ENHANCING MICROCRYSTALLINE-DIAMOND ADHESION ON CEMENTED CARBIDE CUTTING TOOLS USING A NANOCRYSTALLINE-DIAMOND BUFFER LAYER AND HIGH-TEMPERATURE INTERLAYERS

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Abstract

Diamond coatings provide outstanding hardness and wear resistance for cutting tools, however, their application on cemented tungsten carbide (WC–Co) is often limited by insufficient adhesion. This study addresses this challenge by introducing a thin nanocrystalline-diamond (NCD) buffer layer to enhance the adhesion of subsequently deposited microcrystalline-diamond (MCD) coatings. Two types of interlayers routinely employed in industrial applications, TiAlSiN and a SiC-based interlayer, were deposited on commercially available WC-Co inserts using magnetron sputtering.

The NCD buffer, with a thickness of 750 nm, was prepared in a linear-antenna microwave plasma chemical vapour deposition (MWCVD) system at substrate temperature as low as 550 °C. After that, the MCD coatings with a thickness of 11.5 µm were deposited in focused MWCVD at 700 °C on both interlayers (TiAlSiN and SiC-based), with or without an NCD buffer layer. Scanning electron microscopy and Raman spectroscopy (sp^3 diamond peak near 1331-1338 cm^{-1}) confirmed the diamond crystalline quality and film continuity across all investigated conditions. Samples with SiC-based interlayer exhibited significantly less peeling than TiAlSiN under otherwise comparable diamond deposition conditions. Overall, the incorporation of an NCD buffer layer markedly improves adhesion, and interlayer selection is critical: the SiC-based interlayer combined with NCD consistently yields more homogeneous coverage with minimal delamination compared to TiAlSiN. These results provide a robust, industrially relevant pathway towards adherent, uniform MCD coatings on carbide tools.

Keywords: Diamond coatings adhesion, WC–Co cutting tools, nanocrystalline-diamond (NCD) buffer layer, microcrystalline-diamond (MCD) coating, high-temperature interlayers

1. INTRODUCTION

Diamond coatings can dramatically improve the hardness and wear resistance of cutting tools in a range of industries, from aerospace to dental machining. They enable the machining of abrasive composites and hard alloys that would quickly wear out conventional coatings [1, 2]. These benefits stem from the exceptional hardness of diamonds (approximately 9,000 HV, around three times higher than TiAlN) and their superior thermal conductivity. Together, these properties enable diamond-coated tools to maintain sharp edges and keep cutting interfaces cool during high-speed or dry machining. For instance, diamond-coated drills have a significantly longer lifespan when drilling carbon-fibre composites, thereby reducing the need for tool changes and improving hole quality [3, 4]. Consequently, polycrystalline diamond (PCD) inserts and CVD diamond-coated carbide tools are being adopted more widely in the manufacture of abrasive materials [5, 6].

Nevertheless, achieving robust diamond adhesion on cemented tungsten carbide (WC–Co) tools remains a formidable challenge. The cobalt binder in WC–Co facilitates graphitic carbon formation at typical CVD diamond growth temperatures (600–800°C), which poisons diamond nucleation and weakens the interface [7, 8]. Furthermore, the substantial disparity in thermal expansion between diamond and WC–Co results in elevated tensile stresses within the cooling film, which frequently surpass the interface's strength threshold, leading to delamination. The combined effects of these factors result in suboptimal coating adhesion on as-sintered carbide substrates [9, 10]. To address this issue, it is imperative to implement specific measures to either mitigate cobalt or alleviate stress. One approach to consider is the removal or neutralisation of Co at the surface (e.g., by chemical etching) to prevent the formation of graphitic interlayers [11]. Another effective strategy is to introduce intermediate interlayers that act as diffusion barriers and compliant buffers between the WC–Co and diamond. In this study, the latter approach is adopted, involving the deposition of a thin nanocrystalline diamond (NCD) buffer layer at low temperature, followed by the application of a thicker microcrystalline diamond (MCD) coating on inserts with either a TiAlSiN or a SiC-based ceramic interlayer. The NCD buffer functions as an adherent seeding layer, thereby promoting diamond nucleation and facilitating covalent bonding to the substrate. Concurrently, the high-temperature-stable interlayers (TiAlSiN or SiC-based) protect the substrate by trapping cobalt (e.g., via Co silicide formation) and better accommodating thermal stress. Earlier studies have suggested the efficacy of such measures. For instance, Lopez et al. achieved significantly improved diamond adhesion on WC–Co by using a nanograined diamond interlayer, and Hodroj et al. reported well-adhered NCD films on WC–Co when employing an optimal nitride diffusion barrier [12, 13]. The objective of this study is to demonstrate a combined approach that yields a uniformly adherent MCD coating on commercial WC–Co cutting inserts [14]. This aim is built on the insights outlined above. In the following, the deposition process is described, and the coating morphology, quality, and adhesion performance for the different interlayer configurations are analysed. The overarching objective is to devise a pragmatic technique for affixing CVD diamond to carbide instruments without the typical adhesion failures, thereby facilitating the utilisation of diamond-coated tools in challenging applications.

