Ex Situ Residual Stress Analysis of Chemical Vapor Deposited Diamond Coated Cutting Tools by Synchrotron X-Ray Diffraction in Transmission Geometry

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When machining difficult-to-cut, nonferrous materials, chemical vapor deposited (CVD) diamond–coated cutting tools are applied. The tools’ favorable mechanical property profile is based on the hardness of the coating as well as the adaptability of the substrate. Nevertheless, the reproducibility of machining results and process stability are limited by insufficient coating adhesion. The resulting cutting tool failure is based on coating delamination initiated by crack development. By assessing residual stress as an influence of coating adhesion, an analysis of CVD diamond–coated tools is performed using synchrotron X-ray diffraction in transmission geometry. Investigation of a nanocrystalline and multilayer morphology on cobalt-based tungsten carbide (WC-Co) and a silicon nitride–based ceramic (Si$_3$N$_4$) provides the distribution of the principal in-plane residual stress tensor component $\sigma_{22}$ depending on the coating morphology and substrate material. Contrary to microcrystalline CVD diamond, nanocrystalline layers decrease the compressive residual stress. In addition, the CVD diamond coating deposited on the Si$_3$N$_4$ substrate material tends to induce an overall initial tensile residual stress that leads to increased tool performance compared to WC-Co-based coated tools. Variation of the coating morphology as well as the substrate material offers the possibility to extend the current model for residual stress–dependent tool failure.

1. Introduction

Within the field of cutting tool applications, the use of hard cutting materials such as diamond offers an economical machining of difficult-to-cut workpiece materials. Thereby, machining operations of nonferrous workpieces such as fiber-reinforced plastics (FRPs), aluminum–silicon alloys, metal matrix composites, green ceramics, graphite or cemented carbide are considered as the main field of application for diamond cutting tools. Due to the uniform and reproducible wear protection on a complex tool geometry, chemical vapor deposition is industrially used for thin-film diamond coating of high-performance cutting tools with a defined cutting edge. \[1\] Compared to uncoated cutting tool substrates such as tungsten carbides (WCs), chemical vapor deposited (CVD) diamond coatings ensure an extension of the cutting tool performance regarding tool life as well as wear behavior. Based on a balanced combination of mechanical properties such as hardness, toughness, and wear resistance, CVD diamond–coated WC tools meet the challenging requirements for industrial manufacturing processes within a variety of applications. \[2,3\] However, the toughness-related cutting tool behavior particularly reduces the ability to resist stress peaks during challenging machining operations. As a result, crack initiation and crack propagation induce coating delamination, thus critically damaging the cutting tool until complete exposure and subsequent failure of the cutting edge occur. Therefore, these tools primarily fail because of the insufficient adhesion between the CVD diamond coating and the cutting tool substrate materials within the substrate–coating interface during their application in various machining processes. \[4\] Furthermore, the binder phase within the substrate can cause graphitization during diamond deposition when cobalt-bonded tungsten carbide cutting tool substrates are used. \[5\] In addition, high synthesis temperatures during the deposition process of CVD diamond of $T_d = 500–800$ °C and thermal mismatch of the regarded expansion coefficients between the cutting tool substrate and the growing CVD diamond coating result in a disadvantageous residual stress state. \[6\]

Within the scope of this investigation, the principal in-plane residual stress $\sigma_{22}$ of varying CVD diamond–coated cutting tool...
specifications was investigated. Using drilling of carbon fiber reinforced plastics (CFRPs) as an application case of the investigated cutting tools, synchrotron X-ray diffraction in transmission geometry allowed us to observe the residual stress state along the direction of the principal residual stress tensor component $\sigma_{22}$ for multilayer as well as nanocrystalline CVD diamond-coated specimens and cutting tools, respectively.

2. Influences of CVD Diamond Coating Adhesion

Within the application of CVD diamond-coated cutting tools, the lack of consistent coating adhesion between the thin-film coating and substrate material limits the reliability of the cutting tool. Although advantageous mechanical properties are attributed to CVD diamond, the failure of thin-film coatings is based on various influencing factors. These can be differentiated as interconnecting substrate-, coating-, and manufacture-based influences.\(^7\)

2.1. Cutting Tool Substrate Material

Variations in substrate composition and structure of a substrate material affect the mechanical profile of the substrate material.\(^8\) Commonly used cobalt-bonded cemented carbide substrates (WC-Co) vary in terms of cobalt content, grain size, and possible amounts of mixed added carbides.\(^9\) Within the application spectrum of CVD diamond-coated tools, WC-Co substrates with a cobalt content of $w_{\text{Co}} = 6–10$ wt% are used industrially. Depending on the content of the cobalt binder phase, graphitization can occur within the substrate–coating interface during coating deposition. Thus, cemented carbide substrates with a cobalt content of $w_{\text{Co}} > 10$ wt% are solely used by depositing an interlayer of, for example, silicon carbide (SiC).\(^10,11\) The cobalt content increases the toughness of the tool material and is therefore relevant for applications with alternating thermomechanical loading during machining, e.g., in milling and drilling processes.\(^11\)

As an alternative to commonly used cobalt-bonded tungsten carbide substrate materials, ceramic-based cutting tool materials are investigated within cutting tool applications. Based on the mechanical profile of ceramic substrate materials such as the increasingly applied silicon nitride ($\text{Si}_3\text{N}_4$), the potential for application of these tools has been identified. With respect to conventional WC-Co cutting tools, comparable Vickers hardness HV30 can be attested to.\(^14\) In addition, the low density and advantageous thermal shock resistance show the potential for silicon-nitride-based ceramics regarding thermomechanical applications.\(^15\) Nevertheless, the brittleness and the accompanying lack of ductility of these materials result in a comparatively low fracture toughness, thus offering crack induction and fracture propagation throughout the material until tool failure occurs when mechanically overloaded during machining operations.\(^16\)

