



Review



# Tool Coating Characterization Methods for Fibre Metal Laminates (FMLs) Machining Applications: A Narrative Review

Rúben D. F. S. Costa<sup>1,2,\*</sup>, Sónia L. S. Simões<sup>2</sup>, Tiago E. F. Silva<sup>2</sup>, Abílio M. P. Jesus<sup>2</sup>, Francisco J. G. Silva<sup>3</sup>, Daniel A. Figueiredo<sup>4</sup>, Andresa Baptista<sup>3</sup> and Gustavo F. Pinto<sup>3</sup>

<sup>1</sup> INEGI, LAETA, Universidade do Porto, Rua Dr. Roberto Frias, 4200-465 Porto, Portugal

<sup>2</sup> DEMec, Department of Mechanical Engineering, Faculty of Engineering, University of Porto, Rua Dr. Roberto Frias s/n, 4200-465 Porto, Portugal

<sup>3</sup> CIDEM, ISEP, Polytechnic of Porto, Rua Dr. António Bernardino de Almeida, 4249-015 Porto, Portugal

<sup>4</sup> Product Development Department, Palbit, S.A., 3854-908 Branca, Portugal

\* Correspondence: [rdcosta@inegi.up.pt](mailto:rdcosta@inegi.up.pt)

**How To Cite:** Costa, R.D.F.S.; Simões, S.L.S.; Silva, T.E.F.; et al. Tool Coating Characterization Methods for Fibre Metal Laminates (FMLs) Machining Applications: A Narrative Review. *Journal of Mechanical Engineering and Manufacturing* **2026**. <https://doi.org/10.53941/jmem.2026.100021>

Received: 28 February 2026

Revised: 25 March 2026

Accepted: 30 March 2026

Published: 29 April 2026

**Abstract:** Characterization methods constitute a fundamental path of determining the properties of a machining tool coating before experimental works are undertaken, so that its fitness for a certain operation is studied beforehand. When the requirement is to machine fibre metal laminates, this is even a more important matter, since the coating is expected to withstand several challenging solicitations, which most of the times result in an excessive tool wear resultant from the composite fibres' abrasiveness and the dissimilar materials distinct cutting patterns. Nevertheless, this problem can be avoided through a correct characterization of the coating, which will both make it possible to avoid early tool wear and increase its lifetime, resulting in huge economic savings and lower waste, making the overall process much more sustainable. Machining tool coatings can be characterized following diverse physical, mechanical and tribological techniques. The most frequent characterized methods used for this application are the atomic force microscopy (AFM), the scanning electron microscopy (SEM), the x-ray diffraction (XRD), the Raman spectroscopy, the nano/microindentation, the scratch test, the pin-on-disc test and the micro-abrasion. This paper consists of a narrative review containing the state-of-the-art of the literature using these methods, with several combined in some cases for a complete coating characterization, in addition to a SWOT analysis for each methods group. Accordingly, this article's main objective is to collect the important findings regarding machining tools' coating characterization studies performed in the literature from 2020 to 2026 to highlight their differences and the best for each determined application.

**Keywords:** characterization techniques; SEM; XRD; nano/microindentation; pin-on-disc test

## 1. Introduction

One of the most important concerns regarding the modern transportation industry, especially on the aeronautical and automotive fields, is their excessive fuel consumption, causing negative impacts on the atmosphere [1]. For this reason, environmental regulations to reduce airplanes and cars' carbon emissions have been pushing companies to develop new solutions to tackle this challenge. Over the years, this resulted in a growth



**Copyright:** © 2026 by the authors. This is an open access article under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

**Publisher's Note:** Scilight stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.

in the search for lighter components whose properties and performance were equivalent or superior to the previous materials, leading to the development of fibre metal laminates (FMLs) [2]. These are hybrid materials are composed of different layers of thin metal sheets, usually titanium or aluminium, and composites, such as carbon fibre- or glass fibre reinforced polymers (CFRP/GFRP), for example [3]. The combination of different materials resulted in enhanced properties compared to the separate use of each material, namely high strength-to-weight ratio, or fatigue and fracture resistance, making this a desirable solution for the mentioned applications [4]. Their lower weight resultant from the composite component allowed a significant fuel consumption reduction and their high mechanical strength and toughness from the metal part ensured that these multi-materials are able to withstand the challenging solicitations these structures face [5].

The construction of airplanes and cars requires a great variety of components which must undergo a set of operations in different manufacturing processes in order to acquire their final shapes. Machining operations, such as groove milling, turning, drilling, boring or grinding, are still indispensable to comply with the strict dimension requirements these industries demand for their parts. When it comes to joining different components together, the drilling process is still the preferred method, since the use of bolted or riveted connections, mainly in the aeronautical industry, is crucial, as they are able to endure harsher conditions than competing methods, such as the use of adhesives [6]. In spite of this, machining FMLs constitutes a challenging task, since the tool has to cut through dissimilar materials with drastically different thermal and mechanical properties in a single operation, with the aggravating circumstance that the composite materials' fibres are strongly damaging, hindering this process' quality [7,8]. These factors cause an accelerated tool wear, and the thermal shock allied to the imperfect surface of the damaged tool ultimately result in defects observed in the component itself, contributing to a defective batch and a huge economic loss for the manufacturing company. This is a major issue in these industries, where the standard tolerance requirements are very strict. In the drilling process of fibre metal components for aeronautical structures, the maximum acceptable value is 1.6  $\mu\text{m}$  for aluminium and 3.2  $\mu\text{m}$  for CFRP panels [9]. On the other hand, in the case of the delamination factor considering the diameter of the delaminated region, if it surpasses the 1.200 limit, the damage is considered severe and unacceptable [10].

For this reason, in order to tackle these problems, Pai et al. [11] and Giasin et al. [12] adopted and unconventional machining approaches in their work, [11,12] such as abrasive water jet machining, laser jet machining, electric discharge machining, ultrasonic machining or cryogenic machining. Alternative tool sintering processes may also provide good results, such as SPS (Spark Plasma Sintering) or FAST (Field Assisted Sintering Technology), as described in the works by Wachowicz et al. [13] and Kruzel et al. [14], respectively. These resulted in tools with higher abrasive wear and useful lifetime compared to traditional production techniques. Nevertheless, independently of the method or tool used, a tool coating utilization is essential, since it contributes to extend the tool's useful lifetime, thus also leading to a better hole quality, since several studies proved already that the choice of a suitable coating outstands performing machining operations with uncoated tools results [15,16]. The mostly observed wear mechanisms during the drilling process are adhesion, also known as built-up edge (BUE), abrasion, diffusion, debonding and oxidation [17,18], so only resorting to a careful study, achieved through the correct characterization techniques, can they be mitigated. Notwithstanding, apart from the coating's material choice, the process from which it is produced plays a decisive role in its final properties. When they started being developed, only simple coatings were applied to the tools' surfaces, consisting of a single layer or element, but, throughout time, the work carried out by research teams worldwide led to several developments in this field, which culminated in the creation of complex systems, structured to the nanometric scale [19]. As a solution to increase the tool's useful lifetime and component quality, specific deposition methods have been developed in the literature, for example the chemical vapour deposition (CVD) and the physical vapour deposition (PVD), the most prominent among all, although other sputtering processes also exist, such as the HiPIMS [20,21].

Since the need to enhance the efficiency of production processes gave rise to the development of wear resistant hard coatings, basic characterization techniques are not enough to predict the coating's behaviour for multi-material machining anymore. Nowadays, it is fundamental to study the coating's adhesion, its thermal stability and mechanical behaviour in dynamic cutting conditions, key factors that determine its effectiveness [22]. This can only be accomplished through the study of the microstructure and properties of the coating, to which several methods can be employed. To this day, many different characterization techniques have been developed, allowing the analysis and determination of numerous properties in the magnetic, electrical, thermal or optical domains. These are split into diverse categories, with the most prominent being the microscopy (AFM and SEM/EDS), diffraction (XRD), and spectroscopic (Raman spectroscopy); however, other techniques, for example hardness evaluation or wear resistance, are also crucial for a coating's characterization [23–25]. Multi-layered thin films, for example, constitute a real challenge to characterize, explained by their morphology being heavily dependent on the respective deposition techniques, added to the more unstable nature of nanolayers, since they are thinner [26].

Although several reviews exist in the literature concerning different machining topics, none focuses specifically on the advanced characterization of machining tool coatings, especially regarding FMLs, which pose greater challenges and, thus, require special attention in this aspect. Accordingly, this review article aims to critically analyse the trending techniques used to characterize machining tool coatings since the beginning of this decade, compare them with respect to their purpose and identify the best ones to obtain a complete overview of a determined coating, from physical characterization to mechanical and tribological tests.

## 2. Characterization Techniques

For the development of this review, the literature was thoroughly analysed for multiple studies of experimental tests, which were then condensed to highlight the mostly used techniques in the characterization of machining tool coatings. This research was conducted considering only works performed from 2020 to 2026, in order to keep the review as updated as possible, and the keywords used were the mentioned characterization methods. These can be grouped into the following categories: microscopy, diffraction, spectroscopic, all of which are physical characterization methods, mechanical and tribological.

### 2.1. Microscopic Techniques

These use magnification to evaluate a material's surface in detail. The atomic force microscopy (AFM), in its contact, tapping, and non-contact aspects, uses a probe to obtain information regarding the surface of a sample at each point. A scanner registers the movements of the sample in the three space directions: x, y and z, achieving a detailed analysis of its surface.

Despite this, one of the most used microscopic techniques is the scanning electron microscopy (SEM). This method's resources include the examination of surface topography at a micro level, microstructures, grain sizes and cutting edges, performing the thickness measurement and allowing a morphology observation, besides being able to evaluate the surface chemical composition of the material in study with the energy dispersive spectroscopy (EDS) [27]. Additionally, electron backscatter diffraction (EBSD), for example, which is generally associated with the SEM, is mainly used in metals and has several advantages over conventional methods, despite its limited resolution (20–50 nm), allowing to obtain a wide range of characterization possibilities in a single method, which is not possible in others [19]. The EBSD is a powerful tool for crystal orientation mapping, phase imaging, determination of grain size distributions and texture, as well as strain analysis [28]. To complement, profilometry is usually used to measure the surface roughness of the coating [29]. Transmission electron microscopy (TEM) with electron diffraction is also an important technique in this group. It is widely applied in morphology and crystalline structure characterization, and it also provides elemental information of membrane materials [30].

Other examples of microscopic techniques used by some authors in materials characterization are the ion field, scanning probe, and scanning acoustic microscopy [31–33].

### 2.2. Diffraction Techniques

This kind of techniques provide detailed information about a material's crystallographic structure, microstructure, chemical composition, and physical properties. X-ray diffraction, or just XRD, can be used to evaluate diamond crystalline sizes and qualities [34], as well as to measure residual stresses. It allows the determination of the coating phase spectrum, with the different peaks indicating the intensity and crystal orientation of the constituent phases [35]. For the XRD method, the term nano diffraction is related to using X-ray beam diameters of 100 nm or less. The advantage of using such small beam sizes is obtaining depth-resolved information and a high resolution across the surface [36].

Other relevant techniques in this scope are the diffraction of monocrystals, polycrystalline materials and quasi-amorphous phases, neutron diffraction and crystallography [37–39].

### 2.3. Spectroscopic Techniques

These employ light to interact with matter and, thus, probe certain features of a sample to learn about its consistency or structure. Raman spectroscopy assesses diamond purity and bond characteristics, relating molecular vibration frequencies to the inter-atomic bonds. In coatings, it verifies the diamond film quality and stress state evaluation [40].

Furthermore, other examples of spectroscopic methods include synchrotron techniques, such as the X-ray absorption near-edge structure (XANES) and the extended X-ray absorption fine structure (EXAFS); surface analysis techniques, as the auger electron spectroscopy (AES) and the X-ray photoelectron spectroscopy (XPS);

and finally ion beam techniques, with the Rutherford backscattering spectroscopy (RBS) and the particle-induced X-ray emission (PIXE) as two main examples [41–45].

#### 2.4. Mechanical Techniques

Besides the physical characterization techniques, the mechanical properties also play a decisive role in the tool coatings' performance. The most important mechanical property to be considered in this aspect is the hardness, since higher hardness leads to lower tool wear during the machining process and, thus, to its extended lifetime. Accordingly, to evaluate the hardness of materials, some characterization methods can be used, namely nano and micro indentation techniques, which serve the purpose of measuring the elasticity and hardness of the coating, i.e., microhardness [46,47]. Furthermore, the scratch test is used to measure the adhesion critical load of the coating towards the substrate [48]. Additionally, there are different techniques to evaluate the coating's thickness, such as cutting the sample, polishing it and observing the cut zone, cutting the sample and promoting a fragile fracture to observe the thickness and structure of the coating, and finally through a specific test with a spherical cap.