2. EXPERIMENTAL

Cemented WC–Co cutting inserts (15 × 15 mm) were utilised as substrates (**Figure 1a**). Before the diamond coating process, each insert was subjected to a hard interlayer coating through the utilisation of industrial PVD magnetron sputtering. This procedure was implemented with the objective of enhancing the high-temperature stability of the inserts and impeding cobalt diffusion. Two interlayer types were applied (from SHM, s.r.o.): The coating under investigation is a TiAlSiN nanocomposite coating, which is a multilayer structure with a hard base layer and a thermally stable nitride top layer. This is similar to commercial TiAlSiN tool coatings. The second layer is an amorphous silicon-carbon-nitrogen ceramic layer, which is denoted "SiC-based". Following a period of sputtering, the coated inserts were subjected to ultrasonic cleaning in acetone and isopropanol and subsequently seeded with nanodiamond to provide nucleation sites. The inserts were immersed in a colloidal suspension of detonation nanodiamond powder with a diameter of approximately 3 nm, and the suspension was ultrasonicated for a period of 40 minutes. This process resulted in the deposition of a high density of diamond nanocrystals on the surface. The diamond deposition process was executed in two stages. Initially, a thin NCD buffer layer was cultivated at a low temperature by means of a linear-antenna microwave plasma chemical vapour deposition reactor. The plasma (2.45 GHz, 2 kW) was sustained in H_2–CH_4–CO_2 gas (200/5/20 sccm) at a pressure of 0.15 mbar and a temperature of 550°C. Following a period of 60 hours of growth under these conditions (corresponding to a deposition rate of ~12–13 nm/h), a conformal NCD film with a thickness of approximately 750 nm had formed. In the subsequent stage of the process, a thicker MCD coating was deposited on top of the NCD (or directly on the interlayer for the purpose of comparison). The deposition was carried out using a focused-microwave plasma chemical vapour deposition system. The second step was conducted at an elevated substrate temperature of approximately 700°C and at a pressure of 60 mbar with a 3 kW microwave output. The gas mixture used comprised 300 sccm H_2 and 18 sccm CH_4 (6% CH_4). It was demonstrated that a 100-hour growth produced a well-faceted MCD film with a thickness of approximately 11.5 µm. Four sample variants were prepared in order to evaluate the effect of the interlayer and buffer: The following combinations were examined: MCD on TiAlSiN, MCD on SiC-based, NCD+MCD on TiAlSiN, and NCD+MCD on SiC-based. The deposited coatings were characterised by means of scanning electron microscopy (SEM) and Raman spectroscopy. The SEM (TESCAN MAIA3 field-emission microscope) was utilised in secondary-electron mode to examine surface morphology and cross-sections (obtained by fracturing inserts) for thickness measurements. Raman spectroscopy (Renishaw inVia, 442 nm He–Cd laser) was utilised to confirm the diamond phase and assess stress. The instrument was calibrated to the natural diamond line at 1332 cm^{-1}.

