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REVIEW

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Oxidation Behaviour of TiC/TiN Coatings Deposited by Chemical Vapor Deposition: Mechanisms, Structures, and Properties

Osamah Ihsan Ali[1],* and István Gábor Gyurika[1]

[1]*Research Centre for Engineering Sciences, University of Pannonia, Hungary*

Abstract: This review explores the oxidation behavior of titanium carbide (TiC) and titanium nitride (TiN) coatings deposited by chemical vapor deposition, with a focus on their industrial applications as wear-resistant coatings for cutting tools. It examines effect of process parameters—such as temperature, pressure, precursor composition, and substrate preparation—on formation and performance of TiC/TiN coatings. The review evaluates the microstructural characteristics of these coatings, including crystal structure, using X-ray diffraction analysis. Also, the review covers the mechanical properties, such as hardness, wear resistance, and adhesion strength. Additionally, the review covers the thermal stability of TiC/TiN coatings, emphasizing their ability to maintain structural integrity at high temperatures, which is essential for the performance of cutting tools and other industrial components. A major focus is the oxidation behavior of TiC/TiN coatings, including the impact of coating composition, deposition methods, and environmental conditions. The review details oxidation kinetics and mechanisms, revealing various stages of oxidation at different temperatures. It also examines oxide scale morphology and its effect on coating properties. Finally, this review reveals the importance of alloying elements, like silicon, in improving oxidation resistance. Composite coatings such as TiSiN and TiSiCN are shown to offer better high-temperature stability compared to traditional TiN coatings. The effects of coating thickness and the benefits of multilayer coatings for enhanced oxidation resistance are also discussed.

Keywords: chemical vapor deposition, oxidation behavior, surface morphology, TiN/TiC coatings

1. Introduction

The application of chemical vapor deposition (CVD) in the deposition of TiC/TiN coatings has gained significant importance in industrial fields, especially for producing wear-resistant coatings for cutting tools. These coatings are frequently applied to cemented carbide cutting tools to enhance their resistance to wear and extend tool life service. CVD enables the deposition of coatings such as TiC/TiN onto substrates for improving their mechanical and thermal properties [1].

Various factors including temperature, pressure, precursor composition, and substrate preparation have a substantial impact on the formation and quality of these coatings. For instance, the nucleation and initial growth on the layers can significantly influence the phase content of deposited TiC/TiN layers. Moreover, surface conditions and process parameters can effect on the texture of TiC/TiN layers [2].

The crystallographic characteristics of TiC/TiN coatings are also critical in evaluating their performance. The crystal structure and X-ray diffraction (XRD) analysis play an important role in determining the properties of these coatings [3]. Additionally, the mechanical properties, adhesion strength to the substrate, and thermal stability are crucial considerations when assessing the effectiveness of TiC/TiN coatings for industrial applications, of particular interest is comprehending the oxidation behavior of TiC/TiN coatings [4]. Factors that affect oxidation behavior, kinetics and mechanisms of oxidation processes, analysis of oxide scale morphology, and the effects of oxidation on coating properties all require further survey to fully comprehend and optimize the performance of these coatings [5].

This paper aims to more understanding the oxidation behavior of TiC/TiN coatings generated by CVD and provides valuable insights into the principles and mechanisms that govern these processes. Sympathetic these factors are essential for optimizing coating formation, crystallographic characteristics, mechanical properties, thermal stability, and oxidation resistant of TiC/TiN coating.

2. Methodology

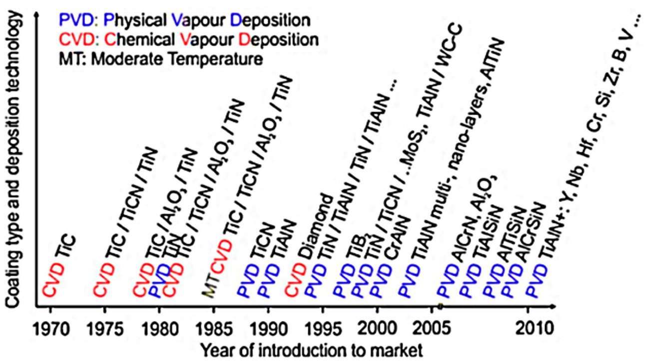
This review article utilized three reputable databases, namely Scopus, ScienceDirect, and Google Scholar, to gather relevant literature. The search was conducted using specific keywords such as “Chemical Vapor Deposition” or “CVD”, “Titanium Carbide” or “TiC”, and “Titanium Nitride” or “TiN”. A total of 68 sources were included in the study, spanning the years 1994 to 2024. The vast majority of these sources, over 90%, consisted of research articles, with approximately 7% being books and less than 3% comprising conference papers. Additionally, recent articles focusing on CVD and TiC/TiN were incorporated to provide a more comprehensive perspective.