#### 2.5. Tribological Techniques

As previously referred, the wear resistance is essential to evaluate the performance of tools and respective coatings, namely, to determine if a certain coating will be able to fulfil its function or not. Regarding this, the pin-on-disc test and micro-abrasion are two techniques which allow a good assessment of the abrasive wear resistance of a certain surface. The first one is a standard tribological test used to measure the friction coefficient and wear rate, where a disc spins at a controlled speed and a pin is pressed against it at a constant normal load. This load generates continuous sliding in a circular trajectory, and the tangential friction force is measured during the test. Finally, the wear volume is measured through the mass loss, track profiling and microscopy [49]. On the other hand, the second replaces the pin by a rotative sphere, rotating on the sample and forming a microscopic spherical crater. By measuring its diameter, the removed volume is possible to be determined and, thus, the same happens to the suffered wear. The advantage of this test is that it allows a wear measurement without destroying the coating completely [50].

### 3. Characterization of Machining Tool Coatings

The most common tool materials for FML machining are cemented carbides, such as the tungsten carbide, followed by high-speed steels (HSS) [51]; however, tools deteriorate quite fast with the multi-material's abrasive nature, which is why a coating is usually applied on them [52]. The mostly used tool coatings for this purpose, mainly in the drilling process, are also addressed in the literature [51,53]. Diamond-like carbon (DLC) coatings have been widely adopted in the difficult-to-machine materials due to their enhanced abilities, such as high hardness and wear resistance [9,52]. AlTiN/TiAlN-, TiN- and TiAlN-coated drills are also often chosen, with the first two achieving a higher hole circularity and lower surface roughness [54–56]. Additionally, it is also possible to use a nanocomposite coated drill, reducing the cutting forces and the surface roughness of the holes remarkably [57].

The PVD coating technique consists of a film deposition process where the coating grows on the substrate one atom at a time. Here, the solid source is physically vaporized by heating or an electron beam, being later deposited on the substrate. With PVD, the films can range from being as thin as some atomic layers to several microns. Although this uses lower temperatures and is a cleaner process, since it has fewer chemical reactions, it requires high vacuum equipment and only shows a good adhesion for non-complex geometries, in which case the adhesion fails [55]. Some examples of this are the thermal evaporation, electron beam evaporation and sputtering. In the latter, a solid target is bombarded using a plasma, usually argon, and the ejected atoms are deposited on the substrate. Specifically, the high power impulse magnetron sputtering (HiPIMS) process emerged to overcome excessive compressive residual stresses which led to film failure. It allows a strong ionization fraction and higher sputtering energy, which tailors the coating's microstructure, making it stronger [20,58].

On the other hand, the CVD process undergoes a high vacuum and provides a solid, high quality and high resistance coating layer on any substrate. It is usually used in mechanical parts in constant contact, which need protection against corrosion and wear. In this technique, the substrate is exposed to a set of volatile material precursors, and, through a chemical reaction, a deposition layer is created on the material's surface. Although the CVD process demands higher temperatures and the use of toxic gases, it is best employed for the deposition of diamond in drilling tools, more precisely considering its mono (MCD), nano (NCD) and polycrystalline (PCD) microstructures, since it has an excellent uniformity, high purity and adheres better to complex geometries [59].

In spite of multi-material machining papers being present in the literature, the tool coatings characterization is rarely used in studies performed to this kind of material. Because of this, the characterization techniques of the

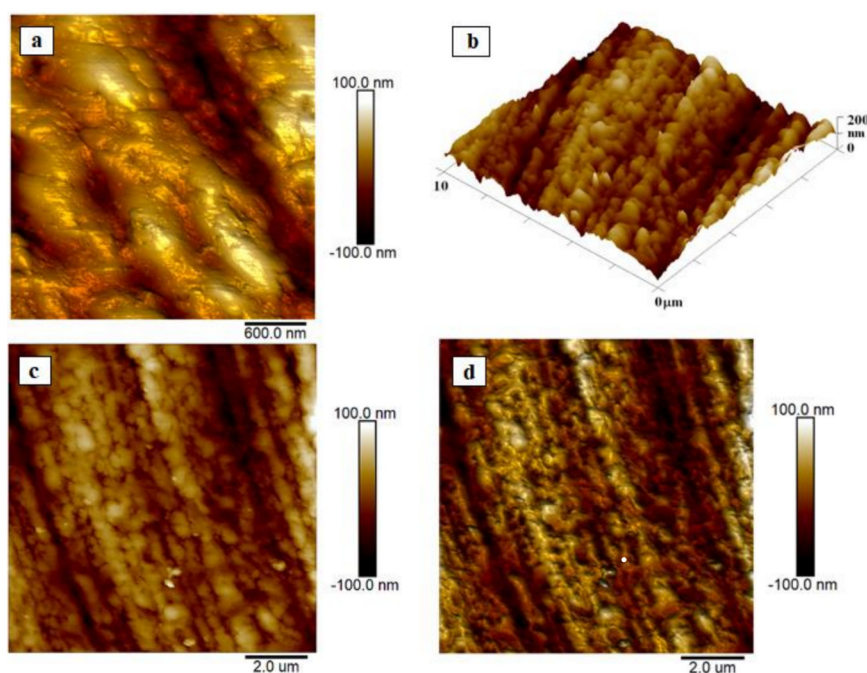
coatings present in this paper are explained based almost entirely in metal machining, but the coatings in study are also used in a multi-material machining process. In this chapter, the characterization methods were divided into three groups regarding their nature: physical (AFM, SEM, XRD and Raman spectroscopy), mechanical (nano/microindentation and scratch test) and wear (pin-on-disc test and micro abrasion). Each subchapter describes one group and contains the state-of-the art of its characterization methods. At the end, a SWOT analysis was performed, highlighting each group's strengths, weaknesses, opportunities and threats. In the case of the physical characterization methods, some studies are presented where the techniques are applied in the context of fibre metal laminates machining tests, but instead of performing these analysis on the machining tools, they are carried out in the multi-material itself, to analyse its quality after machining, namely to identify defects and observe the metal-composite interface damage.

### 3.1. Physical Characterization Methods

#### 3.1.1. Atomic Force Microscopy (AFM)

The AFM method makes it possible to observe surfaces atomically through the proper magnification.

Jahan et al. [60] analysed a polycarbonate micro-milling surface finish with TiAlN-coated carbide tools through AFM and concluded that the average surface roughness ( $R_a$ ) was 3.31 nm with a standard deviation of 0.5 nm, demonstrating that this process is able to produce a very smooth surface finish on polycarbonate, an important requirement for microfluidic applications. Kong et al. [61] performed vibration-assisted nanomachining tests (with ultrasonic excitation) to monocrystalline silicium (Si) with a diamond-coated tip, widely used in nanomachining studies due to its well characterized fragile-ductile behaviour. The purpose of this coating was to increase hardness and wear resistance. The authors did an AFM analysis both to characterize the produced channels morphology and to examine the tip's geometry after the test, allowing to quantify the increment in the tip radius and correlate it with the wear mechanisms. The vibration-assisted nanomachining results were then compared with the conventional mode to evaluate the tip's wear in both cases. Zhang et al. [62] used the contact mode AFM to characterize in detail the surface morphology and roughness of the  $WSi_2$  coatings deposited in tungsten substrates. The AFM images confirmed a compact surface without relevant macroscopic defects, as the coating presented a well-defined granular morphology with relatively uniform distributed grains. Michna et al. [63] used the HiPIMS magnetron sputtering technique to apply a TiAlN composite microlayer coating on the surface of machining tools and applied the AFM method for a microstructural characterization and visualization of the coating surface structures. Figure 1 represents the surface morphology and roughness analysis, in addition to a 3D map of the coating's roughness pattern.



**Figure 1.** AFM analysis of the surface morphology and roughness: (a) tool analysis, (b) tool 3D map and (c,d) tool detailed analysis [63].

Karthikeyan et al. [64] performed EDM machining tests to metal hybrid fibre laminates (MHFL) varying several parameters to evaluate their influence on the material removal rate and post-process surface roughness. In this context, they employed the AFM to carry out the micro-analysis of the MHFL's machined surface.

### 3.1.2. Scanning Electron Microscopy (SEM)

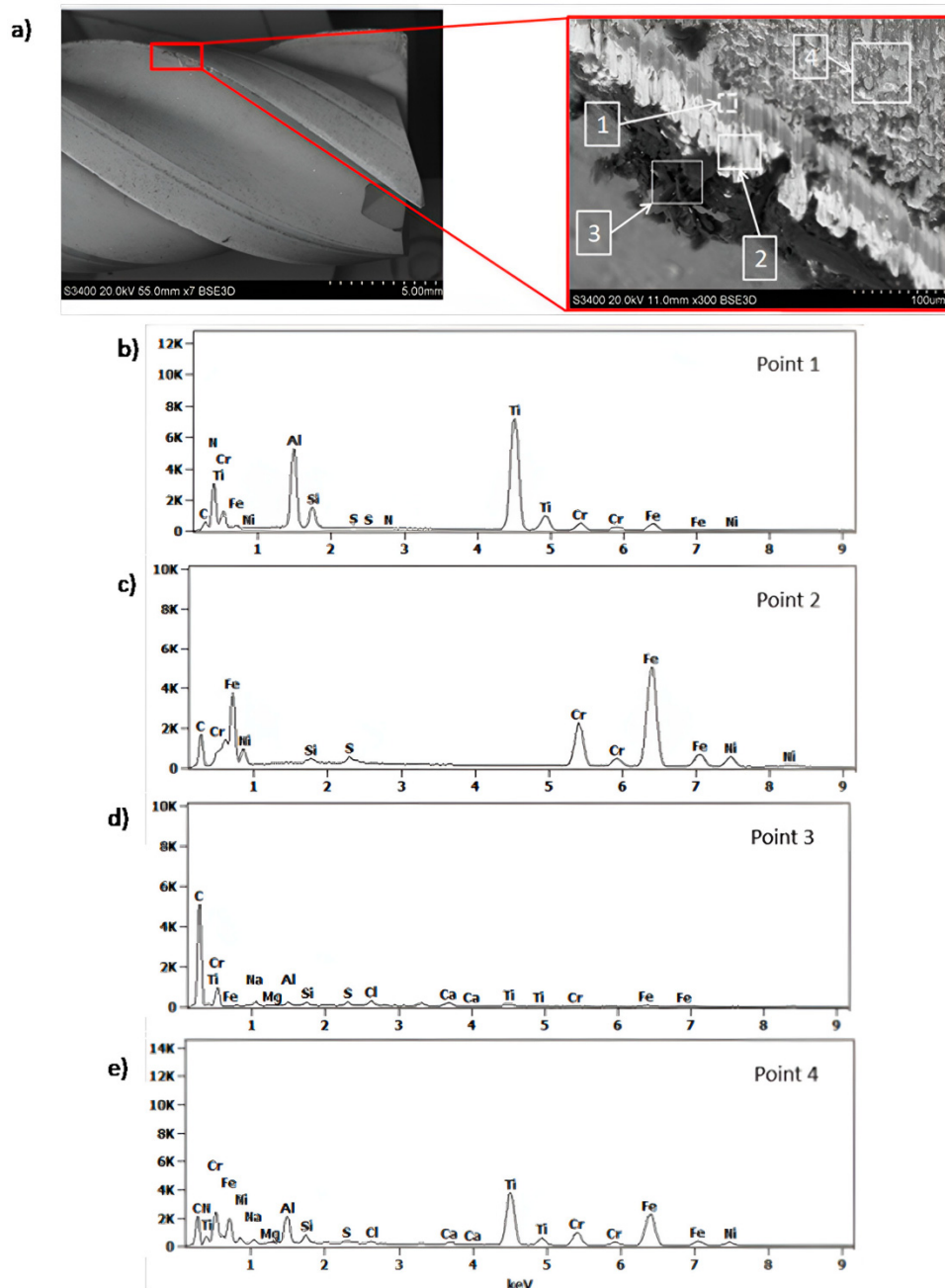
The SEM provides multiple information regarding a tool and its coating's state after a machining operation, making it possible to analyse two different images: the normal one, showing the backscattered electrons, which are the contrast according to the atomic number of the elements present on its surface, and the 3D, showing the secondary electrons, highlighting the surface's topography. In addition, some equipment also include the EDS function, electronic dispersive spectroscopy, where it generates a graph containing the amount of several chemical elements in a determined region of the image. From here, it is possible to notice the absence of a certain element when the coating has been completely removed, or the presence of a new element in a region which suffered adhesive wear, for example.