3. RESULTS AND DISCUSSION

3.1 Diamond Growth Optimization and Buffer Layer Efficacy

Initial direct deposition of MCD on WC–Co (utilising the high-temperature microwave plasma) resulted in substandard adhesion – the diamond film was non-uniform and delaminated in places due to cobalt interference and thermal stresses. These failures provided the motivation for the introduction of the NCD buffer layer, which is grown at a lower temperature (**Figure 1b**). The deposition of approximately 750 nm of NCD at approximately 550°C prior to the MCD process resulted in the formation of a high-density seeding layer, thereby enhancing coating homogeneity to a significant extent. The low-stress NCD film functions as an adherent diamond-on-diamond base, onto which the thicker MCD can grow without immediately delaminating. Consequently, a continuous, well-bonded MCD coating was obtained on the NCD-buffered samples, despite the slow rate of buffer growth. This approach is analogous to that of Lopez et al., who found that a nanograined interlayer promotes covalent bonding at the interface and greatly enhances diamond adhesion [12]. In this instance, the successful management of the low-temperature growth of a uniform NCD layer was a crucial step towards achieving a stress-relieved, adherent MCD film on WC–Co. As illustrated in **Figure 1c**, subsequent deposition of 11.5 μm MCD at approximately 700 °C results in the formation of well-faceted diamond crystallites within the coating. This multilayer strategy has been demonstrated to be effective in the relief of residual stresses and the prevention of delamination, thereby ensuring the formation of a uniform, adherent diamond film.

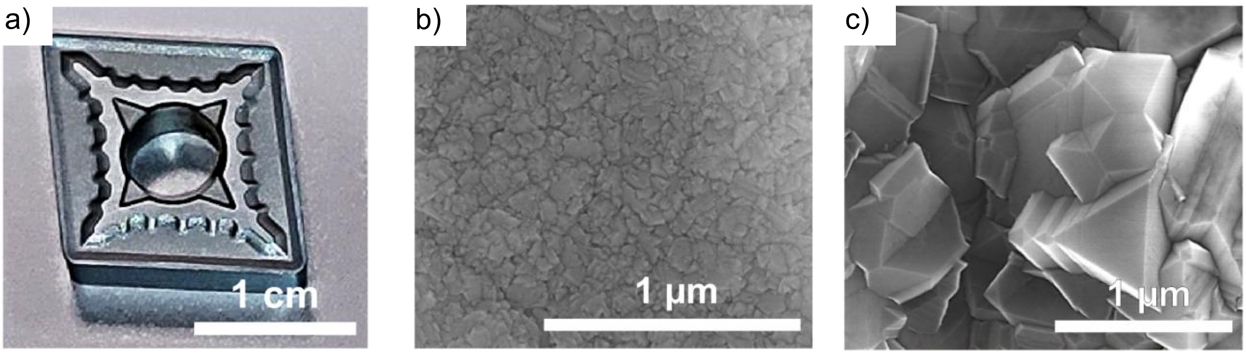

Figure 1 a) WC-Co substrate image, b) SEM top view images of NCD buffer layer, c) SEM top view image of MCD coating layer

3.2 Cobalt Diffusion and Interlayer Effects

A primary function of the PVD interlayers is to address the "cobalt problem" in diamond-coated WC–Co tools. Energy-dispersive X-ray (EDX) analysis of cross-sections revealed marked discrepancies between samples with and without interlayers. As illustrated in **Figure 2a**, an EDX map of a diamond-coated insert devoid of an interlayer reveals a pronounced Co signal that extends from the substrate into the diamond layer, thereby substantiating considerable cobalt diffusion into the coating. This would result in the diffusion of Co, which would catalyse graphitic carbon at the interface, thereby severely undermining adhesion. Conversely, the presence of a SiC-based interlayer, as illustrated in **Figure 2b**, results in the effective obstruction of Co at the substrate–interlayer boundary. This observation is supported by the absence of cobalt detection in the diamond layer situated above. The Si-rich interlayer functions as a diffusion barrier, which has been shown to tie up Co (probably by forming stable Co–Si compounds) and to prevent Co from contaminating the diamond film. The result of this process is a diamond–substrate interface that is uncontaminated by a weak graphitic layer, which would otherwise form in the presence of cobalt. These observations are consistent with previous reports: As Cabral et al. found, the addition of a thin β-SiC interlayer on WC–Co led to a significant enhancement of diamond adhesion, achieved by mitigating Co diffusion [9]. In a similar manner, Wang et al. demonstrated that a composite β-SiC/Co-silicide interlayer enhanced diamond coating adhesion, as evidenced by higher critical loads in scratch tests [14]. The results obtained demonstrate the efficacy of the TiAlSiN and SiC-based interlayers in preventing the interaction between the diamond film and the detrimental Co in the substrate.