*Corresponding author: Osamah Ihsan Ali, Research Centre for Engineering Sciences, University of Pannonia, Hungary. Email: osamah.ihsan.ali@phd.uni-pannon.hu

3. Significance of Surface Coatings in Industrial Applications

The application of surface coatings is of extreme importance in industrial applications, particularly in protection materials against harsh environments. TiC/TiN coatings, for example, offer a wide range of properties, including high hardness, exceptional wear and corrosion resistance, and improved thermal stability [6]. These coatings are extensively utilized in demanding industries such as petrochemicals, mechanics, and cutting tools due to their ability to effectively protect metals or composite substrates [7]. Various surface engineering techniques, such as CVD, physical vapor deposition (PVD), ion beam-assisted deposition, and reactive plasma spraying (RPS), have been employed to develop hard coatings. PVD involves the vaporization of a solid material in a vacuum environment, which is then deposited as a thin film on a surface. RPS, by contrast, uses a plasma jet to heat or melt a material to a semi-molten state, while a reactive gas is added to form a compound coating. Figure 1 [8] provides additional details on these techniques and illustrates the evolution of coating materials used for cutting tools. CVD stands out among these methods due to its high deposition rate, straightforward process and operational conditions, and strong bonding strength resulting from in situ synthesis [9].

Figure 1
Evolution of coating materials for cutting tools



The advancement of designed hard coatings led to improved mechanical properties like hardness and toughness required for cutting tools [10]. Hard coatings like TiC/TiN coatings have gained practical significance due to their excellent wear resistance resulting from the unique combination of high density, high hardness, and moderate levels of fracture toughness [11]. To further enhance the properties of cutting tools, thin layers of wear-resistant materials are deposited onto carbide cutting tool surfaces using CVD technique [12]. Overall, surface coatings play a substantial role in improving the performance and longevity of materials used in industrial applications by providing protection against wear, corrosion, and harsh operating condition [13].

4. Principles and Mechanisms of CVD

4.1. Definition and background of CVD

The method of CVD is widely utilized for applying surface coatings in industrial settings. Coatings comprising tough materials such as TiN, TiC, and TiC/TiN are frequently deposited using CVD, particularly when high coating thickness and thermal stability are necessary. The CVD process involves the use of precursors like CH_3CN to prevent decarburization and the formation of brittle phases. Moreover, dopants are employed to influence the structure and mechanical properties of the coatings. The deposition parameters, including temperature, pressure, gas composition, flow rate, and substrate preparation, have a significant impact on the formation and quality of TiC/TiN coatings [14].

The crystallographic characteristics of TiC/TiN coatings play a vital role in determining their properties. XRD analysis is commonly employed for crystallographic characterization to comprehend the structure and phase composition of the coatings. Additionally, the mechanical properties such as hardness, wear resistance, and adhesion strength of the coatings are crucial factors for their performance in industrial applications [15].

For coatings used in high-temperature applications, thermal stability and resistance to high temperatures are essential properties. It is imperative for TiC/TiN coatings to demonstrate good thermal stability to endure harsh operating conditions [16].

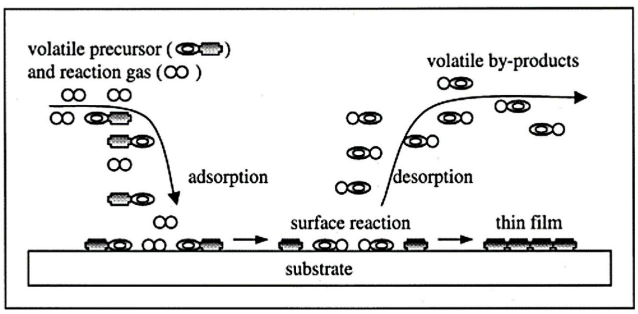
Another critical aspect to consider is the oxidation behavior of TiC/TiN coatings for their reliable performance. Factors influencing oxidation behavior, kinetics, mechanisms of oxidation process, analysis of oxide scale morphology, and effects of oxidation on coating properties all contribute to a comprehensive understanding of how these coatings behave in oxidizing environments. In summary, gaining an understanding of the principles and mechanisms involved in CVD, as well as its impact on the formation and properties of TiC/TiN coatings, is crucial for their successful application in industrial settings [17].

4.2. Process steps involved in CVD

The chemical processes involved in CVD are intricate and multifaceted. Typically, a combination of gas phase and surface reactions occurs during CVD. Volatile precursors are transported to the reaction zone via carrier gas and/or diffusion. These precursors can undergo two types of reactions: homogeneous reactions, where they react with other gases or decompose in the gas phase, and heterogeneous reactions, where they adsorb onto the substrate surface and react or decompose (Figure 2 [2]). Both types of reactions culminate in the formation of a solid thin film on the substrate, along with the generation of gaseous by-products. It is this chemical mechanism that sets CVD apart from physical deposition techniques like evaporation, sputtering, and molecular beam epitaxy [18].