The literature contemplates several studies conducting similar analyses, as this is one of the most common characterization methods: Sousa et al. [65] used the SEM after milling operations in tool steel to assess the wear behaviour of TiAlSiN and TiAlN PVD coatings. Similarly, Montazeri et al. [66] did a SEM/EDS analysis to observe the tool wear morphology and identify the elements present in the damaged regions after turning Inconel 718 with carbide inserts coated a TiAlN PVD coating. The SEM showed a lower chipping occurrence and more stable wear in the tool with the ultra-soft coating, and the EDS confirmed an adhesion of material from the substrate (Ni, Fe and Cr) in the contact regions, which helped to explain the predominant wear mechanisms by adhesion and oxidation. Vats et al. [67] also detected traces of Ni, Fe and Cr in the tool resorting to the SEM/EDS after the turning of an additively manufactured nickel super alloy with TiAlN/AlTiN-coated WC-Co inserts. The analysis showed a predominantly adhesive and oxidative wear and the MQL use reduced the adhered layer forming and the wear severity in comparison with the dry cutting. Bolar et al. [68] drilled carbon-reinforced aluminium laminates (CARALL) with TiAlN-coated drills and used the SEM to analyse both the drills and the multi-material's final quality. They observed an aluminium built-up edge on the drill and several defects on the FML, such as partial fibre exposure, matrix degradation, fibre pull-out and metallic material smearing. Liang et al. [69] performed turning tests in a nickel-based alloy GH4169 using PVD-TiAlN coated carbide tools and analysed the coatings resorting to several techniques, one of which was the SEM/EDS, where they were able to identify the most notorious wear defects present on the tool's surface. Tymczyszyn et al. [70] milled a 316L stainless steel with PVD TiN-AlTiN-AlTiN/Si<sub>3</sub>N<sub>4</sub> nanocomposite coated tools using air cooling and resorted to the SEM/EDS to analyse the tool's surface, as present in Figure 2.

The SEM image shows the coating's elements in point 1, with the prominence of titanium (Ti) and aluminium (Al), the coating's absence with the substrate's elements, mainly iron (Fe) and Chromium (Cr) in point 2, a dark built-up with a peak of carbon (3) resultant from a overheating due to high cutting temperatures, in point 3, and a built-up edge with adhered material in point 4.

On the other hand, Sebbe et al. [71] grinded cork using different coatings and performed a SEM/EDS analysis after the tests to evaluate their wear. The SEM evidenced a predominantly abrasive wear, with parallel grooves, fragile fracture regions and coating delamination, whereas the EDS chemical microanalysis confirmed the presence of W from the substrate, Cr, Ni and Si from the coating, and C and O from the cork, indicating a combination of abrasive and adhesive wear.

In addition, Zhang et al. [72] studied the evolution of a TiC/Ti6Al4V composite coating's microstructure synthesized in-situ using the EBSD technique. They observed a decreasing medium grain size in the  $\alpha$ -Ti phase of the coating, explained due to a restricted grain growth of the TiC particles.

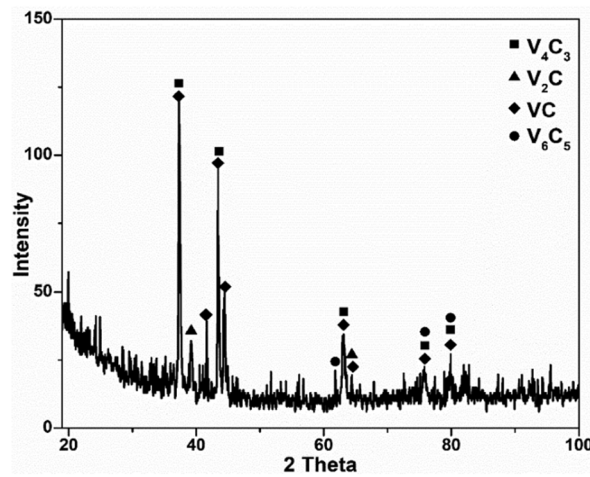


**Figure 2.** (a) SEM image of the multilayer coating after 120 min of cutting, (b–e) EDS spectra of the selected points [70].

### 3.1.3. X-ray Diffraction (XRD)

Bhandarkar et al. [73] used the XRD in TiAlN/AlCrN coated tools for the turning of the AISI 52100 alloy, confirming the presence of cubical crystalline phases typical of the coating's hard nitrides without significant formation of new phases after machining, which indicated a good structural stability of the coating under the studied cutting conditions. Bayraktar and Hekimoglu [74] performed a XRD analysis to Al-(5-35)Zn alloys, which confirmed the presence of the  $\alpha$ -Al phase as a main matrix and the progressive increment of the Zn-rich phases (such as the  $\eta$ -Zn) fraction with the increment of zinc content, evidencing microstructural changes which directly influence the alloys' hardness and machining behaviour. After milling a powder metallurgy nickel-based super alloy using a set of hard coatings (TiAlN, AlTiN and AlCrN), Zhang et al. [75] observed, through XRD, the presence of the  $\gamma$ -Ni matrix with typical hardening phases (such as  $\gamma'/\gamma''$  and carbides). The characterization of these coatings also put the nitrides cubic phases in evidence, showing in a lack of significant structural transformation after milling, which indicates a good coating thermal stability during the process. Hovoron et al. [76] used the XRD to characterize Ti-Al-Si-N coatings and this analysis showed that they presented a predominant cubic structure of the NaCl type based on (Ti, Al)N with slightly displaced peaks due to the Al and Si incorporation. Furthermore, the analysis highlighted the possible formation of amorphous phases rich in  $\text{Si}_3\text{N}_4$ , indicating grain

refinement and the improvement of the coating's mechanical properties. In a study conducted by Elhelaly et al. [77], the XRD technique was employed in vanadium carbide (VC) coatings on cold work tool steel AISI D3 to identify the different formed phases in the coating and confirm the formation of VC obtained by the thermo-reactive deposition/diffusion (TRD) technique. Figure 3 shows the XRD graph with the coating's surface analysis, making it possible to clearly identify the phases  $V_4C_3$  (hexagonal, R-3m),  $V_2C$  (orthorhombic, Pbcn), VC (cubic, Fm-3m) and  $V_6C_5$  (hexagonal, P31).



**Figure 3.** XRD diffractogram of the VC coating's surface at 1000 °C for 4 h [77].

The XRD diffractogram showed characteristic VC peaks, with a cubic crystalline structure. The typical VC reflections confirmed that the formed coating was predominantly monophasic, and the sharpness and intensity of the peaks indicate that the coating presents good crystallinity. Through this analysis, it was confirmed that the TRD process led to the formation of a homogeneous crystalline VC coating which a good adhesion to the tool steel and without the evidence of unwanted phases.

Oppositely, Sousa et al. [78] performed an energy dispersive X-ray spectroscopy microanalysis, where the X-ray is used as a complement to the SEM to measure the DLC/CrN coating thickness and identify phases, contrarily to the XRD, which determines the presence and number of mineral species in a sample. The XRD technique is also commonly used in the evaluation of the multi-material itself, as is the case of the study performed by Wu et al. [79], where they measured the fibre-metal interface residual stresses and analyse their distribution in the material after its drilling process.

#### 3.1.4. Raman Spectroscopy

Regarding spectroscopic techniques, Denkmann et al. [80] used the Raman spectroscopy in PVD TiAlN coatings of WC-Co tools used for turning the AISI 1045 hardened steel. This technique allowed to map the residual stresses in the coating through the deviation observed in the TiAlN's characteristic peak, allowing to correlate the spectral variation with the flank wear width and the stress state induced by the machining process. Celik et al. [81] also performed turning tests, but to tool steel and resorting to a multilayer CVD TiCN/Al<sub>2</sub>O<sub>3</sub> coating. In their study, the Raman spectroscopy was used to analyse chemical and structural changes of the coating in worn regions, evidencing oxidative transformations, layer degradation and progressive exposition of the sublayers as the wear evolved. Lukin et al. [82], for example, used the Raman spectroscopy not only to monitor the transformation of the crystalline phases during the ball milling process, but also to measure the chemical reactions progression through the evolution and disappearing of the vibrational peaks' characteristic of the reagents and chemical products formed during grinding. Furthermore, Wang et al. [83] developed a process of chemo-mechanical abrasive flow machining to polish thin diamond coatings (MCD and NCD) deposited in WC-Co substrates and used the Raman spectroscopy to evaluate the structural quality of a diamond film, the presence of non-diamond phases (carbon sp<sup>2</sup>) and possible structural damage after the polishing. Cygan-Baczek et al. [84] uses different thermal treatments to metal matrix composites used as diamond tool coatings and used the Raman spectroscopy to characterize the coatings, as seen in Figure 4.

From this analysis, the Raman spectre shows an intense peak at ~1332 cm<sup>-1</sup>, characteristic of diamond (carbon sp<sup>3</sup>) and two less intense peaks at around ~1420 and ~3125 cm<sup>-1</sup>, which can be attributed to fluorescence bands of nitrogen-vacancy centers in the diamond lattice. Wang et al. [85] also used the same method to evaluate the structural integrity of diamond coatings (MCD, SMCD, NCD and MCD/NCD), showing that the diamond

characteristic peaks (around  $1332\text{ cm}^{-1}$ ) stayed well defined, allowing to correlate the observed tool wear with surface changes and possible carbon  $\text{sp}^2$  changes in the worn regions. Furthermore, Visuvamithiran et al. [86] used the Raman spectroscopy to analyse a CFRP surface quality after drilling tests, evaluating its machining induced damages and material residual stresses through the Raman band deviations.

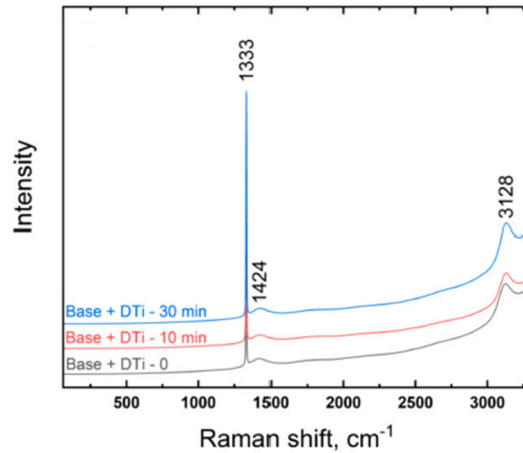


Figure 4. Raman spectre of the diamond graphitization test [84].

Figure 5 shows the SWOT analysis performed to the physical characterization methods, considering the AFM, SEM, XRD and Raman spectroscopy techniques applied to the coatings.



Figure 5. SWOT analysis of the coatings' physical characterization methods.

This analysis makes it clear that the mentioned microscopic, diffraction and spectroscopic techniques provide detailed information regarding the coatings' structure and composition, besides being able to aggregate with other micro-analysis techniques to be more complete. Nevertheless, but they require complex equipment and preparation, are limited and may be surpassed by other emerging techniques. Furthermore, they lack a direct correlation with the mechanical and tribological performance, as they only show the coating's aspect and chemical composition.

### 3.2. Mechanical Characterization Methods

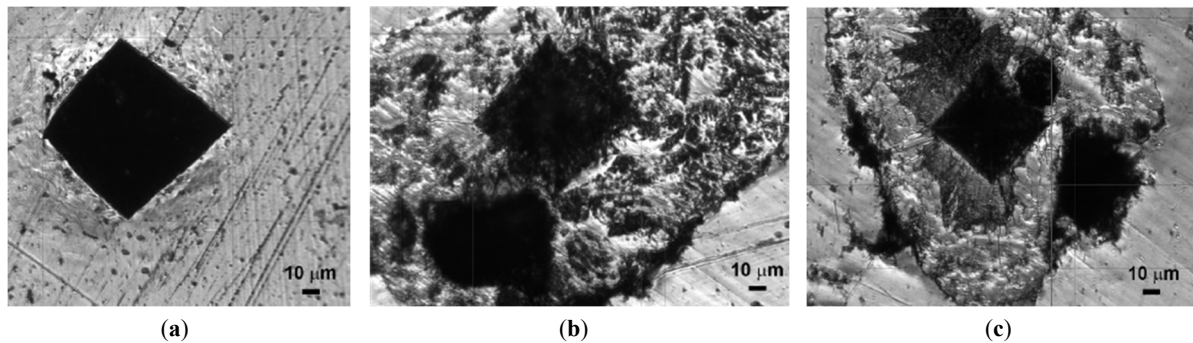
#### 3.2.1. Nano/Microindentation

The nano and micro indentation are two variants of a technique employed to evaluate the hardness of a certain material through the means of an indentation. The main difference between them stands in the scale of the applied load and the penetration depth.

Xiong et al. [87] measured the mechanical properties of diamond films deposited on tungsten carbide and cobalt (WC-Co) substrates with nanoindentation using a Berkovich indenter possessing a radius of 20 nm. Several loads (10, 20, 30, 50, 100 and 150 mN) with 5 or more positions for every load were employed. When the stiffness of the indenter reached 200 N/m, the system associated the location to the surface, subsequently indenting it with

an increasing load up until the maximum in 15 s and, afterwards, keeping this value for 10 s in order to measure thermal drift, finishing with the sample withdrawal in the same unload rates as the loading process. They concluded that the deposited diamond films' mechanical properties improved with the increment in the pressure, mainly hardness.

