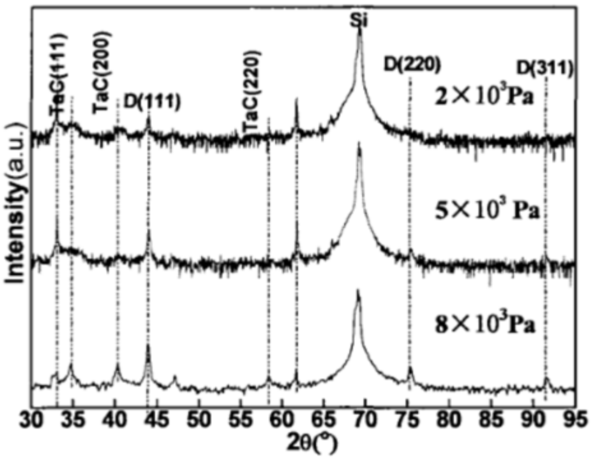
Step	Purpose	Parameter Range	Notes
Substrate Pretreatment	Enhance coating adhesion	Diamond grinding (0.5-1μm), acid etching (H_2SO_4/H_2O_2)	Thoroughly clean to remove residues
Chamber Evacuation	Remove impurity gases	Base vacuum $\leq 10^{-3}$ Pa	Ensure no leaks
Filament Activation	Stabilize the filament	2000-2200°C, 10-20min	Avoid uneven initial carburization
Deposition Process	Grow nanocrystalline diamond	CH_4/H_2=1-5%, 1-5kPa, 600-900°C	Control filament-substrate distance at 3-10 mm
Cooling and Sampling	Prevent thermal stress cracking	Cool to <150°C under inert atmosphere	Slow cooling rate <5°C/min

2.3 Core Process Parameter Optimization Mechanism

Effect of Carbon-to-Hydrogen Flow Ratio: It directly determines the carbon concentration in the reaction system, affecting the growth rate and quality of nanocrystalline diamond coatings. An excessively high carbon source proportion tends to form non-diamond phases and degrade coating quality, while an excessively low proportion leads to a slow growth rate; For example, in “Nanocrystalline Diamond Coating and Its Preparation Method and Process [1]”, the methane-to-hydrogen flow ratio for microcrystalline diamond coatings is 1:30–80, and the optimal coating quality can be obtained in the range of 1:40–60.

Effect of Reaction Pressure: It has a significant impact on coating growth. Low pressure facilitates the diffusion and adsorption of active species, promoting the growth of the diamond phase. However, an excessively low pressure may result in an overly slow growth rate, while high pressure tends to increase the probability of non-diamond phase formation. For example, in “Nanocrystalline Diamond Coating and Its Preparation Method and Process [1]”, the deposition pressure for nanocrystalline diamond coatings is 3–8.5 kPa. This range balances diffusion and reaction efficiency, maximizes active group concentration, and achieves a good balance between coating quality and growth rate.

Figure 12. X-ray diffraction patterns of diamond films deposited at different reaction pressures [14]



Effect of Filament and Substrate Temperatures: The filament temperature (around 2000°C) determines the decomposition efficiency of the reactive gases, which is critical for generating H atoms and carbon active species. Insufficiently low temperature leads to incomplete gas decomposition and slow coating growth, while excessively high temperature accelerates filament degradation and causes reaction runaway. The substrate temperature (600-900°C) regulates the surface behavior of active species, avoiding slow growth at low temperatures and the graphitization of diamond above 1200°C. The combination of 2000°C filament temperature and 850°C substrate temperature is a mature preparation parameter. [7]

Effect of Deposition Time: Deposition time directly determines the coating thickness; thickness increases with prolonged time, but excessively long deposition tends to increase internal stress and induce cracks. It is necessary to control thickness by adjusting time and manage stress through coordinated parameters to balance thickness and structural integrity. For example, 3–8 μm is commonly used for tool coatings, balancing wear resistance and crack resistance.[7]

Effect of Auxiliary and Diluent Gases: Auxiliary gases such as N_2 and Ar enable precise regulation of coating properties. N_2 can improve hardness and control nucleation density as well as grain size.