Also, CVD is predominant technique for coating cemented carbide cutting tools, especially when specific properties like

Figure 2
Simplified illustration of chemical vapor deposition (CVD) process



thermal resistance and hot hardness are required along with high coating thickness [19]. The predominant components of CVD coatings include TiN, TiC, and TiC_xN_y coatings [20]. The medium-temperature titanium carbonitride (MT-TiCN) process is employed to prevent decarburization and the formation of brittle phases, enabling deposition at temperatures below 950°C [21]. In addition to modifying deposition parameters, dopants are utilized to influence the structure and mechanical properties of the coatings. Deposition parameters encompass temperature, pressure, gas composition, flow rate, and substrate preparation [22]. These elements significantly impact the formation and quality of TiC/TiN coatings. The kinetics of coating formation are affected by temperature and pressure, while gas composition and flow rate influence properties such as hardness and wear resistance [23]. Substrate preparation also plays a critical role in determining the adhesion strength of the coating to the substrate. Overall, comprehending and managing the process steps involved in CVD is imperative for achieving high-quality TiC/TiN coatings with desirable mechanical and thermal properties [24]. So, optimization of factors such as temperature, pressure, gas composition, flow rate, and substrate preparation is necessary to ensure the desired characteristics of the coatings [25].

4.3. Factors influencing CVD process

The CVD process for titanium carbide (TiC) and titanium nitride (TiN) coatings is a complex interplay of various factors that significantly influence the outcome of the deposition. So, several parameters influence the CVD process, impacting the quality, thickness, and characteristics of the deposited coatings. These factors [26, 27]:

1) **Temperature:**
   - High temperatures are often required to initiate chemical reactions and ensure proper deposition.
   - Elevated temperatures can influence the crystallinity and phase composition of the deposited material.
2) **Pressure:**
   - Pressure affects the reaction rates and helps control the density and adhesion of the deposited coating.
   - Optimizing pressure conditions is crucial for achieving uniform coatings.
3) **Precursor Gases:**
   - The choice of precursor gases significantly influences the composition and properties of the deposited material.
   - Gases may include metalorganic compounds, metal halides, or other volatile compounds.
4) **Substrate Material:**
   - The substrate's composition can impact adhesion and the growth of the deposited film.
   - Compatibility between the substrate and the deposited material is crucial for coating quality.
5) **Gas Flow Rates:**
   - Proper control of gas flow rates is essential for maintaining uniformity and controlling the thickness of the coating.
6) **Reaction Time:**
   - The duration of exposure to precursor gases influences the thickness and structure of the deposited coating.
   - Longer reaction times can lead to thicker coatings but may also affect other properties.
7) **Catalysts and Additives:**
   - The addition of catalysts or certain additives can enhance the deposition process or modify the properties of the coating.
8) **Carrier Gas:**
   - The choice of carrier gas influences the transport of precursor gases to the substrate and affects the overall deposition rate.
9) **Substrate Temperature:**
   - Controlling the temperature of the substrate is crucial for achieving the desired adhesion and crystallinity of the coating.
10) **CVD Chamber Design:**
   - The design of the CVD chamber influences factors such as gas distribution, heat transfer, and overall deposition uniformity. Optimizing these factors in the CVD process is essential for tailoring coatings with specific properties, ensuring uniformity, and meeting the requirements of various applications.

5. Impact of Process Parameters on TiC/TiN Coating Formation

5.1. Temperature and pressure effects on coating formation

The temperature and pressure settings have a significant impact on TiC/TiN coatings formation. Many studies have shown that these parameters can influence crystallite size, phase composition, and mechanical properties of the coatings [28]. For instance, in a study focused on TiCN coatings deposited on plasma nitrided H13 steel using the PACVD method, it was found that higher deposition temperatures led to better adhesion of the coating to the substrate, along with an increase in crystallite size. This suggests that higher temperatures can promote improved adhesion and enhanced structural characteristics [29].

Similarly, in another study where TiN coatings were prepared by microwave plasma CVD (MPCVD), it was observed that increasing temperature resulted in changes to the phase structure of the coating, with an increase in TiN content leading to improvements in mechanical properties such as hardness and wear resistance [30]. These findings emphasize the importance of carefully controlling temperature parameters during CVD to achieve desired coating characteristics [31].

Furthermore, research on SiN_x/TiN/SiN_x coatings demonstrated that high vacuum and elevated temperatures are necessary for deposition, emphasizing the crucial role of high temperatures in achieving specific coating thicknesses and structural characteristics required for oxidation resistance [32], these studies highlight the critical roles of temperature and pressure in determining the quality and properties of TiC/TiN coatings deposited by CVD. By considering these effects, it is possible to optimize process parameters to enhance coating characteristics according to specific industrial requirements [33].