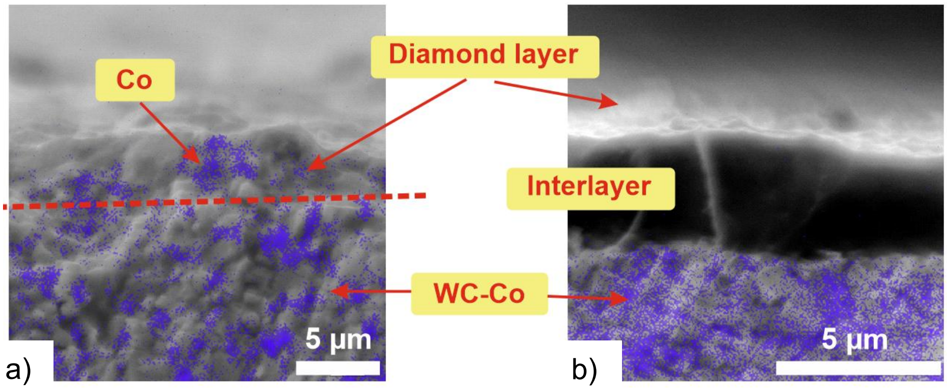

Figure 2 Cross-sectional SEM-EDX elemental maps: a) MCD diamond layer on WC-Co cutting tools without interlayer, exhibiting Co diffusion into the diamond layer, b) MCD diamond layer on WC-Co cutting tools with SiC-based interlayer, effectively blocking Co diffusion into the diamond layer.

3.3 Residual Stress Analysis by Raman Spectroscopy

It is evident that both samples demonstrate a robust diamond sp³ peak (**Figure 3**), its position determined by the interlayer: The MCD on TiAlSiN exhibited a slight downshift, indicative of tensile stress, with a wavenumber of approximately 1331 cm^{-1}. In contrast, the MCD on the SiC-based interlayer demonstrated an upshift, suggesting compressive stress, with a wavenumber of around 1338 cm^{-1}. The absence of any significant D/G bands (at approximately 1350/1580 cm^{-1}) indicates a minimal presence of sp² carbon. Consequently, the shift in the peak indicates the level of stress, rather than the quality of the material. The compressive state of SiC-based materials is associated with the enhanced adhesion that is observed.

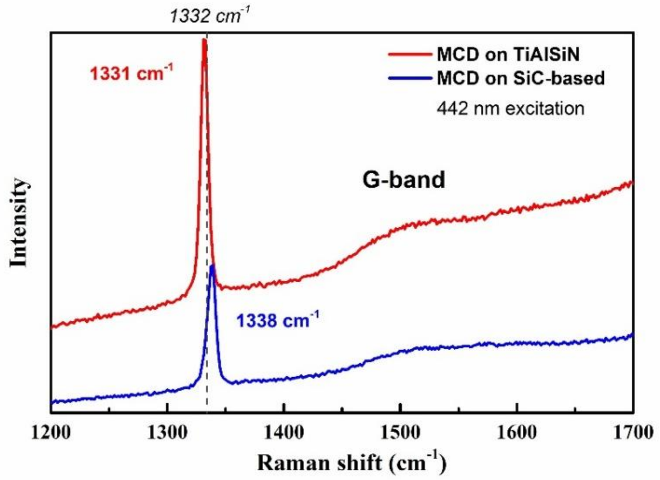

Figure 3 Raman spectroscopy graph MCD diamond coating on TiAlSiN and SiC-based interlayers.

3.4 Adhesion Performance of Diamond Layers

The ultimate benchmark for these coating architectures is the adhesion of the diamond film on cutting inserts. Subsequent to the process of deposition, a thorough inspection of the inserts was conducted for the presence of any film delamination or peeling, with particular attention directed towards the areas adjacent to the cutting edges, where stress levels are known to be elevated. Although a quantitative adhesion test was not conducted, adhesion was qualitatively assessed via SEM inspection for peeling, complemented by Raman confirmation of film continuity and EDX verification of cobalt suppression at the interface. **Figure 4** compares plan-view SEM images of the coated inserts for each case. The difference is striking: when the MCD was deposited directly on TiAlSiN (no NCD buffer, **Figure 4a**), large portions of the diamond film peeled off, particularly along edges and rake faces, exposing the substrate. It is evident that the TiAlSiN in isolation was not sufficient to provide an adequately adherent base, resulting in gross delamination due to thermal stresses and inadequate bonding. The incorporation of the NCD buffer within TiAlSiN (**Figure 4b**) resulted in a marginal enhancement, characterised by slightly more uniform coverage, yet significant peeling remained. Consequently, irrespective of the presence or absence of NCD, the thick MCD film exhibited an inability to maintain adhesion. Conversely, the SiC-based interlayer exhibited significantly superior outcomes. In the case of MCD directly on SiC-based (**Figure 4c**), the diamond coating remained largely intact across the insert; only a small amount of flaking was seen at the outer corners (known stress concentrators). The observation that the majority of the film remained adhered suggests that the SiC ceramic provided a more compatible anchoring interface for diamond than the TiAlSiN did. In conclusion, it was determined that the optimal performance was attained through the integration of the SiC-based interlayer with the NCD buffer (**Figure 4d**). In this configuration, the entire diamond film (11.5 µm) remained firmly attached, even at edges and corners – no visible peeling or cracks were observed. The surface exhibited a uniform distribution of diamond. The enhanced adhesion is attributed to the synergy of the diffusion barrier and buffer layer. The SiC-based interlayer prevents cobalt-induced debonding and offers the possibility of chemical bonding sites (Si–C bonds or silicides) for diamond, while the NCD interlayer provides a compliant diamond-to-diamond interface.