2.2. CVD Diamond Coating Morphology

Various coating morphologies such as microcrystalline, nanocrystalline, and multilayer structures can also affect the tool performance.\(^17\) Microcrystalline coating structures are characterized by columnar crystal growth with an average crystal size of $d_{\text{Dia,micro}} = 5 \mu$m. However, the high hardness is morphologically associated with high roughness regarding the arithmetic mean value of the profile coordinate $R_a$ in the range of $R_{\text{a,micro}} = 1.9 \mu$m. Nanocrystalline coating structures are characterized by a quasi-isotropic arrangement of diamond crystals averaging ranging from $d_{\text{Dia,nano}} = 5–500$ nm. The smooth surface with respect to the arithmetic mean value of the profile coordinate $R_{\text{a,nano}} = 1.0 \mu$m caused by the crystal size is particularly suitable for machining adhesive materials.\(^18–21\) When combining micro- and nanocrystalline coating morphologies alternatingly in multilayer structures, the properties of both layer morphologies are interconnected (Figure 1).

2.3. Cutting Tool Manufacture

Manufacture-based influences complete the interconnected factors that cause an impact on tool performance. Thereby, both substrate pretreatment and coating deposition show the most significant effect on the cutting tool behavior.\(^22\) Depending on the substrate material, an individual substrate pretreatment is mandatory to establish reproducible coating adhesion. Regarding the coating of hard metal substrate materials, the specific pretreatment methods include chemical etching of the WC-Co to reduce the subsurface cobalt content in the boundary zone.\(^23\) Because cobalt accelerates graphite formation within the substrate–coating interface, reduces nucleation of diamond on the substrate surface, and influences crystal growth as well as layer adhesion, the chemically invasive measure is mandatory.\(^24\) Nevertheless, the chemical material removal process weakens the surface of the
substrate material, which has to be considered during tool manufacturing as well as its later application.\cite{25}

Although a pretreatment is also necessary for silicon-nitride-based ceramic substrate materials, the obligation for a penetrating weakening of the subsurface is not given. In contrast to a hard metal, only a superficial surface procedure based on a mechanical or plasma pretreatment process is necessary, thus contributing to the superior coating adhesion in comparison to WC-Co substrates. The roughened surface increases the substrate's surface energy and the thermal expansion coefficient of the CVD diamond coating and the substrate is essential to resist damage initiation.\cite{26}

Apart from the introduced control variables cobalt content \(w_{\text{Co}}\), grain size \(d_s\), and coating morphology, which can be varied or specifically chosen for the later application, material-inherent residual stress \(\sigma_s\) as an accompanying system variable is focused on as an influencing factor in tool performance.\cite{27,28} Although residual stress originates from both intrinsic \(\sigma_{\text{intrinsic}}\) and thermal stress \(\sigma_{\text{thermal}}\), the latter represents the dominating effect of the overall stress state. Based on physical properties such as the thermal expansion coefficient \(\gamma\), substrate-specific residual stress is thermally induced within the coating. Due to deposition temperatures of the CVD diamond coating of \(T_{\text{CVD}} = 650–1000~\text{°C}\), the mismatch in the thermal expansion coefficient of the WC cutting tool substrates \(\gamma_{\text{WC}} = 5.4 \times 10^{-6}~\text{K}^{-1}\) and the CVD diamond coating \(\gamma_{\text{CVD}} = 0.8 \times 10^{-6}~\text{K}^{-1}\) causes thermal residual stress formation.\cite{29} In combination with the intrinsic residual stress \(\sigma_{\text{intrinsic}}\), the residual stress profile is completed (Equation (1)).

\[
\sigma_R = \sigma_{\text{thermal}} + \sigma_{\text{intrinsic}}
\]

Both terms are individually calculated as follows (Equation (2) and (3)).

\[
\sigma_{\text{thermal}} = \frac{E}{1 - \nu} \int_{T_{\text{Ambient}}}^{T_{\text{Deposition}}} \left(\gamma_T - \gamma_s\right) dT
\]

The parameters represent the Young’s modulus \(E\) in gigapascal, the Poisson’s ratio \(\nu\), \(T\) as the ambient as well as deposition temperature in degrees Celsius, and the thermal expansion coefficient in \(\text{K}^{-1}\) of the thin film \(\gamma_T\) and the substrate \(\gamma_s\), respectively.\cite{30}

\[
\sigma_{\text{intrinsic}} = \frac{E}{1 - \nu} \frac{\delta}{d_C}
\]

In addition to the already stated parameters, \(\delta\) refers to the lattice constant of CVD diamond in nanometers and \(d_C\) to the average crystal size in millimeters.

During comparative residual stress measurements of CVD diamond-coated tungsten carbide cutting tools using X-ray diffraction as well as Raman spectroscopy, substrate residual stresses are characterized with compressive stresses \(\sigma_{\text{compressive}}\) close to the substrate surface. Simultaneously, a gradient toward stress relaxation was observed with increasing information depth of the measurement technique independently of influencing factors such as pretreatment or substrate composition. In general, both measurement techniques show reasonable agreement. However, differences in beam spot size are apparent.\cite{27}

According to Hua et al., the intrinsic stress \(\sigma_{\text{intrinsic}}\) in CVD diamond coatings is tensile in nature as measured by X-ray diffraction. In the investigation, intrinsic stresses show direct proportionality to the deposition temperature \(T\) as well as indirect proportionality to the methane content \(w_{\text{CH4}}\) during the deposition process.\cite{31} By controlling deposition process parameters, the coefficient of thermal expansion regarding CVD diamond coating can be adjusted with given substrate materials to reduce the thermal stress component.\cite{32}