5.2. Gas composition and flow rate influence on coating properties

Gas composition and flow rate are crucial in influencing the characteristics of TiC/TiN coatings deposited via CVD. The content of carbon in TiCN coatings enhances hardness, thermal stability, and resistance to wear and corrosion, so it is valuable for protecting surfaces in demanding industrial conditions [34]. Research into super hard "Ti-B-N" coatings using plasma CVD has shown promising results, producing coatings across a wide range of phases [35]. Modifying surfaces with CVD coatings like TiAlN and TiSiCN has potential for improving cutting performance due to their exceptional properties. Multi-layer coatings have been found to significantly improve cutting tool efficiency and lifespan compared to single-component coatings. Extensive research on the impact of B content on the microstructure, phase composition, and mechanical properties of CVD Ti(B,N) coatings has been conducted, showing that increasing B content can lead to superior mechanical properties up to a certain threshold [36]. Understanding the parameters of gas composition and flow rate during the CVD process is essential for improving coating properties to specific industrial applications.

5.3. Effects of moisture and acidic environments on TiC/TiN coatings

The effects of moisture and acidic gases on CVD hard coatings have been extensively studied due to their significant impact on coating performance and longevity. Moisture can accelerate the oxidation process of TiC/TiN coatings, leading to the formation of titanium oxynitride and ultimately rutile titanium dioxide (TiO_2), which can compromise the coating's protective properties [37]. Acidic gases, such as HCl and SO_2, have been shown to exacerbate corrosion in TiN coatings, particularly at elevated temperatures, by promoting the formation of volatile metal chlorides and sulfates [38]. However, the incorporation of elements like silicon in TiSiN coatings has demonstrated improved resistance to both moisture and acidic attack, attributed to the formation of a protective silicon oxide layer [39].

5.4. Substrate preparation and its impact on coating quality

Preparation of the substrate plays important role in the CVD process for creating TiC/TiN coatings [40]. When it comes to providing oxidation protection coating for this coating, it has been discovered that suspending the substrates results in a more even coating thickness by ensuring well-distributed gas flow around them. Previous research has indicated that carbon fibers were utilized to suspend the substrates, underscoring the importance of proper substrate positioning. Additionally, a study on the oxidation behavior and tribological properties of TiN coating emphasized the development of TiCN coatings through various methods such as CVD, magnetron sputtering, and large area filtered arc deposition. The presence of different percentages of covalent, metal, and ionic bonds makes TiC/TiN coatings highly promising for use in cutting and punching tools due to their exceptional wear resistance and chemical stability in corrosive environments [41].

Moreover, it has been noted that the high thermal expansion coefficients of different layers (such as cemented carbide substrate with 4.5–6.5 × 10^6 K^{-1} and top layer with 8.3 × 10^6 K^{-1}) can lead to tensile residual stress and the formation of a crack network in the coating during CVD. Post-deposition treatments like dry- or wet-blasting are employed to overcome these limitations. In terms of enhancing adhesion, there have been suggestions for using functionally graded TiC/TiN coatings through CVD [33].

6. Crystallographic Characteristics of TiC/TiN Coatings

6.1. Crystal structure of TiC/TiN coatings

The mechanical and thermal properties of TiC/TiN coatings are heavily influenced by their crystal structure. These coatings have a nanocomposite structure, with nanocrystalline TiN grains embedded in an amorphous TiN_xC_y phases [42]. This unique microstructure contributes to the coatings' exceptionally high hardness values, which can exceed 40 GPa. Additionally, the presence of the amorphous phase enhances the thermal stability of the coatings, preventing grain coarsening and improving oxidation resistance up to around 1300°C [43].

XRD analysis has been used to study the crystallographic characteristics of TiC/TiN coatings, providing detailed information about their phase composition and residual stress gradients. Scanning electron microscopy has also offered insights into the oxidation behavior of these coatings, revealing a clear oxidation front and the presence of rutile TiO_2 phases within the oxidized zone. This coexistence zone is linked to a reduction in compressive residual stress and the formation of an overlying stratified TiO_2 layer [44].

These findings highlight the intricate connection between the crystal structure of TiC/TiN coatings and their mechanical properties, oxidation behavior, and thermal stability. Understanding these crystallographic characteristics is essential for optimizing the performance of these coatings in industrial applications, particularly in cutting tools where high wear resistance and oxidation stability are critical [45].

6.2. XRD analysis for crystallographic characterization

XRD analysis stands as a pivotal method for scrutinizing the crystallographic characteristics of TiC/TiN coatings. Through XRD analysis, a thorough understanding of the phase constituents and alterations in microstructure within the coating samples post-oxidation at temperatures spanning from 600°C to 950°C was attained. The outcomes showcased a notable enhancement in the oxidation resistance of WC-Co subsequent to the growth of multilayer coatings, unveiling rutile TiO_2 as the primary oxidation product. Moreover, it was noted that the oxidation kinetics of multilayer coating samples adhered to linear and diffusion-controlled parabolic laws under diverse oxidation conditions. Additionally, the investigation underscored the pivotal role of the outermost TiC/TiN layers in augmenting the oxidation resistance of the substrate, with increased thickness correlating with further enhancement [24].