Substrate Temperature: Synergistically regulated by tungsten filament thermal radiation, gas convection and workbench cooling water, it directly affects microscopic reaction processes such as atomic migration and nucleation on the substrate surface. The optimal range is 600–900°C. Too low a temperature results in a slow deposition rate, while too high a temperature tends to form graphite phases. Since diamond graphitizes above 1200°C, the industry commonly uses a deposition temperature of 600~1100°C.

FilamentSubstrate Distance: Must be strictly controlled within 3–10 mm. A distance <3 mm easily causes overheating of the substrate and subsequent graphitization; a distance >10 mm reduces the active species concentration by 60%, causes the nucleation rate to drop sharply by three orders of magnitude, and halts growth. This range balances thermal radiation and active species transport, achieving coordinated matching of temperature zone and carbon source concentration.

Pressure and Gas Flow Rate: Pressure determines gas density and molecular collision probability; an appropriate pressure maximizes active group concentration, with a core range of 15 kPa. Flow rate determines gas residence time, typically 50200 sccm, which avoids defects and reduced compactness caused by abnormal residence and ensures coating uniformity.

Accurate regulation of the above parameters controls coating nucleation, grain size, phase purity and stress state, determines core properties such as coating hardness, adhesion and wear resistance, and serves as the key regulation basis for the industrial application of HFCVD technology.

3. Applications

With the maturity of HFCVD preparation technology, nanocrystalline diamond coatings have been industrially applied in various fields such as mechanical processing, biomedicine, electronics and optics, and special working conditions by virtue of their excellent comprehensive properties, solving performance bottlenecks that are difficult to break through with traditional materials.

3.1 Mechanical Processing and Cutting Tools

Mechanical processing is the most core application field of nanocrystalline diamond coatings. Its ultra-high hardness, low friction and high chemical stability can significantly improve tool wear resistance and service life, solving the pain points in machining difficult-to-process materials.

For example, the nanocrystalline diamond-coated cemented carbide tools prepared in “Nanocrystalline Diamond Coating and Its Preparation Method and Process [1]” exhibit greatly extended service life compared with uncoated tools when cutting difficult-to-machine materials such as fiber composites and hot-bent graphite molds. When machining aerospace aluminum alloys, titanium alloys, CFRP and other materials, the tool life is increased by 8–20 times, while cutting friction and built-up edge are reduced. This coating enables high-precision machining of aerospace high-strength aluminum alloys with a hole position tolerance of ±5 μm and surface roughness Ra < 0.4 μm. It reduces the machining surface roughness of CFRP from Ra 1.2 μm to below 0.8 μm, minimizing burrs and delamination. It also increases the number of tool regrinds and lowers the full-life-cycle cost. When applied to wire drawing and stamping dies, the service life can be prolonged by more than 10 times.

3.2 Biomedical Devices

Nanocrystalline diamond coatings exhibit great application value in the field of biomedical devices due to their good biocompatibility, strong corrosion resistance, excellent wear resistance and tunable surface properties. They are mainly used in surgical knives, dental instruments, orthopedic implants and other scenarios.

In the field of surgical blades, depositing a nanocrystalline diamond coating on the surface of stainless steel scalpels forms a dense physical protective barrier. Its self-corrosion current density is only 1/50 that of uncoated scalpels, effectively resisting corrosion from blood, tissue fluid and disinfectants. Meanwhile, the ultra-high hardness of the coating enables the scalpel to maintain long-term cutting edge sharpness and avoid edge rolling and wear [2]. The micro-nano composite structure coating prepared by high-temperature oxidation post-treatment can achieve a water contact angle of more than 150°, realizing a superhydrophobic state. The contact area between blood and the blade surface is only 1/5 that of traditional stainless steel blades, and blood residue is reduced by more than 90%, significantly decreasing visual obstruction during surgery and lowering the risk of cross-infection. This makes it ideally suitable for high-precision surgical scenarios such as minimally invasive surgery and cardiovascular surgery.

In the field of dental instruments, diamond-coated dental burs can reduce cutting edge wear by more than 60% during machining, improving the accuracy and efficiency of dental tissue processing. In the field of orthopedic implants, nanocrystalline diamond coatings on titanium alloy artificial joints and bone screws reduce the wear rate by more than 80% compared with traditional CoCr alloys, with annual wear volume controlled below 0.1 mm^3, avoiding tissue inflammation caused by metal wear ions. Meanwhile, the micro-nano rough structure of the coating promotes the adhesion and proliferation of osteoblasts, enhancing the bonding effect between implants and bone tissue. It strictly complies with the ISO 10993 biocompatibility standard, showing no cytotoxicity or immune rejection.