Nevertheless, the cumulative stress state of the CVD diamond coating consists of compressive residual stress \(\sigma_{\text{compressive}}\) that suppresses crack formation. However, the external thermomechanical load generated during its application in machining operations causes elastic as well as plastic deformation of the cutting tool material. Thereby, tensile residual stresses \(\sigma_{\text{tensile}}\) are induced within the coating and crack growth is initiated at the substrate-coating interface. In addition, pores that are located in the interface negatively affect the overall mechanical strength of the cutting tool and function as an origin of crack initiation.\cite{33} In case of damage initiation, the formed cracks propagate from the substrate-coating interface toward the surface of the CVD diamond coating, leading to substrate exposure. Thus, critical failure of the cutting tool eventually results.\cite{34,35} Taking into account the influence of the WC-Co substrate material regarding the residual stress state, two effects are represented. First, a decrease in average residual stress is produced as the amount of phases within the substrate decreases. Second, an increase in the stress magnitude is observed if the average grain size of WC particles decreases.\cite{36} In addition, thermal residual stresses \(\sigma_{\text{thermal}}\) are based on tensile residual stress \(\sigma_{\text{tensile}}\) in the binder phase while compressive residual stress \(\sigma_{\text{compressive}}\) is present within WC particles.\cite{37}

### 3. Cutting Tool Application

Overall, CVD diamond-coated tools offer considerable potential for machining high-performance materials.\cite{38} Nevertheless, spontaneous and premature tool failure can limit reliable machining independently of the machining process and regarded workpiece material (Figure 2).

When focusing on CFRP machining as an application case of CVD diamond-coated tools, workpiece-specific material properties, such as low weight combined with high stiffness, are achieved by the anisotropic and heterogeneous bond between the reinforcing fiber and the matrix.

The main reason for cutting tool failure is the varying chip formation mechanism of the fibers embedded in a matrix. With respect to the fiber orientation \(\Phi\) relative to the cutting tool edge, significant differences are present.\cite{39,40} The cutting process of embedded fibers is based on separation and damage mechanisms induced by the cutting edge. Thereby, the spatial position between fiber and tool cutting edge, which is defined as the fiber separation angle \(\theta\), shows importance for the chip formation process.\cite{41,42} In interaction, the abrasive CFRP fiber causes incremental cutting edge runout and asymmetric flank wear. Thus, subsequent coating delamination during the continuing thermomechanical loading from the machining process develops.\cite{43} The resulting increase in the cutting force components \(F_x\) consequently leads to a reduction in the quality of the
CFRP workpiece.\cite{44,45} Based on the emerging lack of process stability, the necessity for a holistic cutting tool improvement is focused on throughout different research approaches.\cite{46}

To assess the complex mechanism of tool failure based on the interaction of tribological and fracture mechanical superposition, model wear tests as well as machining analysis are performed. By isolating wear mechanisms via wear analogy tests, the fatigue strength of nanocrystalline CVD diamond coatings shows temperature-dependent wear behavior.\cite{47} In addition, a correlation between the exceeded shear failure stress in the substrate–coating interface and the observed coating delamination during milling points out the importance of modified adhesion properties. Thus, the substrate–coating interface of CVD diamond–coated WC-Co cutting tools predominantly represents the key location for the initiation of failure mechanisms such as cracks.\cite{48} Therefore, recent developments regarding cutting tool substrates include the application of silicon-nitride-based ceramic composites such as Si₃N₄. In comparison to WC-Co substrates, a superior coating adhesion is attested when combining silicon-based substrates with a CVD diamond coating.\cite{49} Due to the comparable thermal expansion coefficients of CVD diamond and Si₃N₄ as well as the structural compatibility of the coating and the substrate, advantageous conditions for tribological loads are obtained. Furthermore, the low density of Si₃N₄ substrates allows the introduction of high-speed cutting (HSC) process control within the machining strategy due to a reduction of rotational forces of the tool.\cite{50,51}

The resulting decline in spindle load improves the overall performance of the regarded machining process.

Throughout different applications as the cutting tool substrate material, the advantages of silicon-nitride-based ceramics have been identified for different workpiece material constellations.\cite{52} The industrial acceptance and application are currently limited due to the high manufacturing costs of ceramic shank tools that have to be compensated for by either high tool lifetime or short machining process times to provide an overall economical manufacture.\cite{53}

### 4. Experimental Section

Within this investigation, cobalt-bonded carbide-based and silicon-based CVD diamond–coated specimens as well as cutting tools were analyzed. To investigate the cross-sectional principal residual stress σ₂₂ of the specimen, focused ion beam (FIB) preparation was used. After applying two cutting tool specifications in drilling of the CFRP, different failure mechanisms were identified. In addition, synchrotron X-ray diffraction in transmission geometry was applied for the FIB-prepared specimen as well as the nanocrystalline CVD diamond–coated Si₃N₄ cutting tool. Regarding the latter analysis, the residual stress state was determined noninvasively over the course of the cutting tool application in machining the CFRP.

#### 4.1. Investigated Cutting Tool Specifications

To investigate individual influence factors concerning overall cutting tool performance, cutting tool specifications were varied regarding substrate material and coating morphology. For the WC-Co substrate material, the cobalt-bonded proportion included a cobalt content of w_co = 5 wt% (WC-5Co) in combination with a CVD diamond multilayer morphology. Thereby, an individual layer thickness of the alternating morphologies of s_D = 1.5 ± 0.5 μm was deposited. Although the FIB specimen showed a total coating thickness of s_FB ≈ 8 μm, the total coating thickness of the investigated cutting tools was set to an average coating thickness of s_FB = 12 ± 2 μm. To compare the differing influencing factors regarding the substrate material and the coating morphology, a nanocrystalline CVD diamond–coated Si₃N₄ cutting tool specification was used. Within the respective application case, which is based on previous studies, both cutting tool specifications showed most promising results regarding tool lifetime.\cite{7} The relevant substrate material

| Table 1. Mechanical properties of substrate materials. |
|-----------------------------------|------------------|------------------|
| Mechanical property               | Unit             | WC-5Co           | Si₃N₄            |
| Density, ρ                        | kg m⁻³           | 15.05            | 3.21             |
| Vickers hardness\(a\), HV         | –                | 1859             | 1850             |
| Fracture toughness\(b\), K<sub>C</sub> | MPa m⁻¹/²   | 9.3              | 5.8              |

\(a\) Measurement procedure according DIN EN ISO 6507-1\cite{58}. \(b\) Measurement procedure according ISO 28079.\cite{59}
properties such as density $\delta$, Vickers hardness HV30, and fracture toughness $K_{IC}$ characterize the individual mechanical profile of a given substrate material (Table 1).