Yuan et al. [88] used the nanoindentation test to evaluate the mechanical properties of the surface and transverse section of WC-12Ni coatings obtained through thermal spray high velocity oxygen fuel (HFOV) in a small scale. They applied a very low load (in the order of hundreds of mN) with a diamond Berkovich indenter, registering the load-displacement curve to extract the hardness (nano-hardness) and the Young's modulus. Afterwards, they measured the penetration depths and compared these properties between the grain deposition temperature during the spray, correlating the mechanical changes with microstructure changes, such as the porosity. Mamalis et al. [89] studied the properties of a composite diamond coating reinforced with a Fe-Ni-Cu-Sn containing different contents of a nano-VN (vanadium nitride) additive and used the nanoindentation technique through a Berkovich type diamond indenter to measure the respective difference in hardness, as shown in Figure 6.



**Figure 6.** SEM image of an indentation made in the coating's surface with different contents of nano-VN additive: (a) 0%, (b) 4% and (c) 10% [89].

The observation of the images makes it possible to conclude that the increment of the nano-VN content in the coating directly translates into its hardness increase, since the indentation mark is smaller and its borders better defined, due to a lower plastic deformation.

On the other hand, at a bigger scale, the micro indentation makes it possible to analyse the microhardness, which both informs regarding a coating's wear performance and quantifies its hardness loss during the aggressive conditions of the machining process. Micro-indentation analyses were performed by Elias et al. [90] in a WC-Co tool's surface to generate texturized patterns while simultaneously measuring the grooves' depth and width to characterize the precision and repeatability of the texturization process. They found that micro-indentation allows the creation of regular and controlled textures on the tool's surface, which may influence positively the micro-turning behaviour by reducing friction and improving the chip evacuation without compromising the tool's cutting edge. In Rashid et al. [91]'s study, the micro-indentation technique is used in a nanocomposite coating to measure the hardness and elastic modulus in micrometric scale, evaluating the uniformity and local deformation resistance under applied loads. This test determined that the nanocomposite coating possessed a high hardness and consistent elastic modulus, providing higher deformation and wear resistance, which therefore translates into a higher tool useful life and better performance in machining under severe conditions. Trejo et al. [92] conducted micro-indentation tests in Stellite and hard chromium layers applied in saw blades to compare their deformation and abrasion resistance. They concluded that the first coating showed a higher hardness and deformation resistance compared to the second, indicating better load support capacity and higher durability during wood cut.

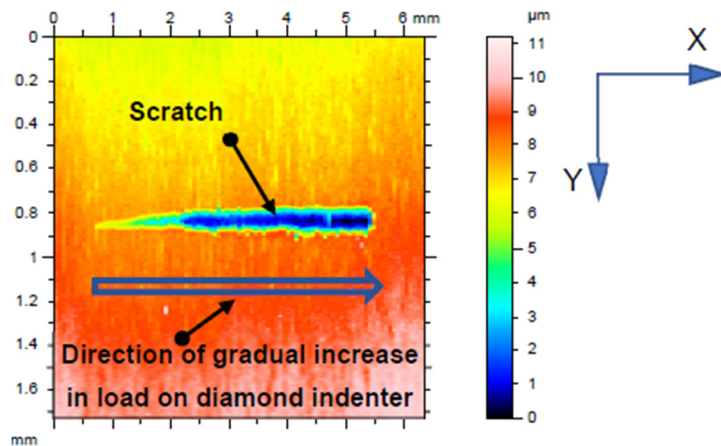
### 3.2.2. Scratch Test

The scratch test, in parallel with the already mentioned methods, is also crucial, since it assesses the adhesion of a coating to its substrate and, as such, can be the difference between a proper cut and a defective part.

Casais et al. [93] evaluated the adhesion of B<sub>4</sub>C and TiB<sub>2</sub> monolayered PVD coatings resorting to the scratch test. Firstly, they made six tests on each coating to increase accuracy and failure mode validation purposes, which made it possible to identify both adhesive and cohesive failures in all the coatings with the expected values. Siddiqui et al. [94] performed a tri-dimensional scratch test where a diamond penetrator covered the coated surface under progressive loads, registering deformations, microcracks and coating delamination to investigate the damage mechanisms in CrN and DLC using different thermal treatments of the X42Cr13/W steel. The results showed that the DLC coatings had a higher resistance to the abrasive wear and lower delamination comparing to the CrN,

whereas the substrate duplex treatment increase the coating’s cohesion, highlighting that the combination of the coating and pre-thermal treatment’s material strongly controls the damage mode and extension during the scratch test. Vopát et al. [95] applied this analysis to AlCrSiN coated inserts under controlled loads to analyse microcracks propagation, delamination and coating detachment, and saw that the coating’s adhesion to the substrate improved with the better edge preparation and a more uniform surface finishing, resulting in a lower delamination occurrence and higher wear resistance during the subsequent machining operations.

Łępicka [96] subjected samples of AISI 440B stainless steel coated with titanium nitride (TiN) to a scratch test with progressive load, where a diamond penetrator swifts the coating’s surface while the normal force increases in a controlled form to promote failures in the film. After the test, the scratch paths were examined using SEM/FIB microscopy in a transversal cut so that the authors could observe directly the damage mechanisms, such as microcracks, delamination and coating failures. Kupczyk and Józwick [97] analysed the laser heating effect on different machining tool coatings and used the scratch test to evaluate their adhesion to the substrate by measuring the critical load at which the coating would peel off the substrate. Figure 7 contains an example image of aa scratch test mapped with a profilometer.



**Figure 7.** Scratch test caused by gradual increase in load on diamond indenter (top view: load increase from left to right) [97].

Additionally, innovating approaches in the scratch test often lead to better results. Storchak et al. [98] applied an advanced multi-pass scratch test method, where a diamond penetrator quickly travels the same trail under a controlled load, which can be constant or progressive, allowing to evaluate the coating’s cohesive resistance and its adhesion to the substrate through a progressive damage accumulation. The authors found that this multi-pass method allows to identify in a more sensitive way the coating’s failure modes, such as microcracking, delamination and substrate exposition, and also to better distinguish the contact fatigue resistance of the different coatings comparing to the conventional single-pass scratch test.

Figure 8 represents the SWOT analysis regarding the mechanical characterization methods, referring to the coatings’ nano/microindentation and scratch test techniques.



**Figure 8.** SWOT analysis of the coatings’ mechanical characterization methods.

This analysis shows that the mechanical characterization techniques, in spite of precisely quantifying the coating's properties, a crucial aspect of their evaluation, and having a broad range of applications, are still limited if the wear patterns are not known. The hardness of a coating only provides part of the data needed to understand the coating's expected performance. In addition, the equipment is generally sensitive and expensive.

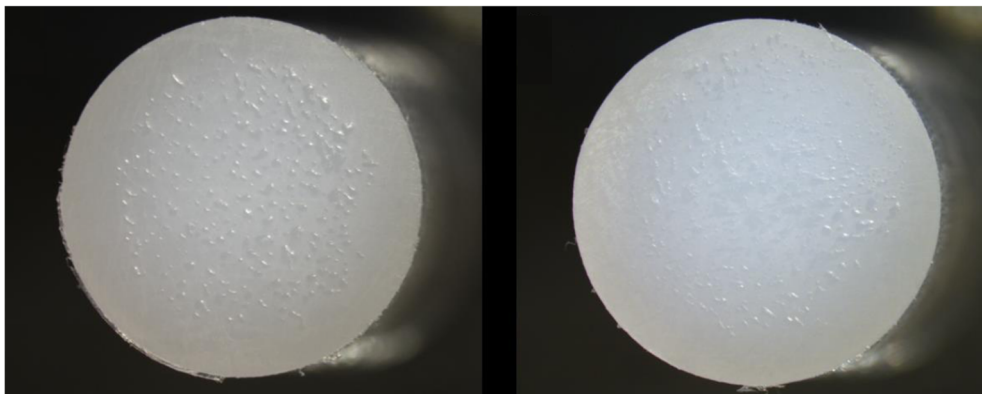
### 3.3. Wear Characterization Methods

#### 3.3.1. Pin-on-Disc Test

In the machining subject, it is not possible to address the performance of tools and respective coatings without mentioning their wear resistance, since this is the most important parameter which will provide information on the effectiveness of a determined coating for each application. The pin-on-disc is one of the most used tests to evaluate this parameter and its main purpose is to determine a material's wear resistance.

Ba et al. [99] investigated the influence that skewness parameters had on the surface of a steel machining process, namely in the turning and milling components, in the pin-on-disc sliding wear test. This allowed the observation of different behaviours in the obtained friction curves, and it was concluded that the orientation of the feed marks in comparison to the sliding direction is able to influence the surfaces' tribological behaviour. On the other hand, Osmond et al. [100] used the same test to determine the tribological properties of multilayer CVD coatings which were deposited on Si-AlON ceramic milling inserts. The results showed that the TiN coating possessed a great wear and abrasion resistance, as the steel balls in this test had lower frictional interactions. In addition, Sivaprakasam et al. [101] performed a pin-on-disc test where a Ti-6Al-4V Grade 23 pin slid against one disc under different loads and sliding speeds, measuring both the friction coefficient and the wear rate over time. The results showed that the load and speed increment increased the wear rate and the friction coefficient, with predominance of adhesive and abrasive wear mechanisms, in addition to the formation of surface oxides which influenced the alloy's tribological behaviour.

Strel'nitskji et al. [102] performed this test to evaluate the wear resistance of DCL films applied in CoCrMo discs, comparing two coating types (with and without Ti intermediate layer). After 2.5 million cycles (Mc), it was possible to compare the wear performance and friction behaviour between the different samples and estimate the protection they provide against wear when they are in contact with polyethylene. Figure 9 shows two examples of the coatings after the tests, with raised, round and bumps on the surface of the pins.



**Figure 9.** Detail on the pins' surface after 2.5 Mc of testing with a 10× magnification [102].

Some authors investigate the change of the pin-on-disc test parameters to understand their effect on the performed analyses. This is the case of Yadav et al. [103], which carried out pin-on-disc tribological tests using different geometries and pin orientations to evaluate the coating's friction coefficient and the wear behaviour. For this, they used a nickel-coated EN8 steel pin against an Al6061 alloy disc, and the results showed that the nickel coating significantly reduced the friction coefficient and the wear rate in comparison to the uncoated steel. Moreover, the pin's geometry and orientation influenced the formation of the transferring film in the Al6061 disc, changing the predominant mechanisms between adhesive and abrasive wear, thus directly affecting the friction stability.

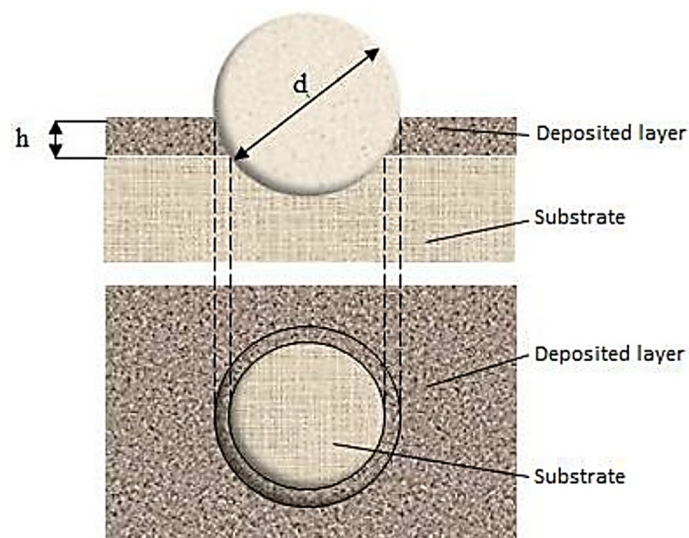
#### 3.3.2. Micro-Abrasion

Besides being appropriate for evaluating the abrasive wear resistance of a given surface, the micro-abrasion technique can also provide information on thin film/substrate interfacial resistance, i.e., film adhesion, although this last evaluation is mostly made by the scratch test.