3.3 Electronics and Optical Devices

Nanocrystalline diamond coatings have excellent optical transparency, ultra-high thermal conductivity, good dielectric properties and chemical stability, and their applications in the field of electronic and optical devices continue to expand. In the field of optical devices, ultra-thin nanocrystalline diamond films with a thickness of 50-100nm have a transmittance of more than 80% in the 200-800nm visible light band, and have excellent wear resistance, anti-fogging and self-cleaning properties. They can be used as transparent protective coatings for optical components such as mobile phone screens, camera lenses and laser windows. While ensuring optical performance, they significantly improve the scratch resistance and environmental adaptability of components.

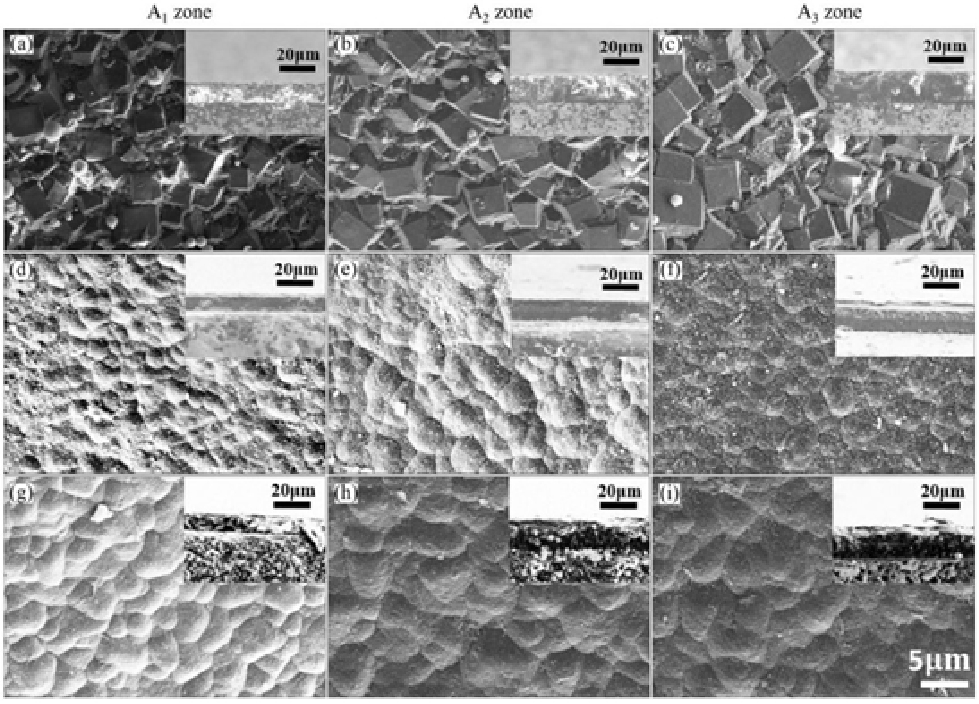
In the field of electronic devices, the wide band gap, high carrier mobility and ultra-high thermal conductivity of nanocrystalline diamond make it a core material for next-generation power semiconductor devices and high-frequency devices. The nanocrystalline diamond coating deposited on the silicon-based surface can significantly improve the heat dissipation capability of chips and solve the thermal management bottleneck of high-power devices. At the same time, nanocrystalline diamond coatings can be used as protective and functional layers for micro-sensors and MEMS devices, improving the sensitivity, stability and service life of devices to meet the application requirements of high-precision and high-reliability electronic devices.

3.4 Core Components Under Special Working Conditions

Nanocrystalline diamond coatings possess a stable sp^3 covalent bond structure, strong corrosion resistance, excellent wear resistance and extreme environmental adaptability, making them a core protective material for special working condition components in aerospace, deep-sea engineering, nuclear energy and other fields. For example, the nanocrystalline diamond composite coating drawing die mentioned in "Preparation and Application Research of Diamond Coated Tools[15]" has broad application prospects in related industries.