All tool specifications were used to a cutting edge radius of $r_p = 15 \pm 3\, \mu m$. For both cutting tool specifications, a macrogeometry for double-edged stage twist drills was defined with a diameter of $d = 5.6\, mm$, a point angle $\alpha = 130^\circ$, and a right-hand helix at an angle of $\delta = 40^\circ$. Both substrates underwent subsequent CVD diamond tool coating under commercial, industrial production conditions. The details of the coating process parameters or pretreatment are subject to the nondisclosure agreement of the manufacturer and are not part of this study.

4.2. Experimental Methods

4.2.1. Specimen Preparation

FIB specimens of both cutting tool specifications were produced. The specimens were prepared by applying a dual beam with a beam current of $I_{FIB} = 1.4\, nA$ at an acceleration voltage between $U_{FIB} = 15$ and $25\, keV$ and a working distance of about $l = 8\, mm$. With a Helios NanoLab600 by FEI, Hillsboro, Oregon, USA, the prepared specimens were provided as lamellae with a thickness of $s_L = 200\, \mu m$, whereby the CVD diamond coating as well as substrate material was transversely exposed (Figure 3). Due to the focused ion radiation on the sample surface, defined material removal for the preparation of a cross-sectional area was achieved. In addition, coupling with an scanning electron microscope (SEM) allowed the observation of relevant artifacts such as pores and defects within the substrate–coating interface. Morphological differences could not be identified as the monolayers and nanolayers showed no differences in the material contrast. Also, after FIB preparation there were no topography differences for morphologic visualization of the investigated nanocrystalline and multilayer CVD diamond coating. However, Figure 1 serves as a schematic visualization of the nanocrystalline and multilayer coating morphology.

4.2.2. Coating Quality Analysis

Surface sensitive field emission electron probe microanalysis (FE-EPMA) with wavelength dispersive X-ray (WDX) spectrometry was used in this study to gain information about the quality of the diamond coating. It offers a qualitative assessment because peak shifts between diamond with $sp^3$ hybridization and graphite-like carbon with $sp^2$ hybridization carbon can be evaluated.[54] To differentiate the bonding of the structure related to either diamond or graphite-like carbon, the energy positions of the peaks with characteristic spectra were compared on FIB-prepared cross-sections of the investigated coating morphologies (Figure 4). The WDX analysis was performed with an FE EPMA JXA-8530 F by Jeol LTD, Tokyo, Japan, using an acceleration voltage of $U_0 = 15\, kV$ and a probe current of $I_p = 20\, nA$ allowing an electron energy of $E = 15\, keV$. The diamond coatings were measured by WDX spectrometer scan implementing a resolution of 1200 points per scan. A local beam spot diameter of $d_s = 3\, \mu m$ was used on the investigated nanocrystalline as well as multilayer CVD diamond coating. The intensity of the setup was chosen to gain a significant and reliable signal for the investigation.

By analyzing the hybridization of the investigated CVD diamond coatings via WDX, the quality of the coating throughout the prepared cross-section could be verified. A possible graphitization of the coating leads to destabilization of the coating-substrate interface and can give indication of its malfunction. The measurement close to the substrate–coating interface evaluated the position of the energy levels and compared it to the given reference materials such as graphite-like carbon with a characteristic energy of $E_G = 0.27\, keV$ and diamond with a characteristic energy of $E_D = 0.32\, keV$. A contiguous proximity to the diamond reference can be attested for both CVD diamond coating morphologies with a characteristic energy of $E_{CVD} = 0.31\, keV$. The high quality of the deposited coating was confirmed by analytically excluding graphitization from the investigated area. Insufficient coating adhesion due to graphitization within the coating and especially in the substrate–coating interface could be excluded from the following analysis.

4.2.3. Residual Stress Analysis

After the successful lamellae preparation, a cross-sectional measurement of the principal residual stress $\sigma_{22}$ along the exposed specimen was possible. To gain details of the present stress state of the investigated specimen and the cutting tools, respectively, synchrotron X-ray diffraction in transmission geometry was

![Figure 3. FIB cross-section: a) WC-5Co substrate; b) Si$_3$N$_4$ substrate.](image-url)
used. In contrast to common residual stress measurement techniques such as Raman spectroscopy and conventional X-ray diffraction, synchrotron X-ray diffraction offers spatially resolved analysis of the residual stress state. Apart from FIB-prepared specimen, the residual stress state of the CVD diamond-coated cutting tools was analyzed nondestructively. Based on synchrotron X-ray nanodiffraction introduced by Keckes et al., a method in transmission geometry was developed to analyze the residual stress state within the multilayer CVD diamond coating on tungsten carbide as well as ceramic drill within local resolution.[55]