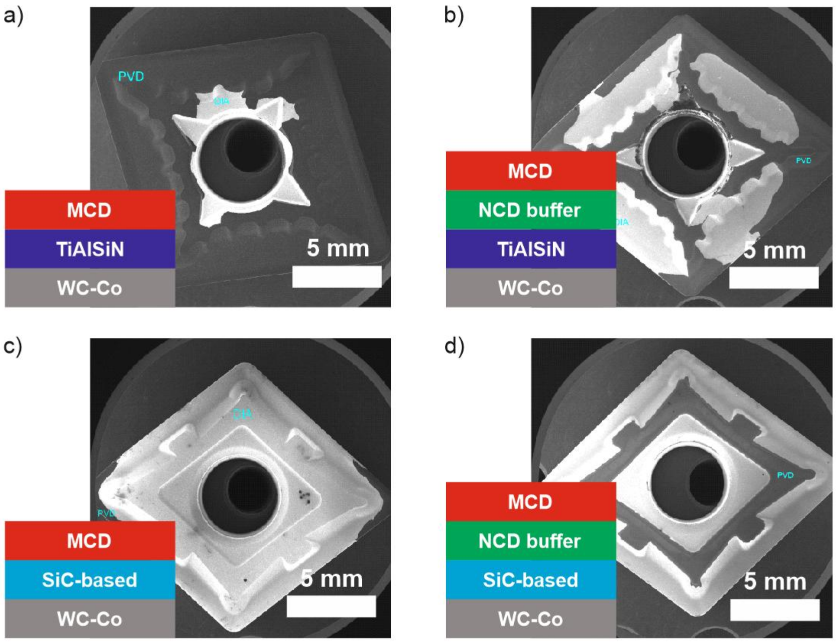

Figure 4 Adhesion outcomes of MCD on WC–Co for different stack designs (top-view SEM), (a) MCD on TiAlSiN: extensive peeling of the diamond film. (b) NCD + MCD on TiAlSiN: delamination persists despite the buffer layer. (c) MCD on SiC-based interlayer: largely intact coating with only minor corner flaking. (d) NCD + MCD on SiC-based interlayer: continuous, well-adhered diamond coating across the insert, including edges and corners.

4. CONCLUSIONS

In summary, this study demonstrates that introducing a thin NCD buffer layer significantly enhances the adhesion of MCD coatings on WC–Co cutting tools, particularly when combined with a high-temperature SiC-based interlayer. Among the interlayer materials tested, the SiC-based layer proved substantially more effective than TiAlSiN in promoting diamond adhesion. Only the SiC + NCD architecture yielded a uniform, crack-free MCD film with no delamination even at cutting edges, whereas the TiAlSiN-based coatings exhibited significant peeling and poor adhesion despite the presence of an NCD buffer. The superior performance of the SiC + NCD combination is attributed to its effectiveness to block cobalt diffusion and better accommodate thermal stresses, thereby firmly anchoring the diamond layer to the substrate. Overall, the synergistic use of a SiC-based interlayer and an NCD buffer provides an industrially viable solution to the longstanding adhesion challenge, enabling the deposition of durable, high-quality diamond coatings on cemented carbide cutting tools.

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DATA AVAILABILITY:

The data that support the findings of this study are openly available at https://doi.org/10.5281/zenodo.17547153

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