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DEVELOPMENT OF PURE AND MODIFIED TICN VIA PULSED PLASMA SINTERING (SPS): A REVIEW

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Abstract: Titanium carbonitride (TiCN) is an ultra-high temperature ceramic (UHTC) material, widely used in cutting tools, semiconductor transistors and wear-resistant coatings. Its high hardness, excellent abrasion resistance, low coefficient of friction and chemical resistance at high temperatures make it a promising candidate to replace WC-Co carbide materials. However, the brittleness and low fracture toughness of pure TiCN limit its use, and it is more commonly used as a hard phase bonded to a metallic material to form wear-resistant composites. Pulsed plasma sintering (SPS) has excelled in achieving high densification in pure materials, due to its unique heating source, high heating rates and reduced sintering times. This minimizes defects caused by high temperatures and long processing times in traditional powder metallurgy. This article analyzed the state of the art on pure TiCN and TiCN with added binders, sintered via SPS. The studies indicated that SPS provides superior results, making it an effective alternative for manufacturing hard, wear-resistant materials.

Keywords: Ceramic Material, Titanium Carbonitride, Pulsed Plasma Sintering (SPS), Wear Resistance.

INTRODUCTION

The development of Titanium Carbonitride (TiCN) has advanced significantly in recent years due to its unique properties, such as high hardness, high abrasion resistance, low coefficient of friction, good deformation and chemical resistance at high temperatures. In addition, it has low cost, low weight, good thermal conductivity and resistance to wear at high temperatures, making it a promising alternative to metallic materials (RAJABI, Armin *et at* .., 2022)

This material has been successfully applied in the development of cutting tools and surface finishing processes, standing out compared to WC-Co hard metals. Its application allows for excellent chip size control, greater dimensional accuracy of the workpiece and better tolerance control, making it highly efficient in industrial processes (ZHOU *et at* .., 2021)

Despite these advantages, pure TiCN is rarely used due to its brittleness and low fracture toughness. For more demanding applications, it is usually combined with metals such as nickel and cobalt to form hard, wear-resistant composites. This approach improves its mechanical properties and extends its use in different segments of the industry (CARDINAL *et at* .., 2009)

In recent years, several studies have sought cost-effective methods to modify the morphology of TiCN by controlling grain growth during synthesis. Strategies such as nanostructuring and the addition of reinforcements have been explored to improve its fracture toughness. These techniques help to limit crack propagation, increasing the mechanical strength of the material (LIU, Jian *et at* .., 2017)

The densification of TiCN requires extremely high sintering temperatures, making field-assisted sintering techniques necessary. Among conventional methods, such as hot uniaxial pressing (HP), hot isostatic pressing (HIP) and atmospheric furnaces, Pulsed Plasma Sintering (SPS) stands out. This technique allows efficient densification with high heating rates, lower temperatures and shorter processing times, reducing grain growth and preventing structural defects (ROMINIYI *et at* .., 2020)

Given the growing interest in sintering pure TiCN and the limited literature on the subject, this article aims to analyze the consolidation of TiCN without binders and the advances of the SPS technique in sintering this material. The research aims to evaluate the impact of SPS in obtaining improved properties, as well as comparing its results with conventional methods, with a view to high-performance industrial applications.

TITANIUM CARBONITRIDE (TICN): RELATIONSHIP BETWEEN PROCESSING, MICROSTRUCTURE AND PROPERTIES

SOLUBILIZATION AND PRECIPITATION OF CARBONITRIDES

Transition metal carbides and nitrides are known to form solid solutions, resulting in carbonitrides whose properties in the solid state vary according to the [C / (C + N)] ratio. This behavior allows for a wide adaptation of the characteristics of carbonitrides, making them versatile materials for various industrial applications. In addition, these compounds show good wettability by liquid metals, a characteristic that is exploited in the manufacture of hard metals (KRAL *et at*., 1998). Studies have shown that modifying the composition of these compounds can result in significant improvements in terms of hardness, corrosion resistance and thermal stability, expanding their potential for application in harsh environments (LEE *et at* .., 2003)

Precipitation hardening in these materials is achieved by the formation of fine precipitates, which can nucleate at the interfaces between austenite and ferrite during phase transformation. The precipitation of carbonitrides associated with these interfaces is a type of discontinuous precipitation known as interphase precipitation. This phenomenon has been observed in proeutectoid and ferritic-perlitic steels, contributing to an increase in the mechanical strength of these materials (OLIVEIRA, 2014) .

The chemical stability of transition metal carbonitrides is high; they are hardly attacked by dilute acids or alkaline solutions, with the exception of oxidizing acids and hydrofluoric acid. Their thermal stability is linked to their free energy of formation. While the transition metal nitrides of group IV, Titanium Nitride (TiN), Zirconium Nitride (ZrN) and Hafnium Nitride (HfN) and carbonitrides of Titanium (TiCN), Zirconium Carbonitride (ZrCN) and Hafnium Carbonitride (HfCN) can be melted without decomposition, the nitrides and carbonitrides of the other groups can decompose before the melting points are reached. As a result, nitrogen is released (LENGAUER, 2000)

In addition, understanding the mechanisms of interphase precipitation and the influence of phase transformations on the microstructure of carbonitrides are fundamental to the development of advanced metal alloys. Recent research focuses on optimizing these processes to obtain materials with superior mechanical properties, with a view to applications in sectors such as the aerospace and automotive industries, where the combination of high strength and lightness is essential (CHEN, Wenge *et at*., 2020)

Continued research into the chemical and thermal properties of transition metal carbonitrides, as well as the exploration of new compositions and processing methods, are crucial to expanding their technological applications. Advances in this field can lead to the development of materials with improved performance, contributing to innovations in various areas of engineering and materials science (RAJABI, Armin *et at* .., 2022)

STRUCTURE AND COMPOSITION OF TITANIUM CARBONITRIDE (TICN)

Titanium carbide (TiC) and titanium nitride (TiN) are the main constituents of titanium carbonitride, represented by the formula Ti(C,N). Both have a face-centered cubic (CFC) crystal structure, in which the edges of the cubic lattice are formed by carbon (in the case of TiC) or nitrogen (in the case of TiN) atoms, located at the (1/2, 0, 0) point of the super lattice made up of titanium atoms (ZHANG, Shanyong, 1993) but the boom of the cutting grades really started in the early 1970s when titanium carbide-based cermets were established. However, because of their superior properties, the Ti(C,N . The lattice parameter of TiN is slightly smaller than that of TiC, but as both are isomorphous, the carbon atoms in the TiC super lattice can be replaced by nitrogen atoms in any proportion. It is therefore possible to obtain a continuous series of solid solutions with the formulation $Ti(C_{1-x}N_x)$, where $0 \leq x \leq 1$

The crystal structure of titanium carbonitride $Ti(C_xN_{1-x})$ follows the NaCl type, maintaining the face-centered cubic (CFC) configuration (CAO, Zhinan *et at* . ., 2021)Figure 1 illustrates this structure, correlating it with the individual structures of TiC and TiN. The interstitial location of the carbon and nitrogen atoms in TiCN has been the subject of study, as it directly affects its mechanical and thermodynamic properties (LEVI; KAPLAN; BAMBERGER, 1998). The formation mechanism of TiCN can be described as a substitution process, in which nitrogen atoms take up positions previously occupied by carbon atoms, resulting in an ordered arrangement of titanium atoms, while the N and C atoms remain disordered (AKINRIBIDE *et at*)..., 2020) relative density, and mechanical properties of TiN ceramics matrix were examined. The investigation was performed on TiN powder with varying graphite content (1–5 wt.%