Furthermore, XRD was employed to assess nanocrystalline TiB_2 coatings fabricated through CVD and their surface oxidation behavior. Innovative cross-sectional nano-analytical techniques were employed to explore the prolonged impacts of surface oxidation on CVD bi-layer coatings, comprising a nanocrystalline TiB_2 protective top layer and a TiN diffusion-barrier bottom layer. XRD analysis provided intricate insights into cross-sectional phase composition gradients in both as-deposited and oxidized states, furnishing valuable information on the ramifications of extended surface oxidation on coating disintegration, microstructure, and mechanical properties. In summary, XRD analysis has emerged as an invaluable tool for characterizing crystallographic properties and comprehending the impact of surface oxidation on TiC/TiN coatings [46, 47].

7. Mechanical Properties of TiC/TiN Coatings

7.1. Hardness and wear resistance

The mechanical properties of TiC/TiN coatings, particularly hardness and wear resistance, are crucial factors influencing their performance in various industrial applications [48]. Hardness, often characterized by techniques such as microhardness testing, represents the coating's ability to resist deformation and penetration [49]. TiC/TiN coatings, known for their inherent hardness, contribute to improved wear resistance, making them ideal for applications where surfaces are subjected to abrasive forces. Wear resistance, a key aspect of the mechanical behavior of these coatings determines their durability under sliding, rolling, or abrasive conditions. The synergy between the hardness of TiC and the toughness of TiN enhances the overall wear resistance of the coating, resulting in prolonged tool life and reduced material loss in high-stress environments. Investigations into the relationship between processing parameters, coating microstructure, and mechanical properties play a pivotal role in optimizing TiC/TiN coatings for specific applications. Understanding the intricate interplay between hardness and wear resistance is fundamental to enhancing the overall performance and longevity of these coatings in demanding industrial settings [50].

7.2. Adhesion strength to the substrate

The adhesion strength of TiC/TiN coatings to the substrate is necessary for their performance in industrial applications [51]. A study on the effects of plasma nitriding revealed a significant increase in the adhesion of TiC/TiN coatings to the hot-worked steel H13 substrate [52]. Moreover, the addition of a TiN functional intermediate layer further enhanced adhesion and mechanical properties. Coatings deposited at 475°C exhibited higher adhesion compared to those deposited at 450 and 500°C, resulting in reduced radial and peripheral cracks within the coating and ultimately increasing wear resistance by 78% compared to other coating [44]. Functionally graded nanostructured TiCN coatings have also shown promise in improving wear resistance and tool life, as they gradually increase carbon and nitrogen content from the substrate to the surface, leading to improved adhesion. Additionally, multi-layered TiN/TiCN hard coatings grown by CVD have been optimized with the addition of gaseous precursor to the feed gas, resulting in increased coating hardness and improved adhesion strength. So, it is obvious from these studies that factors such as deposition temperature, and composition adjustments can significantly impact the adhesion strength of TiC/TiN coatings to substrates, ultimately influencing their mechanical properties and wear resistance [53].

8. Thermal Stability of TiC/TiN Coatings

The ability of coatings to protect substrates from environmental damage, oxidative wear, and corrosion at elevated temperatures is of utmost importance. Several studies have highlighted the role of protective CVD coatings in enhancing the performance and longevity of cutting tools, particularly cemented carbides used in machining and drilling. Modified multilayer coatings have proven to significantly improve cutting tool efficiency and durability. Additionally, the unique properties of CVD TiAlN and CVD TiSiCN coatings have led to their widespread adoption as replacements for previous-generation hard coatings and as promising options for wear-resistant coatings due to their ultra-high hardness, wear, and oxidation resistance [54]. It is important to highlight that the CVD-coated cutting tools were operated within a temperature range of 500 to 900 °C [55].

In addition to protecting against wear and oxidation at high temperatures, thermal stability is also crucial for enhancing the corrosion resistance of hard coatings in corrosive environments. The ability of the coating to withstand high temperatures without degradation or compositional changes is essential for preserving the integrity of cutting tools and other industrial components [54].

Furthermore, extensive research has focused on understanding the impact of process parameters on coating formation, particularly with regard to temperature and pressure effects. This understanding is vital for optimizing the thermal stability and resistance to high temperatures of coatings by influencing their crystallographic characteristics and mechanical properties. Additionally, careful analysis of the factors influencing oxidation behavior is necessary to ensure that the coating remains effective under extreme conditions [3, 56].