While analyzing the multilayer coating morphology, the individual morphological layers, i.e., nanocrystalline and microcrystalline, only differed in microstructure. Thus, matching in their reflection position $2\theta$, a separation of the information of the varying morphological layers was therefore only possible by scanning with a precisely focused X-ray beam. The measurements were conducted on beamline P03 at the X-ray radiation source PETRA III from the German Electron Synchrotron Research Center, DESY, Hamburg, Germany. The prepared FIB lamellae showed the necessary planarity to irradiate the individual layers parallel to the CVD diamond coating without irradiating neighboring layers. In this process, high-energy X-rays are radiated through the sample perpendicular to the normal of the CVD diamond coating. The diffracted beam in the form of Debye–Scherrer spheres was then detected using a 2D detector. The residual stress state as a function of depth $z$ or coating thickness $s_D$ respectively was determined by incremental translation of the specimen at defined intervals of $t_{\text{step}} = 0.20 \mu m$. For the investigations of multilayer CVD diamond coatings, the particularly thin individual layer thicknesses of $s_{D,i} = 1.5 \pm 0.5 \mu m$ posed an additional challenge because beam widths in the micrometer range must thus be achieved. With a focus beam area of $A = 300 \times 260 \text{nm}^2$ and a beam energy of $E = 87.1 \text{keV}$, the residual stress state according to synchrotron X-ray diffraction in transmission geometry was determined. Thereby, it was assumed that the residual stress $\sigma_{zz}$ at the surface is equal to zero.

Stress relaxation and redistribution respectively due to specimen preparation via FIB were determined simulatively (Figure 5). In analogy to the established procedure, the simulations were performed using the commercial simulation software Abaqus by Simulia–Dassault Systèmes, Vélizy-Villacoublay, France.[56] Figure 4 shows the residual stress state regarding $\sigma_{22}$ for the initial condition in the center of the specimen before preparation. The residual stress profile was previously

Figure 4. WDX analysis for qualitative analysis of CVD diamond coatings.

Figure 5. Simulated residual stress redistribution due to specimen preparation.
determined in a cooling simulation for a CVD diamond coating with a thickness of $s_D = 12 \mu m$ on a WC-Co substrate. Subsequently, the effect of reducing the specimen thickness on the individual stress components was determined by stress relaxation as well as redistribution. According to the performed simulation, the residual stress state for the $\sigma_{22}$ component of the bulk material, i.e., the initial state, is comparable to the provided lamellae with a thickness of $s_L = 200 \mu m$.

4.2.4. Machining Trials

Technological investigations regarding machining trials were performed on a machining center of type Ultrasonic 260 Composites manufactured by Sauer GMBH, Stipshausen, Germany. The unidirectional (UD) CFRP workpiece material with a panel thickness of $s_{CPFRP} = 15 \pm 1 \text{ mm}$ serves as industrial reference used in aeronautical wing construction. To evaluate the tool performance, a continuous measurement of the process forces during drilling was conducted. For this purpose, a 9125 A rotary cutting force dynamometer from Kistler Instrumente AG, Winterthur, Switzerland, was used to document the feed force $F_f$. With a measuring frequency of $f = 1000 \text{ Hz}$, the measuring signal was scanned.

The test parameters were kept constant for the machining trials. A cutting speed of $v_c = 105 \text{ m min}^{-1}$ and feed of $f = 0.06 \text{ mm at } n = 6000 \text{ min}^{-1}$ were chosen according to industrial references. Tool wear documentation was continuously carried out on the rake face of the drills’ second stage at an interval of $N = 10$ bore holes regarding the maximum tool wear $VB_{\text{max}}$ on rake as well as flank face. In case of initial coating delamination and exposition of the substrate material, an additional further $N = 100$ holes were drilled with the same tool to investigate the wear behavior progression. The end of the experiment was marked by either the aforementioned condition or in case spontaneous tool breakage of the shaft prevented further use of the cutting tool. In total, three machining trials, i.e., $n = 3$, were performed for each cutting tool specification.

5. Results

First, the FIB-prepared specimen was analyzed according to the mentioned synchrotron X-ray diffraction in transmission geometry (Figure 6a). The experimentally determined course of the reflection intensities and reflection half-widths, i.e. full width at half maximum (FWHM), as a function of the measurement depth $z$ along the coating thickness $s_D$ regarding the multilayer CVD diamond coating deposited on WC-5Co substrate is illustrated (Figure 6b). An increase in (111)-reflection intensities and a decrease in reflection half-width FWHM is observed in...
the microcrystalline regions. Contrasting behavior is documented for the nanocrystalline coating morphology, whereby a decrease in the (111)-reflection intensity correlates with an increase in the measured reflection half-width FWHM. The residual stress profile for the principal residual stress \(\sigma_{22}\) as a function of measurement depth \(z\) along the coating thickness \(s_D\) complements the analysis (Figure 6c). As the measurement was performed at a lamella thickness of \(s_L = 200 \mu m\), the influence of the stress relaxation and redistribution was simultaneously assessed to be negligible. A global increase in compressive residual stress \(\sigma_{\text{compressive}}\) from the substrate–coating interface toward the surface can be observed. The respective measurement depth \(z = 0 \mu m\) represents the surface of the coating in this depiction. Local maxima regarding compressive stress \(\sigma_{\text{compressive}}\) are evident within the microcrystalline coating morphology. Thereby, along the cross-section from the substrate–coating interface toward the coating surface, the principal residual stress ranges from \(\sigma_{22,\text{micro},1} = -1.12 \pm 0.1 GPa\), \(\sigma_{22,\text{micro},2} = -1.18 \pm 0.1 GPa\), \(\sigma_{22,\text{micro},3} = -1.89 \pm 0.1 GPa\), and \(\sigma_{22,\text{micro},4} = -3.24 \pm 0.1 GPa\). In comparison, the analyzed nanocrystalline morphologies tend to decrease the principal residual compressive stress within the alternating coating structure. From the substrate–coating interface toward the coating surface, the peaks of the principal residual stress values quantify as \(\sigma_{22,\text{nano},1} = 0.27 \pm 0.1 GPa\), \(\sigma_{22,\text{nano},2} = -0.35 \pm 0.1 GPa\), \(\sigma_{22,\text{nano},3} = -0.51 \pm 0.1 GPa\), and \(\sigma_{22,\text{nano},4} = -1.80 \pm 0.1 GPa\). For the nanocrystalline CVD diamond coating, the tendency toward an order of tensile residual stress can therefore be attested to. This tendency correlates with varying studies on CVD diamond monolayers, whereby superior compressive residual stress \(\sigma_{\text{compressive}}\) was observed for microcrystalline morphology compared to nanocrystalline morphology. The global increase in compressive residual stress \(\sigma_{\text{compressive}}\) starting at the interface with \(\sigma_{22,\text{micro},1} = -1.12 \pm 0.1 GPa\) toward the coating surface with a residual stress state \(\sigma_{22,\text{nano},4} = -1.80 \pm 0.1 GPa\) presumably originates from the increased mechanical obstruction of growing crystallites with increasing coating thickness \(s_D\).