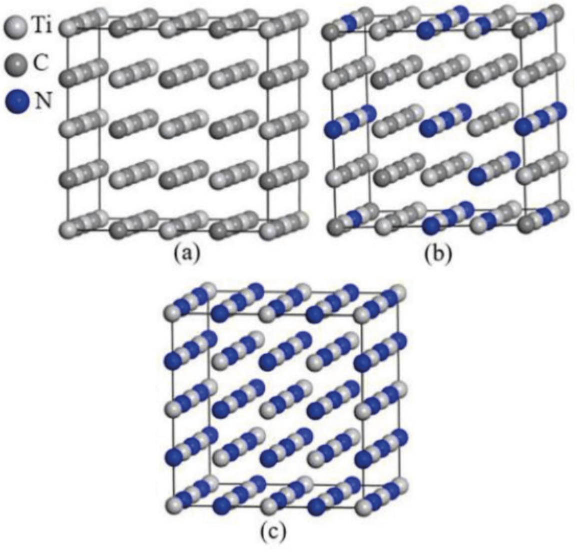


Figure 1 Crystal structures: a) TiC b) $Ti(C_xN_{(1-x)})$ c) TiN. Adapted from (CAO *et at*., 2021).

One of the main advantages of TiCN over pure TiC is its better adhesion to the substrate and lower internal stress. Due to the high internal compressive stress, pure TiC coatings suffer premature fragmentation. In contrast, TiCN coatings have lower residual stress, which improves adhesion and mechanical strength (KNOTEK; LÖFFLER; KRÄMER, 1992). In addition, transition metal carbonitrides, such as TiCN, can be considered a hybrid alloy of carbide and nitride and exist in a wide range of compositions. The value of "x" in the TiC_xN_{1-x} formula can vary between 0 and 1, directly influencing the concentration of vacancies in the interstitial sites. These compositional variations affect the thermodynamic, mechanical, electrical, magnetic and superconducting properties of the material. Studies indicate that although pure TiC is harder than pure TiN, the maximum hardness of TiCN occurs at an optimum C/N ratio within the carbonitride phase (HEYDARI *et at*., 2021)

Titanium plays a fundamental role in the microstructure of TiCN and can act as a grain refiner and precipitation strengthening agent. When present in concentrations above 0.05%, titanium carbides begin to have a significant effect on strengthening the matrix (ALLEN; BOARDMAN, 2005) . Commercially, titanium is widely used to control austenite grain growth, promoting improvements in the toughness and mechanical strength of TiCN-based materials.

CORE-EDGE STRUCTURE OF TICN

The core-edge structure is a characteristic morphology formed by the dissolution and reprecipitation mechanism during the sintering of hard metals based on titanium carbonitride. The resulting microstructure can be described as a hard phase composed of particles with a core-edge structure, embedded in a binder metal matrix (ETTMAYER; KOLASKA, 1995) . Typically, Ti(C,N)-based hard metals are made up of three main phases: the hard phase, the metallic binder phase and the adjacent phases. The core is made up of the residues of the original raw material, consisting of undissolved Ti(C,N) particles, while the edges correspond to the solid solutions reprecipitated during sintering (MARI *et at*., 2003)

The chemical composition of the edges can vary depending on the elements dissolved and reprecipitated during processing. The outer edge is predominantly a solid solution rich in titanium, also containing heavy elements such as tungsten (W), molybdenum (Mo) or tantalum (Ta), forming compounds of the type (Ti,M)(C,N), where M = W, Mo, Ta and/or Nb. The inner edge, on the other hand, has a higher concentration of these heavy elements due to the different activity of nitrogen in the sintering process at high temperatures (USELDINGER *et at*., 2022)). The presence of these phases directly influences the mechanical properties of the material, with undissolved Ti(C,N) cores providing excellent wear resistance, while the interaction between the edge structure and the binding metallic phase determines the toughness of the carbide (AHN; KANG, 2001) .

The formation of the core-edge structure can be controlled by several factors, including the initial chemical composition, the sintering atmosphere and the heating rate. Studies indicate that nitrogen-rich atmospheres during sintering favor the stabilization of the Ti(C,N) phase, reducing excessive dissolution of the core and ensuring a homogeneous edge. In addition, the introduction of grain growth inhibiting agents, such as NbC and VC, can restrict the coalescence of particles during sintering, promoting a more refined microstructure and improving the mechanical properties of the composite (LABONNE *et at*., 2022; LEE *et at* . ., 2003)

Another relevant aspect in the development of hard metals based on Ti(C,N) is the influence of the volume fraction of the binder phase on the final strength of the material. An excessive increase in the metallic phase can compromise hardness and wear resistance, while an insufficient amount can reduce toughness and make the material more susceptible to fracture (RAJABI, A.; GHAZALI; DAUD, 2015) . Recent research suggests that optimizing the balance between the hard phase and the binder phase is essential to maximize the material's efficiency in industrial applications, such as cutting tools and components subject to severe wear (MATIVENGA *et at* .., 2024)

During liquid sintering, the transition metal elements initially dissolve in the binder phase and are then reprecipitated on the undissolved hard particles, thus forming the typical core-edge structure (CAO *et at*., 2021). Controlling this process is essential for optimizing the material's final properties. Elements such as molybdenum (Mo) and tungsten (W) are used to improve the wettability of the hard phase in relation to the binder phase, promoting better cohesion between the grains. On the other hand, tantalum (Ta) and niobium (Nb) are often added to increase the material's thermal resistance, ensuring structural stability at high temperatures (USELDINGER *et at*., 2022; XIONG *et at* . ., 2020)

The growth of the edges occurs by deposition over the cores, favored by the structural similarity, improving the strength of the bond with the binder phase. In some cases, the edge is divided into external and internal, both with the same crystalline structure as the core, but with a higher concentration of heavy metals. However, this compositional variation can generate internal stresses at the core-edge interface, resulting from network incompatibility and differences in physical properties, such as modulus of elasticity, coefficient of thermal expansion, thermal conductivity and diffusion parameters at the interface (PENG; MIAO; PENG, 2013) . Thus, reducing this structural incompatibility is an essential aspect for the development of more resistant and durable Ti(C,N)-based hard metals, either through homogeneous solid solutions or through multi-core-edge structures.

Studies conducted by Li *et at*., (2012) analyzed the formation of the core-edge structure in Ti(C,N) based hard metals, synthesized from Ti(C,N) \ WC \ Mo_2C \ TaC \ Ni \ Co composite powders, by means of vacuum and low pressure sintering. The results indicated that increasing the sintering temperature is directly related to increasing the thickness of the outer edge, significantly influencing the mechanical properties of the hard metals.

Figure 2 shows a schematic illustrating this formation process: (a) absence of initial core-edge structure; (b) small fraction of composition dispersed around the Ti(C,N) particles; (c) significant dissolution of Mo_2C and TaC in the Ni/Co binder phase at 1150 °C; (d) incomplete edge formation, with ceramic grains partially surrounded by a thick white edge phase; (e) partial dissolution of the white phase in the Ni/Co metal matrix at 1350 ºC, followed by reprecipitation on the surface of the black core, forming the outer edge; (f) completely formed core-edge structure, with elimination of porosity and clear definition of the black core - white inner edge - gray outer edge sequence. This increase in the thickness of the outer edge is attributed to the diffusion of heavy metals from the white phase to the black core and from the gray edge to the metallic phase, in addition to the continuous reprecipitation of heavy metals from the Ni/Co solid solution, promoting the structural consolidation of the matrix (LI *et at*., 2012).