9. Oxidation Behavior of TiC/TiN Coatings

9.1. Factors influencing oxidation behavior

The oxidation behavior of Ti (C, N) coatings is influenced by factors such as coating composition, deposition methods, and environmental conditions. Research has shown that adding silicon to TiN coatings can greatly improve their resistance to oxidation, with TiSiN and TiSiCN coatings exhibiting greater stability at high temperatures compared to traditional TiN coatings [12, 57]. The addition of silicon contributes to the formation of a diffusion barrier that inhibits cobalt diffusion from the substrate, improving the adherence and stability of the coatings. Low-pressure CVD processes have been developed for the successful deposition of nanocomposite coatings with enhanced oxidation and wear resistance. The microstructure, composition, and properties of these coatings have been extensively studied, showing that nanocomposite structures comprised of crystalline phases and amorphous matrices contribute to increased hardness and stability at high temperatures. Environmental factors such as temperature and gas composition have also been found to significantly influence coating formation and stability. Understanding these influences is crucial for optimizing the performance of surface coatings in industrial applications where high-temperature oxidation can be a limiting factor. Overall, composition, deposition methods, and environmental conditions play a crucial role in determining the oxidation behavior of TiC/TiN coatings. Research on nanocomposite structures with added silicon has shown promising results in enhancing stability at high temperatures, and further exploration into these factors will contribute to advancing the development of surface coatings with improved oxidation resistance for industrial applications [58].

9.2. Kinetics and mechanisms of oxidation process

The oxidation behavior of TiC/TiN CVD-coated cutting tools significantly impacts their performance and longevity. The oxidation process involves the reaction between the coating material and atmospheric oxygen, leading to the formation of surface oxides. This mechanism can be illustrated schematically as shown in Figure 3 [59], where the initial reaction of TiC or TiN

Figure 3
The diagram depicts the infiltration of oxygen through the oxidized CVD layers

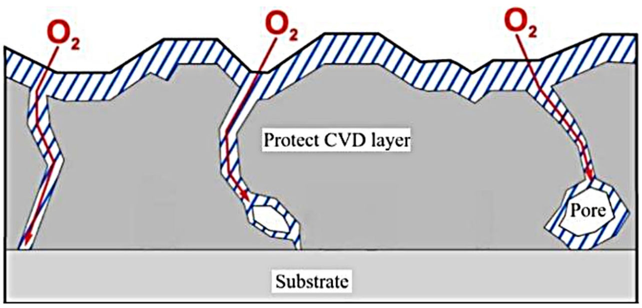
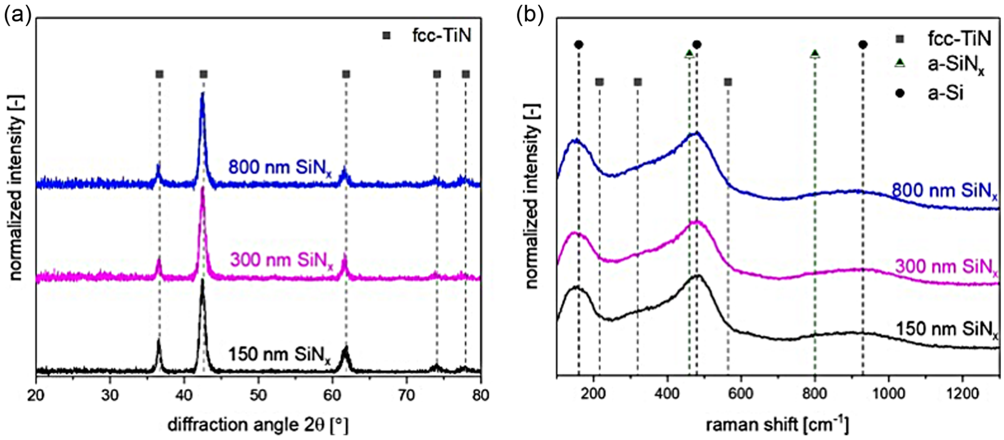


Figure 4
X-ray diffractograms and Raman spectra of SiN_x/TiN/SiN_x coatings with varied SiN_x layer thickness in the as-deposited state



with oxygen forms TiO_2 or titanium nitride oxides, respectively. As the temperature increases, particularly during machining operations, the rate of oxidation accelerates. The oxidation kinetics can be described by the parabolic rate law, which is expressed as $K_{ox} = k_p t$, where K_{ox} is the oxide thickness, kp is the parabolic rate constant, and t is time. This rate constant kp is temperature-dependent, increasing exponentially with temperature according to the Arrhenius equation $k_p = Ae^{-Q/RT}$, where A is the pre-exponential factor, Q is the activation energy, R is the gas constant, and T is the absolute temperature [60, 61]. While these hard coatings exhibit notable resistance to mechanical loads, their effectiveness may diminish at elevated temperatures and in aggressive environments. In such scenarios, the chemical inertness of the coatings becomes more critical than their hardness. High temperatures exacerbate the oxidation rate, resulting in the formation of a thick oxide layer that compromises the coating's mechanical properties and overall performance [62].