Silva et al. [104] used the ball cratering micro-abrasion test to study the wear behaviour of  $B_4C$  monolayered and CrN/CrC/DLC multi-layered coatings under high contact loads using a rotating sphere sliding over the coated samples, using abrasive suspension to create wear craters and allow to determine the material's removal rate. With this test, they concluded that the CrN/CrC/DLC multi-layered coating showed a lower wear rate and higher resistance under high loads comparing to the  $B_4C$  monolayered coating, owing to its layered architecture, which favours stress dissipation and higher crack propagation resistance during micro-abrasion. Mundotia et al. [105] conducted exactly the same test, but using TiN coatings previously subjected to ionic bombarding and micro-blasting to evaluate the influence of those treatments in the wear rate on localized contact. The results showed that the ionic bombardment improved the TiN densification and adhesion, therefore reducing its wear rate, whereas the micro-blasting changed the coating's roughness and surface tensions, so it may either increase or reduce the coating's wear rate depending on the applied pre-treatment conditions. On the other hand, Esteves et al. [106] used this test, but varied the distribution of the abrasive particles' size in the suspension and also the sphere's material to assess the influence of these parameters in the wear coefficient at a microscopic scale. After conducting the micro-abrasion tests, they observed that bigger abrasive particles and wider distributions increase the wear coefficient due to the higher severity of the abrasive mechanism. Furthermore, the sphere's material influences the wear regime, significantly altering the material's removal rate.

Çay [107] performed fixed ball micro-abrasion tests to analyse the AlCrN and AlTiN PVD coatings deposited on a High-Speed Steel (HSS) substrate and the respective wear behaviours. On the other hand, the wear behaviour of AlCrN and AlTiN coatings via PVD on a High-Speed Steel (HSS) was studied by fix ball micro-abrasion tests. Here, loads of 0.5, 1 and 1.5 N were applied during 60 and 180 s, as well as two different kinds of abrasives: alumina ( $Al_2O_3$ ) and silicon carbide (SiC), each one with two different particle sizes. It was concluded that the AlCrN coatings exhibited better wear resistance than the AlTiN ones, and the wear was higher for the SiC abrasive particles and for higher particle size, number of cycles and the applied load, which had a significant effect on the wear mechanisms. Some authors also study the optimization of the micro-abrasion wear test, for example on the influence of the ball surface texture in the dragging of abrasive particles [50]. This can be useful to guarantee the effectiveness of this test and that the achieved results are as correct as possible.

Mariani et al. [108] investigated the production of a niobium carbide (NbC) layered coating on a grey cast iron surface using thermo-reactive treatments (powder diffusion and liquid bath), characterizing them using micro-abrasion tests to evaluate how the coating's layers influenced the material's tribological behaviour. Using a sphere in contact with the coated surface under controlled loads to simulate abrasive wear mechanisms, the wear volume loss and wear rate were measured. They concluded that the NbC layers showed a wear resistance 2 to 12 times higher than the uncoated cast iron. Chisiu et al. [109] compared two different coatings, one obtained through thermal spray and the other through hard chromium, regarding their abrasive wear behaviour. For this, they resorted to the micro-abrasion technique using the ball cratering test, as shown in Figure 10. This method used a rotative sphere with an abrasive suspension and made it possible to observe the transition between rolling and sliding wear of the particles influenced by load, abrasive concentration and coating properties. They concluded that the thermal sprayed coatings may present an equivalent or better adhesion behaviour depending on their microstructure and hardness.



**Figure 10.** Micro-abrasion test diagram representation [109].

Figure 11 depicts the SWOT analysis corresponding to the wear characterization methods, namely the pin-on-disc and micro-abrasion tests.



**Figure 11.** SWOT analysis of the coatings' wear characterization methods.

The wear characterization methods allow a proper measurement of the coatings' behaviour in real applications, making it possible to adjust parameters and develop an effective coating based on their results. Nonetheless, they are also sensitive to the testing conditions, so valuable data can only be acquired from them if these tests are performed with the exact correct parameters, added to the materials and equipment costs.

### 3.4. Summary and Applicable Standards

A coating's characterization is usually not enough by using just a single method, but rather a combination of different methods which will provide a complete analysis of its properties, which is why in the literature many authors use several techniques to characterize their coatings. Table 1 presents every study addressed in the state-of-the-art of this paper, along with all the characterization techniques used in each one and the respective key results resultant from their utilization in the respective applications.

**Table 1.** Characterization techniques used and their application in the state-of-the-art studies.

Study	Characterization Techniques Used	Application
[60]	AFM, SEM/EDS	Identification of the tool wear mechanisms (abrasion/adhesion) and its relationship with surface finishing
[61]	AFM, SEM	Wear quantification with AFM; morphology and wear rate variation according to the assisted vibration
[62]	AFM, SEM/EDS, EBSD, XRD	Texture and crystallographic orientation analysis; correlation between crystalline structure and surface morphology
[63]	AFM, SEM/EDS, XRD, microindentation	Identification of the TiAlN structure; hardness and wear resistance evaluation
[64]	AFM, SEM/EDS	FML topography and surface roughness analysis with AFM after hybrid machining to observe its microstructure
[65]	SEM/EDS	Comparison between the wear mechanisms
[66]	SEM/EDS, XRD, nanoindentation, scratch test	Evaluation of the coating's resistance, adhesion and useful life
[67]	SEM/EDS, XRD	Evaluation of the surface wear and crystalline phases changes induced by the different sustainable lubrication methods used
[68]	SEM	Observation of coating's wear mechanisms and FML microstructure analysis to identify defects and failure modes
[69]	SEM/EDS, XRD	Identification of oxidation and diffusion wear; relationship between the coating's structure and the wear mechanisms
[70]	SEM/EDS	Comparison of wear mechanisms between a nanocomposite PVD coating and uncoated tools
[71]	SEM/EDS	Review of the main wear mechanisms in inserts and identification of solutions based in hard coatings

Table 1. Cont.

Study	Characterization Techniques Used	Application
[72]	SEM, EBSD	Microstructural evolution of the TiC/Ti6Al4V composite and confirmation of the formed phases in situ
[73]	SEM/EDS, XRD	Evaluation of the flank wear; correlation between cutting parameters and wear mechanisms
[74]	SEM, XRD	Influence of the Zn content in the coating's adhesion wear
[75]	SEM/EDS, EBSD, XRD, Raman spectroscopy	Coatings' comparative evaluation and identification of dominant wear mechanisms to be abrasion and adhesion
[76]	SEM, XRD, microindentation	Confirmation of the Ti-Al-Si-N phases; hardness and wear resistance evaluation
[77]	SEM/EDS, XRD, microindentation	Identification of the Vanadium Carbide morphology, chemical composition, crystalline phase formation and coating's hardness and thickness determination
[78]	SEM, XRD	Topography and wear mechanisms characterization, crystalline phases identification
[79]	XRD	Residual stresses measurement and distribution in the FML fibre-metal interface
[80]	Raman spectroscopy	Flank wear width monitoring and residual stress evaluation in the coating
[81]	SEM/EDS, Raman spectroscopy	Identification of the wear-induced structural transformations on the TiCN/Al <sub>2</sub> O <sub>3</sub> /TiN coating's layers
[82]	Raman spectroscopy	Real time monitoring of the mechanochemical reactions during the grinding process
[83]	SEM, Raman spectroscopy	Evaluation of the diamond coatings' polishing, surface roughness and surface integrity
[84]	SEM/EDS, XRD, Raman spectroscopy	Identification of the present phases and evaluation of structural changes on the diamond and the matrix after thermal treatment
[85]	SEM, Raman spectroscopy, nanoindentation	Hardness and wear resistance comparison between different diamond coatings
[86]	Raman spectroscopy	Evaluation of residual stresses and microstructural damage in the CFRP after drilling
[87]	AFM, SEM, XRD, Raman spectroscopy, nanoindentation	Hardness and Young's modulus determination in the diamond coatings
[88]	SEM, XRD, nano/microindentation	Correlation between the coating's deposition temperature and mechanical properties; hardness and lamellar cohesion variation
[89]	SEM/EDS, nanoindentation	Measurement of the coating's hardness and Young's modulus, evaluate the effect of the nano-VN composition in the deformation resistance
[90]	Microindentation	Surface texture evaluation and improvement in the lubricant retention
[91]	SEM/EDS, microindentation	Analysis of the nanocomposite's microstructure and evaluation of its hardness and plastic deformation resistance
[92]	SEM, microindentation	Hardness and wear resistance comparison between stellite and hard chromium
[93]	AFM, SEM/EDS, XRD, nanoindentation, scratch test	Coating's wear resistance evaluation under high loads
[94]	Microindentation, scratch test	Identification of delamination and cracking coating failure mechanisms when comparing CrN and DLC coatings
[95]	SEM, nanoindentation, scratch test	Influence of the tool's cutting edge and deposition voltage on the coatings' adhesion and nano hardness
[96]	SEM, scratch test	Precise detection of the failure critical load and transversal fracture analysis by FIB/SEM
[97]	SEM, scratch test	Evaluation of the coatings' adhesion and failure resistance after laser heating
[98]	Nanoindentation, scratch test	Evaluation of the coating's progressive resistance and failure critical load
[99]	SEM/EDS, pin-on-disc test	Correlation between roughness parameters and tribological behaviour
[100]	SEM, pin-on-disc test	Multilayer's interface morphology observation, friction coefficient and wear rate determination

Table 1. Cont.

Study	Characterization Techniques Used	Application
[101]	SEM/EDS, pin-on-disc test	Identification of the adhesive and abrasive wear; quantification of the friction coefficient
[102]	SEM, pin-on-disc test	Examination of the wear marks morphology and measurement of the coating's friction coefficient and durability under sliding conditions
[103]	SEM, XRD, pin-on-disc test	Wear morphology analysis, crystalline phases identification, and quantification of the pin geometry effect on the friction coefficient and mass loss
[104]	AFM, SEM/EDS, pin-on-disc, scratch test, micro-abrasion	Characterization of the nanometric roughness, wear mechanisms under high loads, friction coefficient, coating's adhesion to the substrate and resistance to abrasive wear
[105]	SEM/EDS, XRD, micro-abrasion	Analysis of the surface morphology after ionic bombardment, identification of the residual stresses in the crystalline phases and evaluation of the TiN coating's resistance to the abrasive particles wear
[106]	SEM, micro-abrasion	Correlation between the particle size and the wear coefficient, with the ball material influence
[107]	SEM, XRD, micro-abrasion	Characterization of the wear craters' morphology, identification of the crystalline phases and residual stresses of the AlCrN/AlTiN coatings and evaluation of the load influence and abrasive type in the high-speed steel wear rate
[108]	SEM, XRD, microindentation, micro-abrasion	NbC layers microstructure characterization and abrasive wear resistance evaluation
[109]	Micro-abrasion	Quantification of the coatings' wear resistance through the ball cratering method

Through Table 1's analysis, it is confirmed that almost every study in the chosen state-of-the-art papers use the combination of more than one characterization methods and the most used technique is the SEM, as it provides valuable information regarding the tools and respective coating's morphology, topology and chemical composition, making it possible to observe their surface in high detail. In spite of this, the XRD technique is also used very often, as it allows the coatings' atomic structure analysis.

Although these techniques can be applied in the majority of coatings, there are some limitations:

- The AFM functions better in relatively smooth surfaces;
- For a proper SEM analysis, isolating coatings may need a previous metallization procedure;
- Methods such as nanoindentation or XRD may not be fit for very thin coatings, since they may reach the substrate and this influences the results. Furthermore, XRD does not work well for amorphous materials, providing limited information;
- The Raman spectroscopy depends on the light-matter interaction and not every material responds well.

All the presented techniques are crucial for the determination of specific properties regarding the tool coatings for machining, as these will represent the decision for the use of a certain coating in detriment of another. The microscopy and spectroscopy methods are too broad for specific normalization to be attributed to, reason why no standards are associated with them; however, the mechanical and tribological tests should be performed according to specific standards, presented below:

- ASTM E2546-07—Standard Practice for Instrumented Indentation Testing [110];
- ASTM E384-17—Standard Test Method for Microindentation Hardness of Materials [111].
- ISO 20502:2005—Fine ceramics (advanced ceramics, advanced technical ceramics)—Determination of adhesion of ceramic coatings by scratch testing [112];
- ISO 18535:2016—Diamond-like carbon films—Determination of friction and wear characteristics of diamond-like carbon films by ball-on-disc method [113];
- ISO 26424:2008—Fine ceramics (advanced ceramics, advanced technical ceramics)—Determination of the abrasion resistance of coatings by a micro-scale abrasion test [114];

Table 2 presents an overview of the contributions provided by the characterization techniques addressed in this paper, as well as examples of standards applicable to each one.