A study by Useldinger *et at*. (2022) determined the influence of temperature on TiCN samples sintered via HIP at different temperatures. Figure 3 shows BSE images of the microstructure of the samples at temperatures of 1440 - 1520 °C, where the core-edge structure formed can be clearly seen.

HARD METALS BASED ON TITANIUM CARBONITRIDE (TICN)

Hard metals with a titanium carbonitride matrix have been developed from titanium carbide (TiC) matrices, with the addition of titanium nitride (TiN). In the study by Voitovich and Pugach, (1972), it was discovered that the addition of TiN to TiC-Mo-Ni carbide significantly improved its mechanical properties both at room temperature and at high temperatures. In addition, this modification provided greater resistance to erosion and oxidation, eliminating the negative perception about the presence of nitrogen in hard metals (PENG; MIAO; PENG, 2013; ZHANG, HOUAN *et at* . ., 2006)

TiCN-based hard metals have been widely studied as potential substitutes for WC-Co cemented carbides, traditionally used in the manufacture of cutting tools. Their main advantages include high hardness, excellent abrasion resistance, low coefficient of friction, chemical resistance at high temperatures, low cost and lower density compared to conventional materials (ZHOU *et at*., 2021)). These characteristics make these composites perform better in various industrial applications, especially where wear resistance and thermal stability are required.

Another factor contributing to the growing industrial interest in TiCN-based hard metals is the versatility of their formulation. Depending on the composition of the matrix and the binder phase, different properties can be adjusted to meet specific requirements. The choice of binder material, such as nickel (Ni), cobalt (Co) or iron (Fe), directly influences the toughness of the composite, while the addition of refractory carbides can modify its mechanical and thermal resistance (XU *et at*., 2015). The

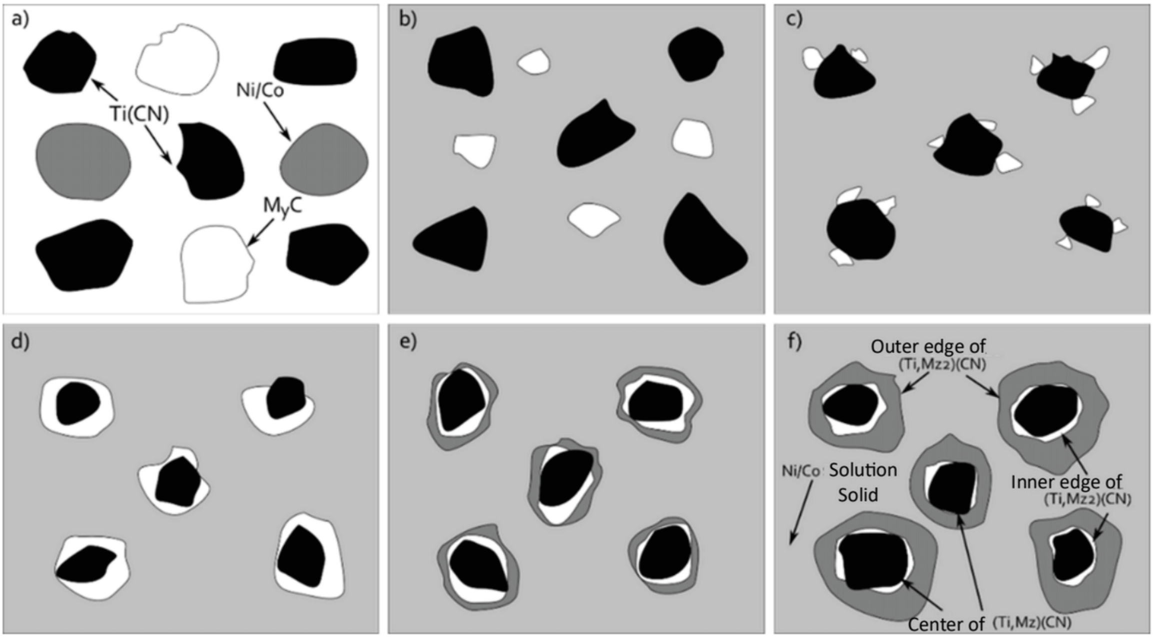


Figure 2: Illustration of the characteristics of the core-edge structure in Ti (C, N)-MxC carbide (a) 20 °C (b) 950 °C (c) 1150 °C (d) 1300 °C (e) 1350 °C (f) 1450 °C. Adapted from (LI *et at*., 2012).

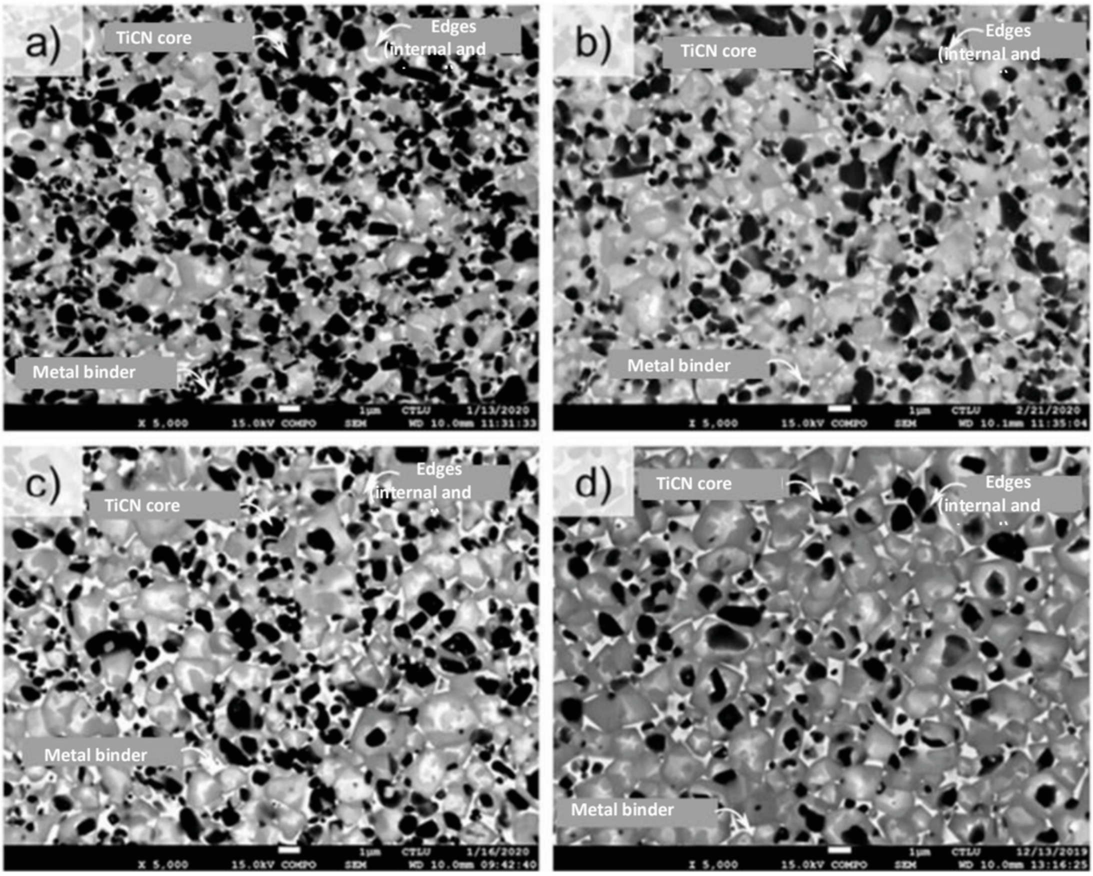


Figure 3. Grade A BSE microstructures (a) sintering temperature 1400 °C, b) 1440 °C, c) 1480 °C and d) 1520 °C Adapted from (USELDINGER *et at*., 2022).