The oxidation process of these coatings is influenced by several features, including the types and thickness of these coatings. Study has demonstrated that SiN_x acts as a shield for TiN against oxidation at high temperatures, and thicker layers of SiN_x have proven to be more effective in preventing oxidation. Once a critical temperature is reached, both the TiN and the amorphous SiN_x layers become porous, allowing oxygen diffusion and resulting in the oxidation of TiN to r-TiO_2. The grain coarsening of r-TiO_2 has been observed, eventually breaking through the top layer of SiN_x. The incorporation of some elements like silicon has been shown to enhance the resistance to oxidation of Ti-based coatings. It has been proven that increasing the oxygen content in TiON coatings can lead to improved resistance to corrosion and increased hardness. Additionally, the formation and shedding of loose oxide layers at high temperatures can speed up the processes of oxidation and corrosion in TiN coatings [63].

Moreover, investigation has indicated that composite coatings with TiNSi exhibit different stages of oxidation behavior. At temperatures above 950°C, the oxidation layers grow parabolically over time due to a diffusion-limited process, while at temperatures below 950°C, rapid oxidation occurs initially before reaching a threshold corresponding to complete passivation.

Understanding the kinetics and mechanisms involved in the oxidation behavior of TiC/TiN coatings is essential for optimizing their performance in various industrial applications. By considering aspects such as alloying elements and the effects of temperature on coating formation, it is possible to enhance both the resistance to oxidation and overall properties of these coatings [64, 65].

9.3. Oxide scale morphology analysis

TiC/TiN coatings' oxidation characteristics have directly impacted their performance and properties. Understanding the structure of the oxide layer formed during oxidation is essential for optimizing coating properties [66]. Research studies have explored structural changes caused by oxidation offering valuable insights into these coatings' behavior. One study focused on sputter-deposited model SiN_x/TiN/SiN_x coatings, using XRD detailed in Figure 4 [67], SEM investigations, and Raman spectroscopy to analyze the structure of both as-deposited and oxidized coatings (Figure 5, [28]). The results

Figure 5
Secondary electron scanning electron microscope (SEM) cross-sectional images depict a three-layer architecture consisting of SiN x /TiN/SiN x with different thicknesses of the SiNx layer: (a) 150 nm, (b) 300 nm, and (c) 800 nm

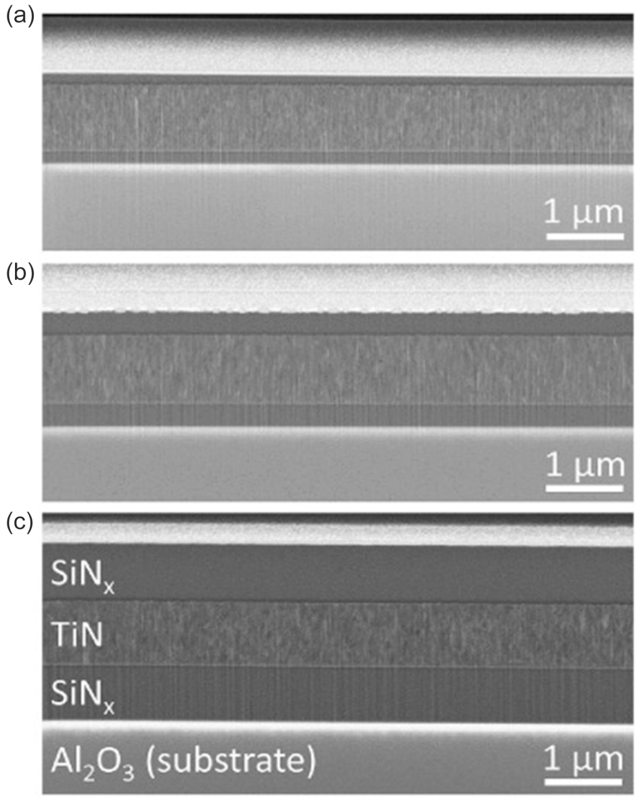
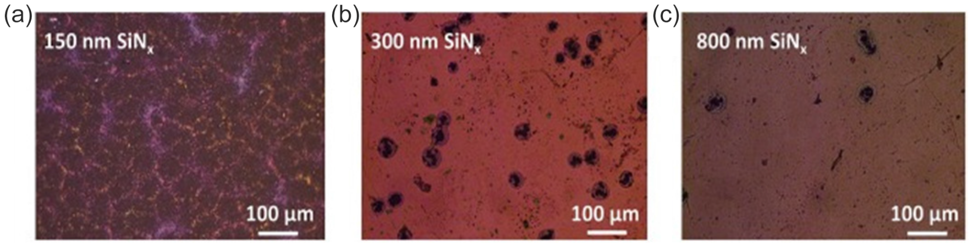


Figure 6
Light optical microscopy was utilized to capture images depicting the surface morphology of SiNx /TiN/SiNx coatings subjected to oxidation at 1200°C, with varying thicknesses of the SiNx layer: (a) 150 nm, (b) 300 nm, and (c) 800 nm