In the field of wear-resistant components such as drawing dies and mechanical seals, the friction coefficient of nanocrystalline diamond coatings can be as low as 0.05-0.1, and the wear rate is reduced by more than 80% compared with traditional WC-Co coatings. The maintenance cycle of mechanical seals for centrifugal pumps and compressors can be extended by 2-3 times, and the service life of drawing dies can be increased by more than 10 times. At the same time, the forming accuracy and surface quality of wires and tubes are significantly improved.

Figure 13. Surface and cross-sectional morphologies of three diamond films on wire drawing dies: microcrystalline diamond (MCD) film; nanocrystalline diamond (NCD) film; composite diamond film [16]



In extreme environment applications, for the 600-800℃ high-temperature and high-speed service conditions of aero-engine bearing seal rings, nanocrystalline diamond coatings can still maintain more than 80% of room-temperature hardness above 600℃ without oxidative weight loss, effectively resisting adhesive wear and ensuring sealing performance. In the field of deep-sea engineering, the coating can form a dense physical barrier, reducing seawater corrosion rate by more than 90% while inhibiting marine biofouling, ensuring the long-term reliable operation of deep-sea equipment in high-pressure and highly corrosive environments. In the nuclear industry, the sp^3 bonds of diamond coatings exhibit excellent radiation stability, resisting erosion by γ-rays and neutron radiation, avoiding substrate embrittlement, and significantly improving the service life of nuclear reactor components.

4. Challenges and Future Research Directions

4.1 Current Technical Bottlenecks

Uniformity of complex-shaped substrates: Complex structures such as deep holes and threads lead to uneven hot-wire radiation and gas convection, resulting in significant differences in coating thickness and composition. The coating thickness difference in deep holes of cylinder blocks reaches 50%, affecting the service life of components.

Internal stress control: During the deposition of thick films >20 μm, the internal stress caused by thermal expansion mismatch between diamond and the substrate (12 GPa for a 25 μm coating) exceeds the bonding strength, making the coating prone to cracking and spalling, which limits the application of thick films.

Surface roughness: Nanocrystalline diamond coatings have Ra > 30 nm due to grain agglomeration, which cannot meet the ultra-smooth requirement of Ra < 10 nm for optical components, restricting penetration in high-end fields.

Large-area deposition cost: The investment of a single HFCVD equipment exceeds ten million yuan, with a small number of parts processed per batch and low efficiency. The monthly production capacity of coated tools is only 1,000 pieces, limiting industrial promotion.

4.2 Advanced Research and Development Directions

Low-temperature deposition process (<400°C): Aiming at the thermal degradation of polymer substrates such as PEEK, develop plasma-enhanced/catalyst-assisted CVD technology to realize low-temperature diamond growth and expand application scenarios in biomedicine and flexible electronics.

Nanocomposite coatings (ND-SiC, ND-TiN): Modified by SiC and TiN phases, the fracture toughness of the coating is increased from 2 $\text{MPa}\cdot\text{m}^{1/2}$ to 5 $\text{MPa}\cdot\text{m}^{1/2}$, meeting the requirements of aerospace impact resistance and tool chipping resistance.

Quantum sensing applications (NV color center regulation): Precisely regulate NV color centers, develop high-sensitivity magnetic sensors, break through the limits of traditional sensing accuracy, and achieve a magnetic field resolution of 1 $\text{nT}/\text{Hz}^{0.5}$, which can be used for magnetocardiography detection.

Smart coating systems: Embed shape memory alloys/piezoelectric materials, adaptively adjust the coating structure through temperature/pressure response, realize self-repair and dynamic performance optimization, and improve service reliability in extreme environments.

5. Conclusion

As an efficient method for preparing nanocrystalline diamond coatings, Hot Filament CVD Technology can achieve controllable growth of high-performance coatings by optimizing material selection, process parameters and post-treatment technologies. With the improvement of process maturity and the decrease of cost, the application of nanocrystalline diamond coatings in machinery, biomedicine, electronics and other fields will continue to expand.

Future research should focus on solving key challenges such as deposition on complex-shaped substrates, reducing surface roughness, and developing low-temperature processes, while exploring the innovative applications of diamond coatings in frontier fields such as quantum technology and intelligent materials. Interdisciplinary integration will promote the development of nanocrystalline diamond coating technology towards higher performance and wider applications.

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Conflict of Interests

The authors declare that there is no conflict of interest regarding the publication of this paper.

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