optimization of sintering, combined with adjustments to the ratio between TiC and TiN, makes it possible to obtain more homogeneous microstructures with better control of grain growth, resulting in more efficient performance in highly demanding applications. Titanium carbonitride is the main raw material for the production of TiCN-based hard metals, being mixed with Ni/Co, molybdenum carbide (Mo_2C) and other refractory carbides (MATSUDA, 2020)N . Compared to traditional cemented carbides based on tungsten carbide (WC), which are widely used, hard metals with a TiCN matrix have greater hardness, a lower coefficient of friction and better resistance to oxidation (KANG *et at*).., 2020)

Today, TiCN-based hard metals are widely used in machining, cutting, tool forming and wear-resistant parts. Thanks to their high hardness, mechanical strength and thermal stability, these materials have been used as protective coatings for carbide inserts, tools and dies, extending their useful life and reducing operating costs (ZHENG *et at* .., 2020)

TiCN-based hard metals are made up of rigid TiCN grains joined by a metallic binder phase, usually composed of cobalt (Co), nickel (Ni) or iron (Fe), formed during liquid-phase sintering at high temperatures. The hard phase is responsible for wear resistance, while the binder phase contributes to increased toughness. The matrix can be reinforced with different transition metal carbides to improve certain properties. The addition of niobium carbide (NbC) or tantalum carbide (TaC) improves performance in interrupted cuts, as it contributes to increased hot hardness and resistance to thermal shock. Vanadium carbide (VC) improves fatigue resistance, while the presence of hafnium carbide (HfC) or zirconium carbide (ZrC) reduces tensions between different microstructural phases. Tungsten carbide (WC) is added to increase toughness (KUMAR; KUMAR; BASU, 2007).

Ti(C,N)-based hard metals have high hardness, good chemical stability and wear resistance, and are widely used in the manufacture of cutting tools and abrasion-resistant parts. However, when compared to tools based on tungsten carbide (WC), they have greater brittleness and lower fracture toughness (XIONG *et at*., 2018). Thus, recent research has focused on the development of compositions and sintering processes to improve the strength and toughness of these materials, making them more competitive for demanding applications. The study of the relationship between microstructure and mechanical properties continues to be one of the main challenges in the evolution of TiCN-based hard metals, attracting great interest in industry and academic research (PARK; NAM; KANG, 2016) .

Table 1 shows the specific properties of TiC and TiN, from which the properties of TiCN can be roughly judged (PENG; MIAO; PENG, 2013). Thus, TiCN has the properties of TiC and TiN: high hardness, high melting point, good corrosion resistance, excellent wear resistance, good chemical stability, high thermal and electrical conductivity, etc. (LIU *et at*., 2004; ZHANG; YAN; TANG, 2010).

Properties	TiC	TiN
Molecular weight	59,9	61,9
Type of crystal structure	NaCl	NaCl
Unit cell parameter (nm)	0,4318-0,4328	0,4240-0,4249
Melting point (K)	3340-3530	3223
Density (g/cm^3)	4,90-4,93	5,39-5,44
Thermal Conductivity ($Wm^{-1}K^{-1}$)	17-24	29
Coefficient of thermal expansion ($10^{-6}/K$)	7,40-7,95	9,35
Vickers micro-hardness (GPa)	30-32	20-20,5
Modulus of Elasticity (GPa)	315-450	251

Table1 . Properties of TiC and Ti. Adapted from (GUO, R. et at., 2002).

It was shown by Zhang (1993) that the properties of Ti(C, N) are largely influenced by the ratio (N / (C + N)). Physical properties were investigated as a function of nitrogen content and showed that electrical and thermal conductivities increase with increasing nitrogen content, while micro-hardness decreases. Varying the carbon content significantly influences the final morphology and properties of Ti-based hard metals (C, N). Proper control of the carbon content can improve wettability between the ceramic grains and the binder phase, reducing oxides in the raw powders (RAJABI; GHAZALI; DAUD, 2015). However, excess carbon can cause the formation of graphite and decrease mechanical properties, especially along the boundaries of the ceramic particles. In Ti-based materials (C, N) this excess generates pores and carbon segregation, which results in decreased hardness and fracture toughness. In addition, the addition of carbon can alter the dissolution behavior of carbides (SEO; KIM; KANG, 2011).

Cheng *et at*. (2011) studied the influence of carbon content on the mechanical and tribological properties of TiCN coatings deposited using the large area filtered arc deposition (LAFAD) technique and concluded that carbon directly influences the material's properties. The effective hardness and modulus of TiCN as a function of the C content in the coatings are shown in Figure 4, where the effective hardness and modulus increase with increasing C content in the coatings, reaching a maximum of 39.5 GPa at a C content of 2.8 at%. Above 2.8 at% the hardness decreases linearly with the further increase in C content.

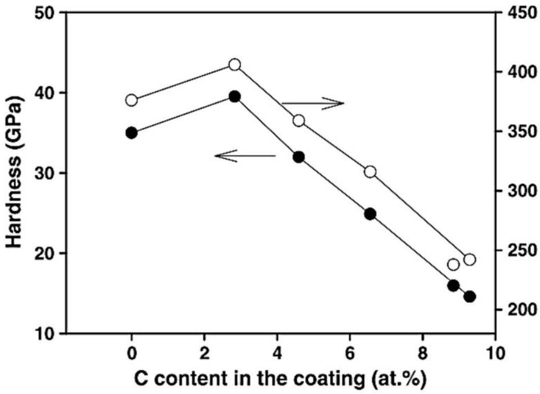


Figure 4 - Effective hardness and modulus of TiCN as a function of C content in coatings. Adapted from (CHENG *et at*., 2011).

In Figure 5, the friction coefficient and wear rate of TiCN coatings in the steady state as a function of C content are shown and state that the wear rate of TiCN coatings is strongly dependent on the C content. For pure TiN coatings, the wear rate is around 2.5 × 10-6 mm^3/Nm. However, after adding 2.8 at.% C, the wear rate reduced by 62% to 9.5 × 10^{-7} mm^3/Nm (CHENG *et at*., 2011).

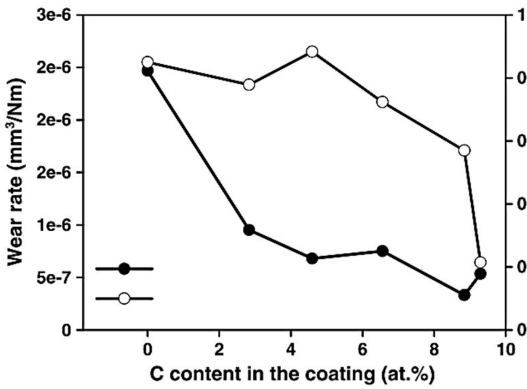


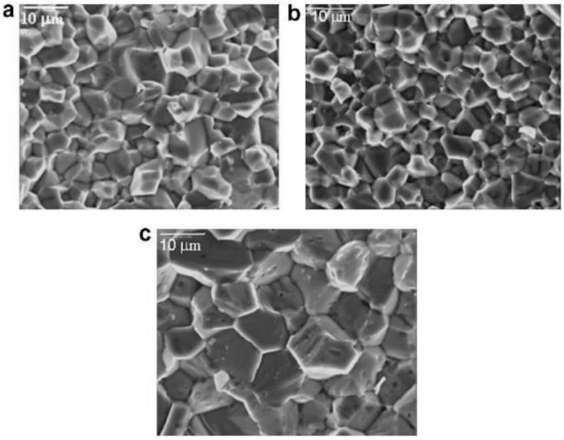
Figure 5 - Coefficient of friction and wear rate of TiCN coatings as a function of the C content in the coatings. Adapted from (CHENG et at., 2011).