In analogy to the aforementioned investigation of the multilayer CVD diamond coating deposited on the WC-5Co substrate, the (111)-reflection intensities, the reflection half-width FWHM, as well as the principal residual stress \(\sigma_{22}\) of the nanocrystalline CVD diamond coating on the Si3N4 substrate were analyzed (Figure 7a). The (111)-reflection intensity qualitatively increases during beam immersion from the coating surface, i.e., measurement depth \(z = 0 \mu m\), to the substrate–coating interface, i.e., measurement depth \(z = 8.6 \mu m\), thus increasing the measuring volume element at the coating surface until an intensity decrease at the substrate–coating interface is documented. The reflection half-width FWHM remains almost constant (Figure 7b). Regarding the principal residual stress \(\sigma_{22}\) for the nanocrystalline CVD diamond coating deposited on Si3N4, tensile residual stresses are uniformly observed (Figure 7c). Contiguous to the coating surface, the minimum of the tensile principal residual stress \(\sigma_{\text{tensile}} = 0.24 GPa\) is measured. The maximum of the

Figure 7. Ex situ residual stress analysis of CVD diamond–coated Si3N4 specimen. a) Schematic depiction of experimental setup. b) Correlation of (111) reflection intensity, reflection half-width FWHM, and depth \(z\). c) Principal residual stress tensor component \(\sigma_{22}\) as a function of measurement depth \(z\).
tensile principal residual stress $\sigma_{\text{tensile},2} = 1.47$ GPa is located at a measurement depth of $z = 3.6$ $\mu$m. The overall determination of the residual stress state with a total average of $\sigma_{22,\text{Si}_3\text{N}_4,\text{average}} = 1.05$ GPa shows the variation of the residual stress values over the course of the measurement depth $z$. The inconsistency hints at an inhomogeneous state of the principal residual stress that can affect the cutting tool performance depending on its degree. Nevertheless, the overall tensile residual stress state of Si$_3$N$_4$ can be attributed to the comparable thermal expansion coefficient $\gamma$ of CVD diamond and Si$_3$N$_4$, which results in lower compressive residual stresses, whereas a thermal mismatch of WC-5Co and CVD diamond is reported. In addition, the increased proportion of intrinsic residual stress $\sigma_{\text{intrinsic}}$ resulting from grain boundary relaxation due to a fine-grained microstructure contributes to this. Furthermore, the lattice mismatch between the CVD diamond and the Si$_3$N$_4$ substrate causes an elongation of the lattice within the CVD diamond structure, i.e., tensile stress, and a compression of the lattice within the Si$_3$N$_4$ substrate.

Using synchrotron X-ray diffraction in transmission mode with a focused beam, it was possible to investigate the residual stress state on CVD diamond–coated silicon-nitride-based ceramic drills (Figure 8). Thereby, the residual stress $\sigma_{22}$ was locally resolved initially as well as after machining of a total bore hole quantity of $N = 650$. For this purpose, the CVD diamond coating was radiated parallel to the flank face of the cutting tool along the coating thickness in two measuring locations (Figure 8a). For the residual stress analysis, the nanocrystalline CVD diamond–coated Si$_3$N$_4$ cutting tool was radiated at initial as well as after exceeding the defined abort criteria for wear behavior at a total borehole quantity of $N = 650$ (Figure 8b). In the initial state, the CVD diamond coating exhibits principal residual stresses ranging from $\sigma_{\text{initial},\text{MP}1} = -1$ to 1 GPa centered on the flank face as measuring point 1 (MP1). In contrast, tensile residual stresses of up to $\sigma_{\text{initial},\text{MP}2} = 0.8$ GPa are predominantly present at the cutting edge corner at measuring point 2 (MP2). During cutting tool application in the regarded machining operation, the thermomechanical load of the cutting process leads to wear on the flank face. Over the course of the coating thickness $s_D$, this causes an overall shift of the principal residual stress $\sigma_{22}$ toward a compressive stress state by $\Delta\sigma_{22,\text{MP}2}/C251$ GPa at the cutting edge corner or MP2. In comparison, after the cutting tool application only a marginal difference regarding principal residual stress $\sigma_{22,\text{MP}1}$ can be detected. Compared to MP1, the cutting edge corner, i.e., MP2, is exposed to higher mechanical stress due to the kinematic engagement conditions of the drilling process. Also, when in contact with the workpiece material, a higher rotational velocity compared to MP1 causes additional load on the respective location. Thus, the distinct shift in residual stress state is quantified.