**Table 2.** Contributions of the presented advanced characterization techniques.

Characterization Technique	Standard	Contribution
Atomic Force Microscopy (AFM)	-	Surface topography and morphology analysis at an atomic scale
Scanning Electron Microscopy (SEM)	-	Surface topography and morphology analysis at nano scale
X-ray Diffraction (XRD)	-	Crystallinity and phases evaluation, residual stresses determination
Raman Spectroscopy	-	Coating purity assessment
Indentation test	ASTM E2546-07	Hardness and stiffness quantification
Micro indentation	ASTM E384-17	Hardness and stiffness quantification
Scratch test	ISO 20502:2005	Coating adhesion and cohesion measurement
Pin-on-disc test	ISO 18535:2016	Surface wear evaluation
Micro-abrasion	ISO 26424:2008	Surface wear and friction evaluation

#### 4. Conclusions

No matter how developed tool coatings are, delamination still represents a major issue in this industry, since they start to fail when certain conditions are tested, making them unfeasible for a large scale production. Should the coating not possess the right characteristics for its intended role, a huge economic loss occurs for the company, resultant from the combined effect of the defective tool, defective material and time spent from a more frequent tool changing. Due to these factors, completely characterizing a coating is fundamental for a proper assessment of the machining tools performance, so that it may later be selected according to specific requirements, guaranteeing a higher tool lifespan and reducing costs and waste for the company, which also attains its sustainability goals.

The thin coatings characterization may be achieved using one or a combination of several techniques, each of them having specific properties. In the studies collected from the literature, through the state-of-the-art analysis, many authors opt to combine more than one method to assess the coating's quality, evaluating its chemical composition and reach a final decision regarding its fitness for the application in study. A SWOT analysis performed for each group of techniques (physical, mechanical and wear characterization methods) highlighted their strengths and opportunities, but also their weaknesses and threats, which confirmed the need to combine with other methods for a more effective characterization. From the studies gathered in the literature, the most commonly used methods for tool coatings physical characterization to obtain the topography of the coating's surface and the analysis of its morphology at an atomic and nano scale are the AFM and SEM, respectively. In order to assess the diamond crystallinity, for example, but also to evaluate the residual stresses formed during the creation of hard coatings, the XRD is widely used. The purity of the coating is also quantified resorting to the Raman spectroscopy. In addition to the already mentioned techniques, mechanical characterization methods are also usually performed, such as the nano and microindentation, for the coating's hardness identification, ensuring that it will resist the high stresses and solicitations arising throughout the machining process, as well as the scratch test, to measure its adhesion to the substrate. Finally, regarding wear characterization methods, the authors use the pin-on-disc test and the micro-abrasion with the purpose of evaluating the coating's surface wear after the machining tests are conducted. From all the mentioned techniques, the state-of-the-art table showed that the SEM is the mostly used throughout the different studies.

This joint set of analyses makes it possible to understand the effect which a determined coating will have on the daily operations of tool manufacturing company, positively contributing to improve the process and reduce waste and costs for this industry.

#### Author Contributions

R.D.F.S.C.: conceptualization; R.D.F.S.C. and S.L.S.S.: methodology; R.D.F.S.C.: data curation, visualization; R.D.F.S.C.: writing—original draft preparation; R.D.F.S.C.: investigation; A.M.P.J. and F.J.G.S.: supervision; S.L.S.S., T.E.F.S. and D.A.F.: validation; A.B. and G.F.P.: writing—reviewing and editing. All authors have read and agreed to the published version of the manuscript.

#### Funding

The authors gratefully acknowledge the “Fundação para a Ciência e a Tecnologia” (FCT) for the financial support through Grant Number 2024.05867.BDANA. The authors also acknowledge Project Hi-rEV—Recuperação do Setor de Componentes Automóveis (C644864375-0000002) cofinanced by Plano de Recuperação e Resiliência (PRR), República Portuguesa through NextGeneration EU.

### Institutional Review Board Statement

Not applicable.

### Informed Consent Statement

Not applicable.

### Data Availability Statement

Not applicable.

### Conflicts of Interest

The authors declare no conflict of interest. Given the role as Editor-in-Chief, Francisco J. G. Silva had no involvement in the peer review of this paper and had no access to information regarding its peer-review process. Full responsibility for the editorial process of this paper was delegated to another editor of the journal.

### Use of AI and AI-Assisted Technologies

No AI tools were utilized in this paper.

### References

1. Abrantes, I.; Ferreira, A.F.; Magalhães, L.B.; et al. The Impact of Revolutionary Aircraft Designs on Global Aviation Emissions. *Renew. Energy* **2024**, *223*, 119937. <https://doi.org/10.1016/j.renene.2024.119937>.
2. Blanco, D.; Rubio, E.M.; Marín, M.M.; et al. Advanced Materials and Multi-Materials Applied in Aeronautical and Automotive Fields: A Systematic Review Approach. *Procedia CIRP* **2021**, *99*, 196–201. <https://doi.org/10.1016/j.procir.2021.03.027>.
3. Doğan, M.A.; Yazman, Ş.; Gemi, L.; et al. A Review on Drilling of FML Stacks with Conventional and Unconventional Processing Methods under Different Conditions. *Compos. Struct.* **2022**, *297*, 115913. <https://doi.org/10.1016/j.compstruct.2022.115913>.
4. Giasin, K.; Atif, M.; Ma, Y.; et al. Machining GLARE Fibre Metal Laminates: A Comparative Study on Drilling Effect between Conventional and Ultrasonic-Assisted Drilling. *Int. J. Adv. Manuf. Technol.* **2022**, *121*, 3657–3672. <https://doi.org/10.1007/s00170-022-10297-x>.
5. Costa, R.D.F.S.; Sales-Contini, R.C.M.; Silva, F.J.G.; et al. A Critical Review on Fiber Metal Laminates (FML): From Manufacturing to Sustainable Processing. *Metals* **2023**, *13*, 638.
6. Rizzo, A.; Goel, S.; Grilli, M.L.; et al. The Critical Raw Materials in Cutting Tools for Machining Applications: A Review. *Materials* **2020**, *13*, 1377. <https://doi.org/10.3390/ma13061377>.
7. Hari Nath Reddy, R.; Alphonse, M.; Bupesh Raja, V.K.; et al. Evaluating the Wear Studies and Tool Characteristics of Coated and Uncoated HSS Drill Bit—A Review. *Mater. Today Proc.* **2020**, *46*, 3779–3785. <https://doi.org/10.1016/j.matpr.2021.02.022>.
8. Costa, R.D.F.S.; Moreira, R.D.F.; Silva, T.E.F.; et al. Study on Multi-Material Drilling and Defects Modelling Using a Fracture Mechanics Approach. *Procedia CIRP* **2025**, *131*, 119–124.
9. Franz, G.; Vantomme, P.; Hassan, M.H. A Review on Drilling of Multilayer Fiber-Reinforced Polymer Composites and Aluminum Stacks: Optimization of Strategies for Improving the Drilling Performance of Aerospace Assemblies. *Fibers* **2022**, *10*, 78. <https://doi.org/10.3390/fib10090078>.
10. Sobri, S.A.; Whitehead, D.; Mohamed, M.; et al. Augmentation of the Delamination Factor in Drilling of Carbon Fibre-Reinforced Polymer Composites (CFRP). *Polymers* **2020**, *12*, 2461. <https://doi.org/10.3390/polym12112461>.
11. Pai, A.; Kini, C.R.; Shenoy, B.S. Scope of Non-Conventional Machining Techniques for Fibre Metal Laminates: A Review. *Mater. Today Proc.* **2022**, *52*, 787–795. <https://doi.org/10.1016/j.matpr.2021.10.150>.
12. Giasin, K.; Dad, A.; Brousseau, E.; et al. The Effects of through Tool Cryogenic Machining on the Hole Quality in GLARE® Fibre Metal Laminates. *J. Manuf. Process.* **2021**, *64*, 996–1012. <https://doi.org/10.1016/j.jmapro.2021.02.010>.
13. Wachowicz, J.; Dembiczak, T.; Stradomski, G.; et al. Properties of WC-Co Composites Produced by the SPS Method Intended for Cutting Tools for Machining of Wood-Based Materials. *Materials* **2021**, *14*, 2618. <https://doi.org/10.3390/ma14102618>.
14. Kruzel, R.; Wachowicz, J.; Dembiczak, T.; et al. New Tools for the Processing of Wood Materials in the Construction Industry. *Materialy Budowlane* **2025**, *1*, 174–179. <https://doi.org/10.15199/33.2025.10.19>.
15. Wang, R.; Yang, D.; Wang, W.; et al. Tool Wear in Nickel-Based Superalloy Machining: An Overview. *Processes* **2022**, *10*, 2380.

16. Giasin, K.; Gorey, G.; Byrne, C.; et al. Effect of Machining Parameters and Cutting Tool Coating on Hole Quality in Dry Drilling of Fibre Metal Laminates. *Compos. Struct.* **2019**, *212*, 159–174. <https://doi.org/10.1016/j.compstruct.2019.01.023>.
17. He, Q.; Paiva, J.M.; Kohlscheen, J.; et al. An Integrative Approach to Coating/Carbide Substrate Design of CVD and PVD Coated Cutting Tools during the Machining of Austenitic Stainless Steel. *Ceram. Int.* **2020**, *46*, 5149–5158. <https://doi.org/10.1016/j.ceramint.2019.10.259>.
18. Chen, J.; Zhang, X.; Xue, Z.; et al. Multi-Material Stage-Specific Analysis and WSTVF Based Feature Engineering for Enhanced Tool Wear Monitoring in CFRP/Ti Stacks Drilling. *Mech. Syst. Signal Process.* **2025**, *234*, 112829. <https://doi.org/10.1016/j.ymsp.2025.112829>.
19. Aditharajan, A.; Radhika, N.; Saleh, B. Recent Advances and Challenges Associated with Thin Film Coatings of Cutting Tools: A Critical Review. *Trans. Inst. Met. Finish.* **2022**, *100*, 205–221.
20. Kumar, A.; Bauri, R.; Naskar, A.; et al. Characterization of HiPIMS and DCMS Deposited TiAlN Coatings and Machining Performance Evaluation in High Speed Dry Machining of Low and High Carbon Steel. *Surf. Coat. Technol.* **2021**, *417*, 127180. <https://doi.org/10.1016/j.surfcoat.2021.127180>.
21. Grzesik, W.; Nieslony, P.; Habrat, W.; et al. Flank Wear and Rake Wear Studies for Arc Enhanced HiPIMS Coated AlTiN Tools during High Speed Machining of Nickel-Based Superalloy. *Surf. Coat. Technol.* **2020**, *381*, 125190. <https://doi.org/10.1016/j.surfcoat.2019.125190>.
22. Costa, R.D.F.S.; Jesus, A.M.P.; Simões, S.L.S.; et al. Advanced Characterization Techniques of Multi-Material Machining Tool Coatings. In *Lecture Notes in Mechanical Engineering*; Springer: Berlin/Heidelberg, Germany, 2023; pp. 248–256.
23. Kang, K.; Su, S.; Yu, B.; et al. The Review and Prospect of Tool Coating Technology. *Int. J. Adv. Manuf. Technol.* **2025**, *137*, 3107–3139.
24. Ramezani, M.; Mohd Ripin, Z.; Pasang, T.; et al. Surface Engineering of Metals: Techniques, Characterizations and Applications. *Metals* **2023**, *13*, 1299.
25. Khan, S.U.; Jamshed, W. Finite Element Analysis and Wear Rate Analysis of Nano Coated High Speed Steel Tools for Industrial Application. *Babylon. J. Mech. Eng.* **2023**, *2023*, 12–19. <https://doi.org/10.58496/BJME/2023/002>.
26. Moganapriya, C.; Vigneshwaran, M.; Abbas, G.; et al. Technical Performance of Nano-Layered CNC Cutting Tool Inserts—An Extensive Review. *Mater. Today Proc.* **2021**, *45*, 663–669.
27. Wang, Q.; Jin, Z.; Zhao, Y.; et al. A Comparative Study on Tool Life and Wear of Uncoated and Coated Cutting Tools in Turning of Tungsten Heavy Alloys. *Wear* **2021**, *482–483*, 203929. <https://doi.org/10.1016/j.wear.2021.203929>.
28. Doddapaneni, S.; Kumar, S.; Sharma, S.; et al. Advancements in EBSD Techniques: A Comprehensive Review on Characterization of Composites and Metals, Sample Preparation, and Operational Parameters. *J. Compos. Sci.* **2025**, *9*, 132.
29. Al-Tameemi, H.A.; Al-Dulaimi, T.; Awe, M.O.; et al. Evaluation of Cutting-Tool Coating on the Surface Roughness and Hole Dimensional Tolerances during Drilling of Al6061-T651 Alloy. *Materials* **2021**, *14*, 1783. <https://doi.org/10.3390/ma14071783>.
30. Vereschaka, A.; Grigoriev, S.; Tabakov, V.; et al. Influence of the Nanostructure of Ti-TiN-(Ti,Al,Cr)N Multilayer Composite Coating on Tribological Properties and Cutting Tool Life. *Tribol. Int.* **2020**, *150*, 106388. <https://doi.org/10.1016/j.triboint.2020.106388>.
31. Lei, Y.; Ni, J.; Hu, Z.; et al. Surface Modification of Li-Rich Mn-Based Layered Oxide Cathodes: Challenges, Materials, Methods, and Characterization. *Adv. Energy Mater.* **2020**, *10*, 2002506. <https://doi.org/10.1002/aenm.202002506>.
32. Trentin, A.; Pakseresht, A.; Duran, A.; et al. Electrochemical Characterization of Polymeric Coatings for Corrosion Protection: A Review of Advances and Perspectives. *Polymers* **2022**, *14*, 2306.
33. Koller, M.; Cizek, J.; Janovská, M.; et al. Scanning Acoustic Microscopy Characterization of Cold-Sprayed Coatings Deposited on Grooved Substrates. *J. Therm. Spray. Technol.* **2024**, *33*, 1941–1954. <https://doi.org/10.1007/s11666-024-01806-3>.
34. Tang, H.; Yuan, X.; Cheng, Y.; et al. Synthesis of Paracrystalline Diamond. *Nature* **2021**, *599*, 605–610. <https://doi.org/10.1038/s41586-021-04122-w>.
35. Sjögren-Levin, E.; Pantleon, W.; Ahadi, A.; et al. Separation of XRD Peak Profiles in Single-Phase Metals with Bimodal Grain Structure to Analyze Stress Partitioning. *IOP Conf. Ser. Mater. Sci. Eng.* **2022**, *1249*, 012040. <https://doi.org/10.1088/1757-899x/1249/1/012040>.
36. Dolabella, S.; Borzi, A.; Dommann, A.; et al. Lattice Strain and Defects Analysis in Nanostructured Semiconductor Materials and Devices by High-Resolution X-Ray Diffraction: Theoretical and Practical Aspects. *Small Methods* **2022**, *6*, 2100932.
37. Wang, N.; Zhang, X.; Tan, S.; et al. X-Ray Diffraction Studies of Single-Crystal Materials for Broad Battery Applications. *Chem. Rev.* **2025**, *125*, 9834–9874. <https://doi.org/10.1021/acs.chemrev.5c00394>.
38. Yang, W.Y.; Liang, D.; Kong, X.D.; et al. Neutron Diffraction Studies of Permanent Magnetic Materials. *Rare Met.* **2020**, *39*, 13–21.