The inclusion of carbon atoms in the TiN network substantially increases hardness and reduces the coefficient of friction. Titanium carbonitride (TiCN) has high chemical stability and superior mechanical properties, such as a low coefficient of friction (0.21), high hardness (HV 2500-3000), high melting point (3050 °C), high thermal conductivity (35 $Wm^{-1}K^{-1}$) electrical conductivity and excellent wear resistance. The better wear properties of TiCN compared to TiN are attributed to its greater hardness and the presence of carbon, which acts as a lubricant, resulting in less friction and wear (AJIKUMAR *et at.*, 2012; MATSUDA, 2020; SIOW *et at.*, 2013).

Narasimhan *et at.*, (1995) .found that the TiN coating had larger crystallite sizes and better adhesion properties than TiCN, but TiCN had a higher microhardness than TiN The smaller grain sizes of TiCN produce a smoother and slightly higher surface lubricity than TiN and therefore the coefficient of friction of TiCN is constantly lower than that of TiN and the TiCN coating can significantly reduce the cutting force better than the TiN coating. It also reported that TiCN-coated cutting tools have better wear resistance and longer service life than TiN-coated cutting tools when turning AISI 4340 steel. However, TiCN becomes thicker with increasing nitrogen content.

Despite the interesting properties of titanium carbonitride, a sintered body of pure TiCN is hardly ever used due to its brittleness and low toughness. Therefore, TiCN is generally used as a hard phase in carbide matrices or as composites for the most demanding applications, where it constitutes the hard phase and is alloyed with nickel and/or cobalt, for example, to form a hard and wear-resistant material (CARDINAL *et at.*, 2009).

Cardinal *et at.*, (2009), synthesized pure TiCN at Hot Press (HP), where TiC and TiN powders were mixed for 24 hours and then hot pressed in a graphite matrix under argon for 1 hour at 1850 °C, at a pressure of 50 MPa. Samples with a diameter of 37 mm and a height of around 5 mm were obtained. Three Ti (C, N) compositions were studied (70/30, 50/50 and 30/70 at%). Table 2 shows the properties obtained with the different proportions. As expected, hardness increases steadily with increasing carbon percentage, since TiC hardness (3000Hv) is higher than TiN hardness (1800Hv), results confirmed by Chen *et at.*, (2000) and Gibas *et at.*, (1997). However, the fracture toughness and flexural strength reach the maximum value for the $TiN_{0.5}C_{0.5}$ composition. In fact, the $TiN_{0.5}C_{0.5}$ composition has the best microstructure compared to the other compositions, shown in Figure 6. The elastic moduli are quite similar, except for the $TiC_{0.7}N_{0.3}$ composition, which shows the best value of 510 GPa.



Scanning electron microscopy of the fracture surface of TiCN-based materials: a) TiC0.3N0.7 b) TiC0.5N0.5 and c) TiC0.7N0.3. Adapted from (CARDINAL et at., 2009).

Zhang *et at.* (2006) studied some variations in the composition of TiCN and found that carbide matrices with $TiN_{0.5}C_{0.5}$ had greater hardness and transverse rupture strength than the others, which is due to the microstructure with a finer and more dispersive hard phase and a thinner edge.

Kang *et at.*, (2020), sintered samples with pure TiCN, as well as other samples with combinations of carbides (WC, Mo_2C, TaC), and pre-bonded solid solution (Ti, W, Mo, Ta) (C, N), in a vacuum sintering furnace. As the temperature reached 1150°, nitrogen

	p (%)	Network parameter (nm)	K_{IC} (MPa.m$^{1/2}$)	σ_f (MPa)	$HV_{0,3}$ (kg/mm^2)	E (GPa)	Electrical Resistivity (μΩcm)
$TiC_{0.3}N_{0.7}$	>98	0,4265	5,4 ± 0,62	360 ± 30	1740 ± 150	467	52,3
$TiC_{0.5}N_{0.5}$	>98	0,4280	6,3 ± 0,03	435 ± 10	2100 ± 165	473	76,6
$TiC_{0.7}N_{0.3}$	>98	0,4296	5,7 ± 0,1	330 ± 40	2120 ± 84	510	95,1

Table2 . Mechanical properties of TiC + TiN powders hot pressed at 1850 °C. Adapted from (CARDINAL et at., 2009).

	Size (μm)	Sintering pressure (MPa)	*p* (%)	HV (kg/mm^2)	K_{IC} (MPa.m$^{1/2}$)	Transverse Breaking Strength (MPa)
TiCN	3,0	35	96	1760 ± 18,5	5,2 ± 0,17	650

Table3 . Mechanical properties of the materials studied by Kang et at. (2020). Adapted from (KANG et at., 2020).

was introduced at a flow rate of 50 L/min. Finally, the temperature was maintained at 1450 ºC for a reaction time of 2h and then cooled under vacuum. They reported that the pure TiCN samples have high hardness and that wear resistance depends on hardness and other comprehensive mechanical properties of the materials. The relatively rough surface and the high coefficient of friction bring a higher shear force to the particles. On the other hand, the low strength and toughness cannot effectively prevent the generation of cracks and flaking of the particles, leading to intense abrasive wear. Table 3 shows the properties found.

Liu *et at.* (2017) sintered pure TiCN using SPS at a temperature between 1600 °C - 2200 °C and achieved relative densities above 90% from a temperature of 1750 °C. They reported that with the increase in relative density from 90% to 96.43% the Vickers hardness also increased from 4326 HV to 6762 HV, respectively. Figure 7 shows a micrograph of the morphology of the samples sintered at 1600 °C and 1700 °C.

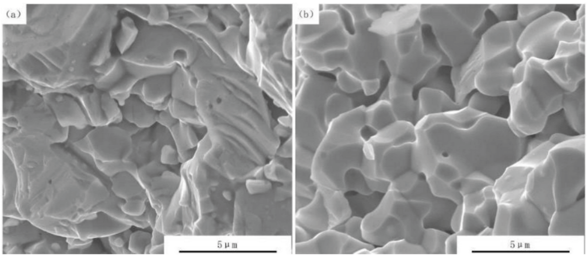


Figure 7. SEM images of fracture morphologies at (a) 1600 °C and (b) 1700 °C (isothermal plateau of 30 min, and applied pressure 50 Mpa). Adapted from (LIU et at., 2017a).

Zgalat-Lozynskyy *et at.* (2011) sintered pure nanostructured TiCN powder using SPS, and presented some properties of the sinter, shown in Table 4. Samples were sintered in linear and non-linear regimes and at temperatures up to 1600 °C.

Properties	TiCN	TiCN
SPS regime	Non-linear	7 MPa-200 °C/min
Final Temperature	1400	1600
Density (g/cm^3)	0,98	0,94
Grain size (nm)	150	320
Fracture toughness (MPa.m$^{1/2}$)	3,2	-
Vickers hardness (GPa)	21,6 ± 0,9	11,3 ± 2,4
Vickers Nanohardness (GPa)	33 ± 2,4	-
Modulus of Elasticity (GPa)	378	-

Table4 . Properties of TiCN sintered via SPS. Adapted from (ZGALAT-LOZYNSKYY; HERRMANN; RAGULYA, 2011).