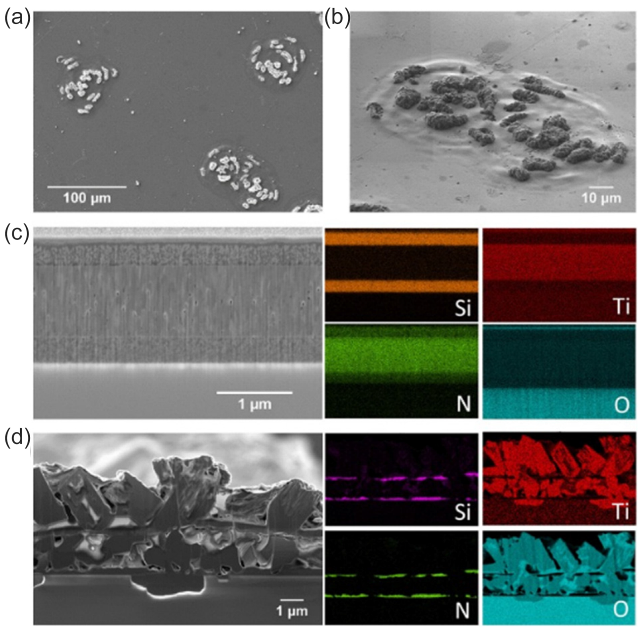


showed the formation of r-TiO_2 grains and increased porosity in the oxidized coating, attributed to nitrogen released during oxidation accumulating at the interface between the coating and oxide.

Another study investigated a CVD-$(ZrC/SiC)_3$ alternate coating on C/C composites under oxyacetylene with varying heat fluxes, showing excellent adhesion and forming dense and stable ZrO_2 layers covering C/C composites after rapid consumption of SiC layers (Figure 6, [67]). In a separate study, titanium silicide Ti_5Si_3 was introduced into TiN coatings using in situ CVD to enhance their resistance to oxidation. The study identified two distinct oxidation regimes for TiNSi, with a diffusion-limited process occurring above 950°C and rapid initial oxidation below 950°C, followed by complete passivation.

These investigations offer valuable insights into the structure of oxide scales formed during the oxidation of TiC/TiN coatings, providing a better understanding of structural changes and oxidation behavior of these coatings.

Figure 7
SEM surface micrographs were captured of the SiNx/TiN/SiNx coating, featuring a 300 nm SiNx layer, after exposure to 1200°C. The images were taken at both low and high magnification. Additionally, cross-sectional images and corresponding EDX maps were obtained, highlighting both non-oxidized and oxidized regions



9.4. Effects of oxidation on coating properties

A study on TiSiN coatings found they resist oxidation up to 830°C, forming TiO_2 at higher temperatures and becoming porous. Another investigation on $SiN_x/TiN/SiN_x$ coatings showed that thicker SiN_x layers delayed oxidation onset, and amorphous SiN_x was more resistant than TiN, as shown in Figure 7 [67]. The study also found different layers impacted overall oxidation resistance [68]. A study on CVD TiB_2 hard coatings used advanced techniques to analyze oxidation behavior, revealing gradients in phases on coating thickness and how surface oxidation can cause disintegration. These studies highlight the complex relationship between process parameters, properties, and layers within a coating system on its resistance to oxidation, crucial for optimizing them for industrial use [12].

10. Conclusion

This review has studied the oxidation behavior of TiC/TiN coatings deposited by CVD, highlighting their significance in industrial applications, particularly as wear-resistant coatings for cutting tools. The study revealed that, CVD process parameters significantly influence coating formation and properties, with optimization crucial for achieving desired characteristics.

Crystallographic analysis, particularly XRD, has proven invaluable in understanding the composite structure of these coatings, which contributes to their exceptional hardness and thermal stability. The addition of functional intermediate layers and development of functionally graded structured coatings have shown promise in improving adhesion strength and wear resistance.

The oxidation behavior of TiC/TiN coatings is influenced by factors such as coating composition and manufacturing conditions. The addition of alloying elements, particularly silicon, has demonstrated remarkable improvements in oxidation resistance. Analysis of oxide scale morphology has provided crucial insights into structural changes during oxidation, identifying different oxidation regimes at various temperatures.

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Ethical Statement

This study does not contain any studies with human or animal subjects performed by any of the authors.

Conflicts of Interest

The authors declare that they have no conflicts of interest to this work.

Data Availability Statement

Data sharing is not applicable to this article as no new data were created or analyzed in this study.

Author Contribution Statement

Osamah Ihsan Ali: Conceptualization, Methodology, Validation, Formal analysis, Investigation, Data curation, Writing – original draft, Writing – review & editing, Visualization, Supervision, Project administration, Funding acquisition. **István Gábor Gyurika:** Conceptualization, Funding acquisition.

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