The tool performance regarding average bore hole quantity $N_A$ as well as feed force development was assessed in machining trials (Figure 9). The machining investigations provide insights into the interaction between the CVD diamond coating morphology, the substrate material, and the coating adhesion when machining the CFRP workpiece material. In general, a varying application behavior of the tool specifications in terms of average number of holes $N_A$, tool wear VB, and process forces can be identified. Generally, the observed damage behavior leading to ultimate tool failure is dependent on the substrate material. The performance of WC-5Co tool specifications is limited by delamination of the CVD diamond coating. This can only partially be detected on tool specifications with the Si$_3$N$_4$ substrate. These Si$_3$N$_4$ tools primarily fail due to the fracture behavior of the
tool shank. Both modes of damage cause the present variation of machining test results (Figure 9a). Thus, depending on the cutting tool substrate, cutting tool failure is attributed to deficient CVD diamond coating adhesion or insufficient mechanical strength of the substrate material. For WC-5Co, an average bore hole quantity of \( N = 190 \) was achieved. Throughout the machining operation, maximum feed forces of \( F_f = 53.18 \text{ N} \) were documented for the maximum achieved bore hole quantity of \( N_{\text{max, WC-5Co}} = 420 \) (Figure 9b). When comparing the coating morphologies of the multilayer CVD diamond coating, the microcrystallinity correlates with the high hardness of \( \text{HV}_{\text{micro}} = 118 \text{ GPa} \), increased elastic modulus of \( E_{\text{micro}} = 1143 \text{ GPa} \), superior fracture strength \( \sigma_{\text{f,micro}} = 7.1 \text{ GPa} \), and preferable adhesion characteristics within the substrate–coating interface. Nanocrystallinity exhibits a comparable high number of grain boundaries as well as a low friction coefficient of \( \mu_{\text{nano}} = 0.04 \). By alternating the structure of the coating multilayer morphology, the tendency for crack initiation and development is primarily reduced because the morphological interfaces act as a mechanical barrier within the coating. However, a sharp interface transition structure potentially offers the possibility for separation during external loading due to the increase in graphitic content at the interface of the nanocrystalline morphology. Dominant tool wear behavior occurs primarily on the rake face before coating delamination exposes the substrate material at the cutting edge over a large area. As a result, the delamination propagates onto the flank face. The continuous increase in feed force \( F_f \) is assumed to correlate with an increase in the cutting edge radius \( r_b \) due to abrasive interaction with the workpiece material. Partial decrease in feed force \( F_f \) may correspond to local coating delamination that ultimately results in disadvantageous contact conditions between the cutting tool and workpiece material. In correlation to the residual stress state, the comparably higher proportion of initial compressive residual stress \( \sigma_{\text{compressive}} \) initiates critical coating delamination as well as crack development when exceeding the critical residual stress \( \sigma_R \) as well as the shear stress \( \sigma_S \). The specific quantification of the stated stress components of critical failure remains a current scientific issue.

In comparison, superior tool performance regarding tool life as well as feed force development of the nanocrystalline CVD diamond–coated Si₃N₄ cutting tool specification is shown. The tool wear of this cutting tool specification differs both in its characteristics and in its progression. The tool performance is not limited due to insufficient coating adhesion between the Si₃N₄ substrate material and the nanocrystalline CVD diamond coating. Instead, the performed machining trials were constantly terminated by an occurring tool breakage of the shank tool, causing high variation in machining trial results. The presented failure mechanism equally limits the application of this cutting tool specification as the aforementioned coating delamination of the WC-5Co cutting tool specification. Although tool wear can generally be identified on the cutting edge, it is primarily

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**Figure 9.** Cutting tool performance: a) tool life time; b) feed force development.
localized on the flank face. An average bore hole quantity of $N = 320$ was achieved. A continuous increase of feed force development until a feed force of $F_f = 66.11 \text{ N}$ without notable coating development shows the general superiority in coating adhesion. Although higher process forces of $\Delta F_f = 19.71\%$ affect the cutting edge for a higher bore hole quantity of $\Delta N_A = 40.62\%$ on average, tool failure is not based on coating delamination. Instead, breakage of the shank tool due to insufficient mechanical strength regarding the comparably low fracture toughness of $K_{IC, \text{Si}_3\text{N}_4} = 5.8 \text{ MPa m}^{1/2}$ of the $\text{Si}_3\text{N}_4$ substrate shows the dominant failure behavior within this cutting tool specification. Thus, during application-related deformation of the shank tool during contact of the cutting edge and workpiece material, inevitable development of cracks in the crystal lattice is initiated. In contrast to the WC-5Co cutting tool specification, an unpredictable coating failure is not evident but rather a continuous wear progression of the CVD diamond coating. Regarding the ex situ analysis of the residual stress state during machining, the coating adhesion evidently shows increased properties. The documented initial tensile residual stress $\sigma_{\text{Res tensile}}$ is shifted throughout the cutting tool application without exceeding the critical principal residual stress $\sigma_R$ that may cause coating delamination. Thus, coating adhesion is correspondingly reproducible, which impacts industrial machining processes with respect to machining process stability and constant workpiece quality. Because the two cutting tool specifications differ in failure mechanisms, assumptions regarding the residual stress state and its effects on CVD diamond coating behavior can be derived. According to previous investigations, CVD diamond–coated cutting tools possessing compressive residual stress in combination with a high coating quality potentially have increased tool life. Nevertheless, the universal validity of this statement cannot be accepted. Depending on the cutting tool application and the related machining process, the growth of an interface crack can be promoted distinctively by the impact of local compressive stresses that arise in thermomechanically loaded components. When these local stresses superimpose with the initially distributed residual stress, favorable conditions for delamination are produced. As soon as a critical threshold of compressive residual stress is reached, the local stresses individually can cause growth of a delamination in the substrate–coating interface of CVD diamond–coated cutting tools. Compressive residual stress indicates to restrain the formation and propagation of cracks while simultaneously the existence of the tensile residual stress induces crack propagation. In the investigated application case partly opposing results are produced based on the cutting tool specification.