TICN APPLICATIONS AND RELATED TECHNOLOGIES

Since the development of the liquid-phase sintering technique for the manufacture of composite materials based on carbides and iron group metals, it has been possible to achieve a highly effective combination of hardness and toughness. This innovation has allowed these materials to be used in a wide variety of cutting and drilling tools, making them essential for the hard metals industry (LENGAUER, 2000).

In terms of cutting performance, TiCN's high enthalpy of formation provides greater resistance against material build-up on the tool edge, fouling and pore formation, resulting in a longer service life. In addition, reduced flank wear when cutting hardened steels at high speeds improves operational stability and increases productivity. Thus, TiCN-based cutting tools are widely used in high-speed milling, roughing and semi-finishing of carbon alloys, stainless steels, high-strength low-alloy (ARBL) steels, as well as steels used in prosthetics. These tools guarantee excellent surface finish and close tolerances, even on difficult-to-machine materials such as superalloys, for which conventional TiC-based hard metals are not suitable (ZHANG, 1993).

Titanium carbonitride has a wide range of applications in highly demanding mechanical and thermal environments. As an ultra high temperature (UHTC) ceramic material, it has been widely used in cutting tools, semiconductor transistors for gate electrodes, high wear resistance coatings in aggressive conditions, optical devices and refractory crucibles. In addition, its application extends to low-barrier Schottky diodes and high-temperature furnaces, due to its thermal resistance and chemical stability. TiCN is progressively replacing high carbon steels in machining processes, as it maintains high mechanical strength even at extreme temperatures. Its durability and tolerance to mechanical impacts make it ideal for ceramic-coated brake discs, extruded shafts subjected to cold turning, shaft grooves in gearboxes and coatings for vertical lathes and high-speed tools (AKINRIBIDE *et at* .., 2020).

In addition to applications in cutting tools and protective coatings, titanium carbonitride also plays a key role in the automotive industry. The carbonitriding process is widely used as a thermochemical treatment to improve the surface hardness of steel components, making them more resistant to wear. This treatment consists of enriching the surface layer of the components with carbon and nitrogen by heating them in a controlled atmosphere within the austenitizing temperature range. After this process, the parts are tempered to induce martensitic transformation, which increases their mechanical strength. This treatment is widely applied to power transmission parts, such as gears and components subjected to high mechanical stress, ensuring greater durability and operational performance (KAROLAK *et at* .., 2017)

Coatings obtained by *chemical* vapor *deposition* (CVD) based on titanium carbonitride (TiCN) have been developed to improve the mechanical properties of cutting tools while keeping the carbide interface intact. These coatings provide improved adhesion, greater toughness and resistance to wear, making the tools more durable. On the other hand, coatings obtained by *Physical Vapor Deposition* (PVD) provide greater resistance to wear, mainly due to their high hardness. In addition, the residual compressive stresses generated by PVD coatings increase the toughness of the cutting edge and resistance to thermal cracks, improving tool performance in severe conditions (SANDVIK, 2020).

Layers of carbides, nitrides and carbonitrides of IVB group transition metals, produced by CVD and PVD, are widely used to extend the useful life of carbide tools used in cutting and milling operations. Currently, around 90% of cutting tools are coated, as the surface of these inserts must be highly resistant to interaction with the workpiece (e.g. steel), as well as being resistant to oxidation and having excellent adhesion to the sintered substrate. The material itself must also have high hardness, mechanical strength and fracture toughness. Over the years, various binary, ternary and quaternary compounds have been studied and applied industrially, most of which contain titanium (Ti) due to its beneficial properties in these coatings (SENNA *et at* .., 1997)

After the development of the first titanium nitride (TiN) monolayers, multilayer systems emerged, composed of TiN, titanium carbide (TiC), TiCN and aluminum oxide (Al_2O_3). Currently, the most widely used multilayer systems are TiC/TiCN/TiN and TiN/TiCN/TiN, as they are more resistant to wear and thermal crack formation than TiN monolayers. Although TiCN is highly efficient at interacting with the workpiece, the top layer is often made up of TiN, due to its characteristic golden-yellow color, which makes it easier to identify wear. In addition, Al_2O_3 is widely used to provide a tough ceramic interface and can be applied either directly in contact with the workpiece or in multilayer systems such as Al_2O_3/TiN. Most of these coatings are deposited by CVD due to their excellent adhesion and thermal stability (SU; KAO, 1998).

The TiCN coating makes the rotary cutting tool harder and more suitable for applications in abrasive materials where greater resistance is required (SANDVIK, 2020). Figure 8 shows hard metal inserts with TiCN layers applied.

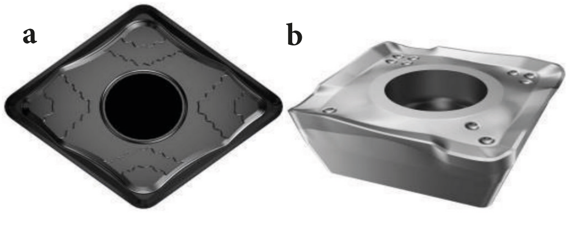


Figure 8. Carbide inserts with a) CVD coating b) PDV coating. Adapted from (SANDVIK, 2020).

Figure 9 highlights a microstructure of a TiN / TiCN / Al_2O_3 / ZrCN coating produced by WIDIA (2020) for a type of carbide used for turning steels. Figure 10 shows a microstructure of a WC / Co coated cutting tool for turning steel (WIDIA TN250) where the layer is made up of three sub-layers. Figure 11 shows a real image of one of these tools in operation.

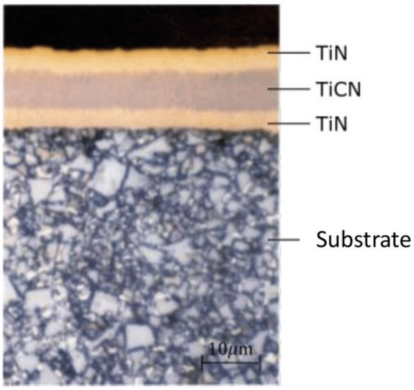


Figure 10. Microstructure of a WC/Co coated cutting tool for turning steel (WIDIA TN250) where the layer is made up of three sub-layers. Adapted from (LENGAUER, 2000).

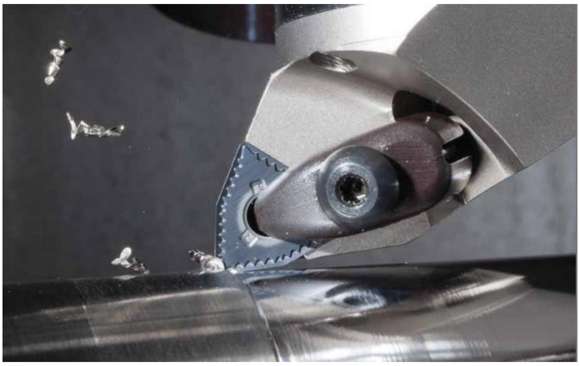
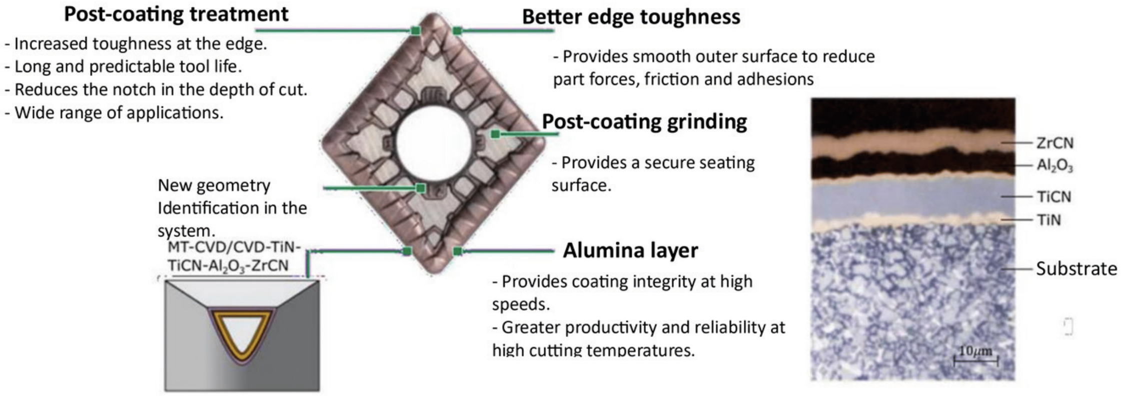


Figure 11. Real image of a carbide insert with carbonitride coating in a turning operation. Adapted from (WIDEA, 2020).