39. Shoja, S.; Norgren, S.; Andrén, H.O.; et al. On the Influence of Varying the Crystallographic Texture of Alumina CVD Coatings on Cutting Performance in Steel Turning. *Int. J. Mach. Tools Manuf.* **2022**, *176*, 103885. <https://doi.org/10.1016/j.ijmachtools.2022.103885>.
40. Prieske, M.; Vollertsen, F. Picosecond-Laser Polishing of CVD-Diamond Coatings without Graphite Formation. *Mater. Today Proc.* **2020**, *40*, 1–4.
41. Rogström, L.; Moreno, M.; Andersson, J.M.; et al. Structural Changes in Ti<sub>1-x</sub>Al<sub>x</sub>N Coatings during Turning: A XANES and EXAFS Study of Worn Tools. *Appl. Surf. Sci.* **2023**, *612*, 155907. <https://doi.org/10.1016/j.apsusc.2022.155907>.
42. Petrik, P.; Racz, A.S.; Menyhard, M. Complementary Physicochemical Analysis by Ellipsometry and Auger Spectroscopy of Nano-Sized Protective Coating Layers. *Appl. Surf. Sci.* **2020**, *534*, 147593. <https://doi.org/10.1016/j.apsusc.2020.147593>.
43. Ba, E.C.T.; Dumont, M.R.; Martins, P.S.; et al. Deconvolution Process Approach in Raman Spectra of DLC Coating to Determine the Sp<sup>3</sup> Hybridization Content Using the Id/Ig Ratio in Relation to the Quantification Determined by X-Ray Photoelectron Spectroscopy. *Diam. Relat. Mater.* **2022**, *122*, 108818. <https://doi.org/10.1016/j.diamond.2021.108818>.
44. Wang, Z.; Tian, C.; Tolstogouzov, A.; et al. Microstructure and Rutherford Backscattering Spectrometry of Hard/Lubricant Mo-Ti-Al-N Multilayered Coatings Prepared by Multi-Arc Ion Plating at Low Substrate Rotation. *Coatings* **2020**, *10*, 101. <https://doi.org/10.3390/coatings10020101>.
45. Boivin, F.; Vallières, S.; Fourmaux, S.; et al. Quantitative Laser-Based X-Ray Fluorescence and Particle-Induced X-Ray Emission. *New J. Phys.* **2022**, *24*, 053018. <https://doi.org/10.1088/1367-2630/ac6767>.
46. Vijaya, G.; Muralidhar, S.M.; Kumar, M.; et al. Nano Indentation Studies on Ceramic Thin Films Coatings Deposited Using Sputtering Process for Energy Applications. *Mater. Sci. Energy Technol.* **2024**, *7*, 115–123. <https://doi.org/10.1016/j.mset.2023.08.001>.
47. Abubakar, A.A.; Adesina, A.Y.; Arif, A.F.M.; et al. Evaluation of Residual Stress in Thick Metallic Coatings Using the Combination of Hole Drilling and Micro-Indentation Methods. *J. Mater. Res. Technol.* **2022**, *20*, 867–881. <https://doi.org/10.1016/j.jmrt.2022.07.081>.
48. Zawischa, M.; Bin Mohamad Supian, M.M.A.; Makowski, S.; et al. Generalized Approach of Scratch Adhesion Testing and Failure Classification for Hard Coatings Using the Concept of Relative Area of Delamination and Properly Scaled Indenters. *Surf. Coat. Technol.* **2021**, *415*, 127118. <https://doi.org/10.1016/j.surfcoat.2021.127118>.
49. Bheemappa, S.; Gurumurthy, H.; Badami, V.V.; et al. Tribological Behavior of Polymeric Systems in Lubricated Surfaces or Conditions. In *Tribology of Polymers, Polymer Composites, and Polymer Nanocomposites*; Elsevier: Amsterdam, The Netherlands, 2022; pp. 357–399.
50. Baptista, A.; Silva, F.J.G.; Pinto, G.; et al. Influence of the Ball Surface Texture in the Dragging of Abrasive Particles on Micro-Abrasion Wear Tests. *Wear* **2021**, *476*, 203730. <https://doi.org/10.1016/j.wear.2021.203730>.
51. Boughdiri, I.; Giasin, K.; Mabrouki, T.; et al. Effect of Cutting Parameters on Thrust Force, Torque, Hole Quality and Dust Generation during Drilling of GLARE 2B Laminates. *Compos. Struct.* **2021**, *261*, 113562. <https://doi.org/10.1016/j.compstruct.2021.113562>.
52. Bonhin, E.P.; David-Müzel, S.; de Sampaio Alves, M.C.; et al. A Review of Mechanical Drilling on Fiber Metal Laminates. *J. Compos. Mater.* **2021**, *55*, 843–869. <https://doi.org/10.1177/0021998320957743>.
53. Giasin, K.; Hawxwell, J.; Sinke, J.; et al. The Effect of Cutting Tool Coating on the Form and Dimensional Errors of Machined Holes in GLARE<sup>®</sup> Fibre Metal Laminates. *Int. J. Adv. Manuf. Technol.* **2020**, *107*, 2817–2832. <https://doi.org/10.1007/s00170-020-05211-2>.
54. Sousa, V.F.C.; Fernandes, F.; Silva, F.J.G.; et al. Wear Behavior Phenomena of TiN/TiAlN HiPIMS PVD-Coated Tools on Milling Inconel 718. *Metals* **2023**, *13*, 684. <https://doi.org/10.3390/met13040684>.
55. Pinto, G.F.; Silva, F.J.G.; Silva, E.; et al. Doping Strategies for TiAlN-Based Cutting Tool Coatings: Progress and Perspectives. *J. Mech. Eng. Manuf.* **2026**. <https://doi.org/10.53941/jmem.2026.100010>.
56. Lopes Correia Pinto, G.F.; Almeida, D.; Silva, F.; et al. Correlating Cutting Performance and Surface Roughness under Different Bias Using TiAlTa<sub>n</sub> Coated Milling Tools. *J. Mech. Eng. Manuf.* **2025**, *1*, 7.
57. Kumar Das, A. Recent Advancements in Nanocomposite Coating Manufactured by Laser Cladding and Alloying Technique: A Critical Review. *Mater. Today Proc.* **2022**, *57*, 1852–1857. <https://doi.org/10.1016/j.matpr.2022.01.078>.
58. Baptista, A.; Silva, F.J.G.; Porteiro, J.; et al. Mechanical and Tribological Properties of Ti-Al-Y-N and Ti-Al-Ta-N Coatings Deposited by HiPIMS: Influence of the Bias Voltage. *Results Eng.* **2026**, *29*, 109120. <https://doi.org/10.1016/j.rineng.2026.109120>.
59. Dabees, S.; Mirzaei, S.; Kaspar, P.; et al. Characterization and Evaluation of Engineered Coating Techniques for Different Cutting Tools—Review. *Materials* **2022**, *15*, 5633. <https://doi.org/10.3390/ma15165633>.
60. Jahan, M.P.; Ma, J.; Hanson, C.; et al. Tool Wear and Resulting Surface Finish during Micro Slot Milling of Polycarbonates Using Uncoated and Coated Carbide Tools. *Proc. Inst. Mech. Eng. B J. Eng. Manuf.* **2020**, *234*, 52–65. <https://doi.org/10.1177/0954405419862479>.