Based on the technological investigations conducted, it was possible to qualitatively extend the original residual stress model by Uhlmann et al. with respect to the cutting tool substrate (Figure 10).[54] Thereby, a critical substrate-specific maximum residual compressive stress state is assumed that is reached during cutting tool application. Subsequently, coating delamination results. The extended model assumption depends on the used cutting tool substrate material as well as on the CVD diamond coating morphology deposited. In direct comparison, the differing wear behavior of the investigated WC-5Co and $\text{Si}_3\text{N}_4$ substrate materials originating from tungsten carbide and the silicon-nitride-based ceramic substrate, respectively, can be attributed to the investigated inherent residual stress state. Based on the presented results, the previous qualitative correlation between tool life $t_c$, principal residual stress $\sigma_R$, and coating quality $Q_c$ is extended. The latter represents the ratio of sp$^3$-hybridized carbon to sp$^2$-hybridized carbon, i.e., diamond–graphite relation. Thereby, high coating quality generally correlates with an increased tool lifetime compared to moderate or low coating quality. Although CVD diamond–coated WC substrates tend to dominantly show an overall compressive stress state, CVD diamond–coated silicon-nitride-based ceramic substrate materials are prone to a moderate tensile residual stress state. Due to the thermomechanical load during machining, the compressive residual stress $\sigma_{\text{compressive}}$ generally increases. Exceeding a critical stress state leads to delamination of the CVD diamond coating and tool failure. In direct comparison, this damage behavior is prematurely achieved with CVD diamond–coated WC-5Co cutting tool specifications. The diverging premise of principal residual stress $\sigma_R$ represents the most significant influence on the later cutting tool application. Nevertheless, regarded influencing factors such as substrate material and coating morphology exert influence on the extent of the demonstrated dominating factor. Regardless of the aforementioned tool breakage of CVD diamond–coated silicon-nitride-based ceramic cutting tools, the overall tensile residual stress state has a beneficial effect on the coating adhesion and tool performance. However, a critical threshold regarding tensile stress that initiates cracks or coating failure is assumed likewise in analogy to a critical threshold within compressive residual stress.

6. Conclusion

Based on the presented investigation, varying principal residual stress states were identified in CVD diamond–coated cutting tool specifications. According to previous studies, a higher principal residual compressive stress tensor component $\sigma_{\text{compressive}}$ promotes an increase in tool life. Even though the compressive residual stress $\sigma_{\text{compressive}}$ within CVD diamond coatings enhances the coating adhesion, the substrate material can be overstressed. The desired contribution to mechanical roughness
locking in the substrate–coating interface may be superimposed by deterioration of coating adhesion and subsequent delamination and tool failure. Regarding nanocrystalline CVD diamond coatings, the initiated cracks dominantly show intercrystalline orientation due to the presence of sp²-hybridized carbon, i.e., graphite, along the crystal boundaries. Damage progression within microcrystalline CVD diamond coating morphologies is identified to show both the inter- and transcrystalline directions. When observing fracture in multilayer morphologies, the interfaces of sequential nano- and microcrystalline morphologies antagonize the progressing crack development, independently of the substrate material. Thereby, a general compressive residual stress state potentially increases tool life due to suppression of crack initiation or propagation. Nevertheless, with respect to the cutting tool application and the regarded machining process, delamination of the CVD diamond coating occurs when the compressive residual stress exceeds a critical threshold. According to Mylvaganam and Zhang, the delamination mode varies, indicating that the stress ratio is an important factor that influences the nature of the delamination initiation and the failure mechanism. This threshold shows dependence on the cutting tool specifications, i.e., the substrate material and coating morphology. Possibly, additional factors such as the coating thickness \( \sigma_D \) may further influence the critical threshold of compressive residual stress for delamination. The performed analysis extends the current state of the art, indicating a beneficial tool performance when an initial tensile principal residual stress tensor component \( \sigma_{\text{tensile}} \) is present. To enable the determination of the principal residual stress tensor component \( \sigma_R \) in different cutting tool specifications, a novel synchrotron transmission measurement methodology was developed. The focused synchrotron radiation allows the determination of the residual stress state as well as its gradient over the course of the coating thickness \( \delta_D \) in the deposited CVD diamond coating morphology on varying substrate materials. The following conclusions can be drawn for the investigated application of the cutting tools. 1) Tool performance regarding tool life and wear behavior of the investigated cutting tool specifications is dominantly influenced by the state of residual stress \( \sigma_R \), within the deposited CVD diamond coating. 2) CVD diamond-coated substrate materials based on a hard metal primarily show coating delamination as the critical wear mechanism due to an initial compressive principal residual stress tensor component \( \sigma_{\text{compressive}} \). 3) CVD diamond-coated substrate materials based on a silicon-nitride-based ceramic are prone to mechanical failure of the shank tool due to the low fracture toughness. 4) External mechanical loading of the cutting tool during machining causes a continuous shift of the residual stress state within the CVD diamond coating toward increasing compressive residual stress until coating failure occurs. 5) The initial tensile principal residual stress tensor component \( \sigma_{\text{tensile}} \) tends to allow an increase in tool performance regarding tool life, wear behavior, and coating failure.

To transfer the gained knowledge about the investigated cutting tool specifications and the regarded application case of CFRP machining, a defined residual stress state within deposited CVD diamond coatings may be taken into account during cutting tool development. By adapting deposition process parameters such as the deposition temperature \( T_D \) or the reactive gas concentration \( c \), beneficial manufacturing conditions leading to a modified state of residual stress \( \sigma_R \) for an advantageous tool performance can be achieved independently of the cutting tool substrate.

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### Conflict of Interest

The authors declare no conflict of interest.

### Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

### Keywords

chemical vapor deposited diamond, cutting tools, residual stresses

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