Post-coating treatment
- Increased toughness at the edge.
- Long and predictable tool life.
- Reduces the notch in the depth of cut.
- Wide range of applications.

Better edge toughness
- Provides smooth outer surface to reduce part forces, friction and adhesions

Post-coating grinding
- Provides a secure seating surface.

New geometry Identification in the system.

MT-CVD/CVD-TiN-TiCN-Al_2O_3-ZrCN

Alumina layer
- Provides coating integrity at high speeds.
- Greater productivity and reliability at high cutting temperatures.

Figure 9. Microstructure of a WC/Co coated cutting tool for turning steel. Adapted from (WIDIA, 2020; LENGAUER, 2000).

PROCESSING AND STRUCTURAL MODIFICATION OF TICN FROM THE ADDITION OF NB, ZR TA AND GRAPHENE

One of the most common ways to prepare Titanium carbonitride is to *hot press* (HP) a mixture of TiC and TiN powders in a vacuum or argon atmosphere at 1700-2400 °C (CHEN *et at*., 2000; ETTMAYER; KOLASKA, 1995). One of the most common ways of preparing titanium carbonitride is to hot press (HP) a mixture of TiC and TiN powders in a vacuum or argon atmosphere at 1700-2400 °C. In recent years, the pulsed plasma sintering (SPS) method has been used to densify Titanium carbonitride powders at a relatively lower temperature, as well as a shorter sintering time compared to conventional sintering methods (ROMINIYI *et at*., 2020). Some ways of synthesizing titanium carbonitride (TiCN) are presented below.

Wu *et at*. (2019) synthesized titanium carbonitride by a two-step carbothermic reduction method. A single-phase precursor TiC_nO_{1-n} was first prepared from the reaction between titanium dioxide and carbon black in an argon atmosphere. The precursor was then used as a raw material to prepare TiC_xN_{1-x}. To produce TiC_xN_{1-x}, the precursor was further carbothermally reduced and nitrided in a nitrogen gas atmosphere. TiC_xN_{1-x} was obtained at a temperature of 1773 K for 4 h in the first stage and 1873 K for 8 h in the second stage. Figure 9 shows some scanning electron microscopy (SEM) images of the products obtained.

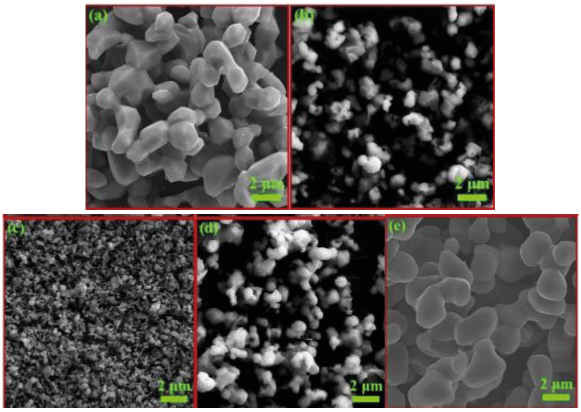


Figure 9. SEM images of the products obtained at 1873 K for 8 h: a) TiN; b) TiC0.3N0.7; c) TiC0.5N0.5; d) TiC0.7N0.3; e) TiC. Adapted from (WU *et at*., 2019).

In a study by Zhang *et at*. (2020b), the formation of Titanium and Niobium carbonitrides was identified. A hot-rolled pipeline steel was used, which was reinforced with the addition of microalloying elements such as Nb and Ti. Cylindrical specimens with a diameter of 5 mm and a length of 10 mm were machined from the experimental steel. A dilatometer was used in the austenitizing process, which involved increasing the temperature to various austenitizing temperatures at a rate of

5° C/s for 600 s, followed by cooling to room temperature at a cooling rate of 1° C/s. Figure 10 shows a microscopy taken of the samples showing the points where the carbonitrides formed.

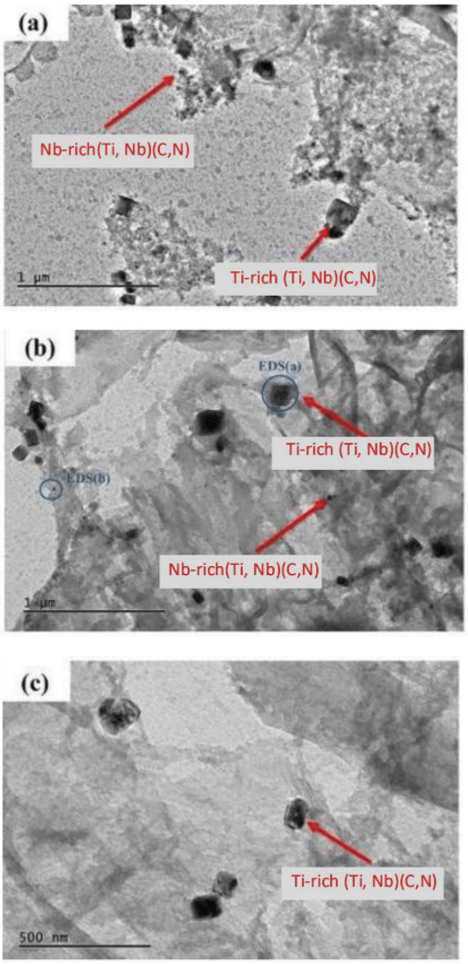


Figure 10. The two types of precipitates in the samples after being kept at the different austenitizing temperatures for 600 s: (a) 950 °C, (b) 1050 °C and (c) 1150 °C. Adapted from (ZHANG *et at*., 2020)

El Azhari *et at*. (2020) synthesized hard metal samples (WC-6%Co) by depositing coating/substrate systems consisting of Titanium Carbonitride (TiCN) and Zirconium Carbonitride (ZrCN) layers. The films were deposited in a hot-wall CVD reactor using metal chloride, acetonitrile and hydrogen reagent at temperatures between 885 and 930 °C (moderate temperature CVD process, MT-CVD). Tests were also carried out on industrial milling inserts, and it was found that systems coated with ZrCN exhibit higher mechanical integrity than TiCN counterparts, the main reasons being relatively different thermal residual stresses generated during CVD cooling as a result of the dissimilar coefficient of thermal expansion between the coating and the substrate, as well as the intrinsic cohesive strength of the coatings studied. The schematic model of the synthesized samples is shown in Figure 11.