61. Kong, X.; Deng, J.; Dong, J.; et al. Study of Tip Wear for AFM-Based Vibration-Assisted Nanomachining Process. *J. Manuf. Process.* **2020**, *50*, 47–56. <https://doi.org/10.1016/j.jmapro.2019.12.013>.
62. Zhang, Y.; Cui, K.; Gao, Q.; et al. Investigation of Morphology and Texture Properties of WS<sub>2</sub> Coatings on W Substrate Based on Contact-Mode AFM and EBSD. *Surf. Coat. Technol.* **2020**, *396*, 125966. <https://doi.org/10.1016/j.surfcoat.2020.125966>.
63. Michna, Š.; Hren, I.; Novotný, J.; et al. Comprehensive Research and Analysis of a Coated Machining Tool with a New TiAlN Composite Microlayer Using Magnetron Sputtering. *Materials* **2021**, *14*, 3633. <https://doi.org/10.3390/ma14133633>.
64. Karthikeyan, M.; Pandian, S.M.V.; Vijayakumar, R. Investigation on Metal Hybrid Fibres Laminate (MHFL) in Hybrid Machining. *J. Manuf. Process.* **2023**, *101*, 835–853. <https://doi.org/10.1016/j.jmapro.2023.06.046>.
65. Sousa, V.F.C.; Silva, F.J.G.; Alexandre, R.; et al. Study of the Wear Behaviour of TiAlSiN and TiAlN PVD Coated Tools on Milling Operations of Pre-Hardened Tool Steel. *Wear* **2021**, *476*, 203695. <https://doi.org/10.1016/j.wear.2021.203695>.
66. Montazeri, S.; Aramesh, M.; Rawal, S.; et al. Characterization and Machining Performance of a Chipping Resistant Ultra-Soft Coating Used for the Machining of Inconel 718. *Wear* **2021**, *474–475*, 203759. <https://doi.org/10.1016/j.wear.2021.203759>.
67. Vats, P.; Khanna, N.; Kumar, A.; et al. Lubrication-Driven Surface and Tool Performance in Sustainable Machining of Additively Manufactured Nickel Superalloy. *Surf. Coat. Technol.* **2025**, *515*, 132637. <https://doi.org/10.1016/j.surfcoat.2025.132637>.
68. Bolar, G.; Satish Shenoy, B.; Zitoun, R.; et al. Analysis of Machining Induced Damage When Drilling Hybrid Carbon Fiber-Reinforced Aluminum Laminates. *J. Compos. Mater.* **2026**, *60*, 123–145.
69. Liang, J.; Gao, H.; Xiang, S.; et al. Research on Tool Wear Morphology and Mechanism during Turning Nickel-Based Alloy GH4169 with PVD-TiAlN Coated Carbide Tool. *Wear* **2022**, *508–509*, 204468. <https://doi.org/10.1016/j.wear.2022.204468>.
70. Tymczyszyn, J.; Szajna, A.; Mrówka-Nowotnik, G. Influence of Nanocomposite PVD Coating on Cutting Tool Wear During Milling of 316L Stainless Steel Under Air Cooling Conditions. *Materials* **2025**, *18*, 1959. <https://doi.org/10.3390/ma18091959>.
71. Sebbe, N.P.V.; Baptista, A.; Pinto, G.; et al. A Review of Wear Mechanisms and Solutions Regarding Inserts for Grinding Applications. *J. Mech. Eng. Manuf.* **2026**, *2*, 4.
72. Zhang, L.; Zhao, Z.; Bai, P.; et al. EBSD Investigation on Microstructure Evolution of In-Situ Synthesized TiC/Ti6Al4V Composite Coating. *Mater. Lett.* **2021**, *290*, 129449. <https://doi.org/10.1016/j.matlet.2021.129449>.
73. Bhandarkar, L.R.; Behera, M.; Mohanty, P.P.; et al. Experimental Investigation and Multi-Objective Optimization of Process Parameters during Machining of AISI 52100 Using High Performance Coated Tools. *Measurement* **2021**, *172*, 108842. <https://doi.org/10.1016/j.measurement.2020.108842>.
74. Bayraktar, Ş.; Hekimoğlu, A.P. Effect of Zinc Content and Cutting Tool Coating on the Machinability of the Al-(5–35) Zn Alloys. *Met. Mater. Int.* **2020**, *26*, 477–490. <https://doi.org/10.1007/s12540-019-00582-y>.
75. Zhang, X.; Zhang, K.; Dang, J.; et al. Analysis of Tool Wear and Cutting Characteristics in Milling of Powder Metallurgy Nickel-Based Superalloy by Various Coatings. *Wear* **2024**, *552–553*, 205429. <https://doi.org/10.1016/j.wear.2024.205429>.
76. Hovorun, T.; Khaniukov, K.; Varakin, V.; et al. Improvement of the Physical and Mechanical Properties of the Cutting Tool by Applying Wear-Resistant Coatings Based on Ti, Al, Si, and N. *J. Eng. Sci.* **2021**, *8*, C13–C23.
77. Elhelaly, M.A.; El-Zomor, M.A.; Youssef, A.O.; et al. Characterization of VC Coatings on Cold Work Tool Steel Produced by TRD. *Manuf. Technol.* **2021**, *21*, 600–605. <https://doi.org/10.21062/mft.2021.084>.
78. Sousa, V.F.C.; Castanheira, J.; Silva, F.J.G.; et al. Wear Behavior of Uncoated and Coated Tools in Milling Operations of Ampco (Cu-Be) Alloy. *Appl. Sci.* **2021**, *11*, 7762. <https://doi.org/10.3390/app11167762>.
79. Wu, T.; Degener, S.; Tinkloh, S.; et al. Characterization of Residual Stresses in Fiber Metal Laminate Interfaces—A Combined Approach Applying Hole-Drilling Method and Energy-Dispersive X-Ray Diffraction. *Compos. Struct.* **2022**, *299*, 116071. <https://doi.org/10.1016/j.compstruct.2022.116071>.
80. Denkmann, N.; Jaquet, S.; Garcia Carballo, R.; et al. Determining and Predicting the Flank Wear Width and Residual Stress of Coated Tools by Raman Imaging. *Wear* **2026**, *584–585*, 206397. <https://doi.org/10.1016/j.wear.2025.206397>.
81. Celik, S.; Bekoz Ullen, N.; Akyuz, S.; et al. Raman Spectroscopic Investigation of the Wear Effect on the Titanium Carbonitride/Aluminum Oxide/Titanium Nitride Coated Cutting Tool. *Spectrosc. Lett.* **2022**, *55*, 172–182. <https://doi.org/10.1080/00387010.2022.2041039>.
82. Lukin, S.; Užarević, K.; Halasz, I. Raman Spectroscopy for Real-Time and in Situ Monitoring of Mechanochemical Milling Reactions. *Nat. Protoc.* **2021**, *16*, 3492–3521. <https://doi.org/10.1038/s41596-021-00545-x>.
83. Wang, X.; Zhang, B.; Qiao, Y.; et al. Chemo-Mechanical Abrasive Flow Machining (CM-AFM): A Novel High-Efficient Technique for Polishing Diamond Thin Coatings on Inner Hole Surfaces. *J. Manuf. Process.* **2021**, *69*, 152–164. <https://doi.org/10.1016/j.jmapro.2021.07.042>.
84. Cygan-Bączek, E.; Cygan, S.; Wyżga, P.; et al. Improvement in Abrasive Wear Resistance of Metal Matrix Composites Used for Diamond-Impregnated Tools by Heat Treatment. *Materials* **2023**, *16*, 6198. <https://doi.org/10.3390/ma16186198>.
85. Wang, H.; Yang, J.; Sun, F. Cutting Performances of MCD, SMCD, NCD and MCD/NCD Coated Tools in High-Speed Milling of Hot Bending Graphite Molds. *J. Mater. Process. Technol.* **2020**, *276*, 116401. <https://doi.org/10.1016/j.jmatprotec.2019.116401>.

86. Visuvamithiran, R.; Narayanaperumal, A.; Ramachandran, V.; et al. Analysis of Drilling Performance of Carbon Fiber Reinforced Polymer with Special Tool Geometries and Raman Spectrums. *Hybrid. Adv.* **2025**, *10*, 100455. <https://doi.org/10.1016/j.hybadv.2025.100455>.
87. Xiong, J.; Liu, L.; Song, H.; et al. Mechanical Properties Evaluation of Diamond Films via Nanoindentation. *Diam. Relat. Mater.* **2022**, *130*, 109403. <https://doi.org/10.1016/j.diamond.2022.109403>.
88. Yuan, J.R.; Zhu, X.P.; Lei, M.K. Nano-, Micro- and Macro-Indentation Tests of Thermal Spray WC-Ni Coatings with Lamellar Microstructure at Different Particle Deposition Temperatures. *Mater. Charact.* **2024**, *215*, 114234. <https://doi.org/10.1016/j.matchar.2024.114234>.
89. Mamalis, A.; Mechnik, V.; Morozow, D.; et al. Properties of Cutting Tool Composite Material Diamond-(Fe-Ni-Cu-Sn) Reinforced with Nano-VN. *Machines* **2022**, *10*, 410. <https://doi.org/10.3390/machines10060410>.
90. Elias, J.V.; Venkatesh, N.P.; Lawrence, K.D.; et al. Tool Texturing for Micro-Turning Applications—An Approach Using Mechanical Micro Indentation. *Mater. Manuf. Process.* **2021**, *36*, 84–93.
91. Rashid, M.M.U.; Tomkowski, R.; Archenti, A. Extending Tool Life: High-Dynamic Stiffness Nanocomposite Coating for Improved Machining Performance. *Surf. Coat. Technol.* **2025**, *515*, 132604. <https://doi.org/10.1016/j.surfcoat.2025.132604>.
92. Trejo, J.; Tolosa, R.; Ruiz, N.; et al. Comparative Study of the Main Properties Associated with Thin Layers of Coatings with the Cobalt-Chromium-Tungsten Alloy (Stellite) and Hard Chromium Plating Used as Reinforcements for Wood Sawing. *Mech. Mater.* **2021**, *152*, 103637. <https://doi.org/10.1016/j.mechmat.2020.103637>.
93. Casais, R.; Baptista, A.M.; Silva, F.J.; et al. Experimental Study on the Wear Behavior of B<sub>4</sub>C and TiB<sub>2</sub> Monolayered PVD Coatings under High Contact Loads. *Int. J. Adv. Manuf. Technol.* **2022**, *120*, 6585–6604. <https://doi.org/10.1007/s00170-022-09182-4>.
94. Siddiqui, S.A.; Favaro, G.; Berkes Maros, M. Investigation of the Damage Mechanism of CrN and Diamond-Like Carbon Coatings on Precipitation-Hardened and Duplex-Treated X42Cr13/W Tool Steel by 3D Scratch Testing. *J. Mater. Eng. Perform.* **2022**, *31*, 7830–7842. <https://doi.org/10.1007/s11665-022-06812-6>.
95. Vopát, T.; Sahul, M.; Haršáni, M.; et al. The Tool Life and Coating-Substrate Adhesion of AlCrSiN-Coated Carbide Cutting Tools Prepared by LARC with Respect to the Edge Preparation and Surface Finishing. *Micromachines* **2020**, *11*, 166. <https://doi.org/10.3390/mi11020166>.
96. Łepicka, M. Coating Failure Detection in Scratch Testing: A Cross-Sectional SEM/FIB Microscopic Study Coupled with Nonlinear Analysis Methods in a Model Titanium Nitride/Stainless Steel System. *Wear* **2025**, *562–563*, 205670.
97. Kupeczyk, M.J.; Józwiak, J. Effect of Laser Heating on the Life of Cutting Tools Coated with Single- and Multilayer Coatings Containing a TiN Layer. *Materials* **2022**, *15*, 4022.
98. Storchak, M.; Zakiev, I.; Zakiev, V.; et al. Coatings Strength Evaluation of Cutting Inserts Using Advanced Multi-Pass Scratch Method. *Measurement* **2022**, *191*, 110745. <https://doi.org/10.1016/j.measurement.2022.110745>.
99. Talibouya Ba, E.C.; Dumont, M.R.; Martins, P.S.; et al. Investigation of the Effects of Skewness Rsk and Kurtosis Rku on Tribological Behavior in a Pin-on-Disc Test of Surfaces Machined by Conventional Milling and Turning Processes. *Mater. Res.* **2021**, *24*, e20200435.
100. Osmond, L.; Cook, I.; Slatter, T. Tribological Properties of Multilayer CVD Coatings Deposited on SiAlON Ceramic Milling Inserts. *J. Manuf. Mater. Process.* **2023**, *7*, 67.
101. Palani, S.; Hailu, T.; Elias, G. Experimental Investigation on Wear Behavior of Titanium Alloy (Grade 23) by Pin on Disc Tribometer. *Results Mater.* **2023**, *19*, 100422.
102. Strel'nikskij, V.; Vasylyev, V.; Makarov, V.; et al. Diamond-like Carbon Coatings Pin-on-Disk Wear Testing. *Phys. Chem. Solid. State* **2023**, *24*, 520–529. <https://doi.org/10.15330/PCSS.24.3.520-529>.
103. Singh Yadav, S.P.; Lakshmikanthan, A.; Ranganath, S.; et al. Effect of Pin Geometry and Orientation on Friction and Wear Behavior of Nickel-Coated EN8 Steel Pin and Al6061 Alloy Disc Pair. *Adv. Mater. Sci. Eng.* **2022**, *2022*, 3274672.
104. Silva, F.J.G.; Casais, R.C.B.; Baptista, A.P.M.; et al. Comparative Study of the Wear Behavior of B<sub>4</sub>C Monolayered and CrN/CrCN/DLC Multilayered Physical Vapor Deposition Coatings Under High Contact Loads: An Experimental Analysis. *J. Tribol.* **2022**, *144*, 031701.
105. Mundotia, R.; Kothari, D.C.; Kale, A.; et al. Effect of Ion Bombardment and Micro-Blasting on the Wear Resistance Properties of Hard TiN Coatings. *Mater. Today Proc.* **2019**, *26*, 603–612.
106. Esteves, P.J.; Seriacopi, V.; de Macêdo, M.C.S.; et al. Combined Effect of Abrasive Particle Size Distribution and Ball Material on the Wear Coefficient in Micro-Scale Abrasive Wear Tests. *Wear* **2021**, *476*, 203639. <https://doi.org/10.1016/j.wear.2021.203639>.
107. Çay, V.V. Effects of Abrasive Particle Type, Load and Sliding Distance on Micro-Abrasion Resistance of High Speed Steel Coated with AlCrN or AlTiN. *Medziagotyra* **2021**, *27*, 50–56. <https://doi.org/10.5755/j02.ms.25776>.
108. Mariani, F.E.; Rêgo, G.C.; Bonella, P.G.; et al. Wear Resistance of Niobium Carbide Layers Produced on Gray Cast Iron by Thermoreactive Treatments. *J. Mater. Eng. Perform.* **2020**, *29*, 3516–3522.

109. Chişiu, G.; Gheţa, R.A.; Stoica, A.M.; et al. Comparative Micro-Scale Abrasive Wear Testing of Thermally Sprayed and Hard Chromium Coatings. *Lubricants* **2023**, *11*, 350.
110. *ASTM E2546-07*; Standard Practice for Instrumented Indentation Testing. Advanced Standards Transforming Markets: West Conshohocken, PA, USA, 2007.
111. *ASTM E384-17*; Standard Test Method for Microindentation Hardness of Materials. Advanced Standards Transforming Markets: West Conshohocken, PA, USA, 2017.
112. *ISO 20502:2005*; Determination of Adhesion of Ceramic Coatings by Scratch Testing. International Standard Organization: Geneva, Switzerland, 2005.
113. *ISO 18535:2016*; Diamond-like Carbon Films—Determination of Friction and Wear Characteristics of Diamond-like Carbon Films by Ball-on-Disc Method. International Standard Organization: Geneva, Switzerland, 2016.
114. *ISO 26424:2008*; Fine Ceramics (Advanced Ceramics, Advanced Technical Ceramics). International Standard Organization: Geneva, Switzerland, 2008.