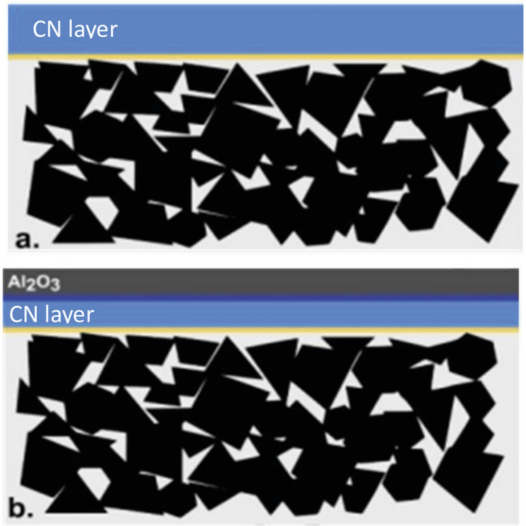


Figure 11. Layer assembly sketch for the coating/substrate systems studied: a) Single-layer [(C, N)-s] coated cemented carbide, including a thin TiN interlayer (yellow) deposited on the substrate, prior to the deposition of the CN coating. b) Multi-layer [(C, N) -m] coated cemented carbide where, in addition to the aforementioned TiN layer, an interlayer of Ti (C, N, O) (dark blue) is deposited on the CN layer, prior to the deposition of Al_2O_3. Adapted from (EL AZHARI *et at*., 2020)

Figure 12 shows the separate parts of the carbide and carbonitrides used, as well as transverse cracks, nucleating on the surface and propagating into the substrate, which were evident in both cases after indentation.

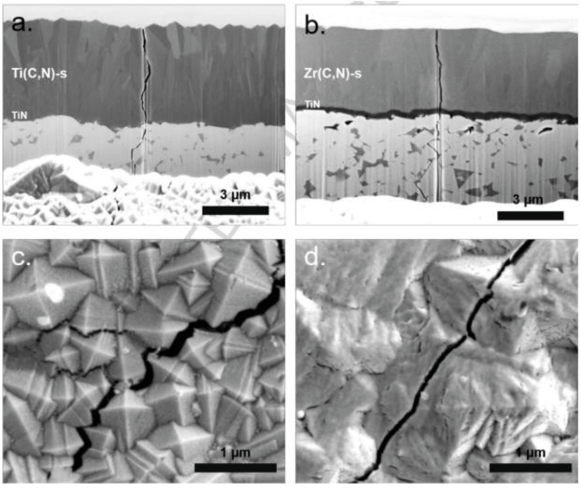


Figure 12. SEM images: a, b) Cross-sections in circumferential cracks (located at the edge of residual impressions) induced by spherical indentation under an applied load of 2200N for TiCN-s and ZrCN-s specimens, respectively. c, d) Referred circumferential cracks for TiCN-s and ZrCN-s specimens, respectively. Adapted from (EL AZHARI *et at.*, 2020).

Graphene has been widely explored to improve the functional and mechanical properties of metal matrix composites (MMCs) for a wide range of applications due to its optimal mechanical, electrical and thermal properties (CHEN; JIANG, 2020; MADHANKUMAR; ANTHONY XAVIOR, 2024). Many attempts have been made to improve the fracture toughness of ceramics through the use of grain nanostructuring and the addition of reinforcements (GAVALDA-DIAZ *et at.*, 2025) . These methods have the similar aim of providing newly formed intrinsic grain boundaries or extrinsic interfaces that prevent dislocation movement and make a long path for crack propagation (CHENG CHEN, 2024; LIU, *et at.*, 2017)

The mechanisms responsible for the increase in fracture toughness in graphene-reinforced ceramic matrices can be classified into three main categories: (1) crack deflection, (2) crack containment and bridging and (3) pull-out of graphene from a few layers (SHI *et at.*, 2023)). Crack deflection occurs when the natural propagation of the crack is altered when it encounters a slab of graphene, diverting its trajectory to a path of least resistance. This happens when the crack's energy is not sufficient to pass directly through the barrier imposed by the graphene, resulting in it being redirected along the material's surfaces. This effect is particularly effective when the graphene nanoplates are well distributed in the ceramic matrix, as these structures act as highly resistant physical barriers, preventing the direct propagation of the crack and contributing significantly to increasing the strength of the composite.

The greater effectiveness of the stiffening mechanisms of graphene nanoplates over other fibers or reinforcements stems from their ability to completely enclose the grains due to their 2D nature, which leads to a larger interfacial area with the matrix and reduces the lower energy pathways available for crack propagation. However, nanoplates also have intrinsic energy dissipation mechanisms, i.e. independent of the matrix, which make them ideal reinforcements for providing toughness (NIETO *et at.*, 2017)

Cui *et at.* (2020) investigated the cutting performance and failure mechanisms of the ATG (short for Al2O3 / Ti (C, N) / GNPs) reinforced ceramic cutting tool in continuous dry turning of Inconel 718, up to a cutting speed of 500 m/min, and compared the results with those of the commercial Sialon KY1540 tool. The authors concluded that the addition of GNPs to the Al2O3 / Ti (C, N) tool not only increases hardness and fracture toughness at high temperatures, but also reduces the coefficient of friction between the inserts and the workpiece. Consequently, ATG tools showed greater wear and fracture resistance and better cutting performance than the KY1540 insert when machining Inconel 718, especially at ultra-high cutting speeds (> 300 m/min). Cui and colleagues show that the main strengthening mechanisms are mixed mode intergranular and transgranular fracture, matrix grain refining, crack deflection, crack bridging and GNP pullout.

In general, it has been proven that the thermal conductivity and mechanical properties of composite materials depend to a large extent on the graphene content, the degree of dispersion and the bonding of the interface between the graphene and the metal/ceramic matrix. Low Graphene content will lead to better performance, while higher content will reduce the performance of the composite material, which is mainly due to the formation of Graphene agglomerates (SABOORI *et at* .., 2018)

Many structural materials operate at high temperatures, where good thermal properties, such as conductivity, are needed to transfer heat efficiently and thus avoid heat buildup that could lead to deterioration of structural integrity. Several studies have indirectly observed that thermal conductivity is improved with GNPs in ceramics due to improvements in densification (NIETO *et at*., 2017).

Rutkowski *et at*., (2015) directly verified a variety of thermal properties of the Al2O3-GNP composite. The composite was sintered by HP and SPS with up to 16.5% GNP by mass, to investigate the effects of GNPs on thermal stability, thermal diffusivity, specific heat and thermal conductivity. In the case of composites obtained by SPS, the conductivity is lower than for the pure alumina material and also lower than the values for the same Graphene content (except 10% by mass) and the measurement of the thermal conductivity of composites sintered with pulsed plasma shows that the type of Graphene with low content has no significant influence on the thermal conductivity.

Li, *et at*., 2014) investigated the wear performance of ZrO2 - GNP coatings prepared by a plasma spraying technique, under normal loads of 10 - 100 N. The authors found that the addition of 3.36% GNP by volume led to reduced wear volumes at all loads. A reduction of approximately 50% in wear volume was achieved under a load of 100 N.

Cui *et at*. (2018) synthesized by hot pressing Al2O3 / (W, Ti) C / Graphene, and found that the fracture toughness and flexural strength of the composite added with only 0.2% by weight of GNPs were significantly improved by about 35.3% (~7.78 MPam1/2) and 49% (~608.54 MPa), respectively, compared to the samples without GNPs, while the hardness was maintained at around 24.22 GPa. The uniformly dispersed GNPs form a network, which helps to improve the thermal conductivity of composite tools. In addition, it can also be seen that multilayer graphene (MLG) provides sufficient self-lubrication and reduces the wear rate and coefficient of friction. All these features contribute to extending tool life (CUI *et at*., 2018).

Cao, *et at*., 2017) synthesized a Ti6Al4V matrix composite with 0.5% by weight of graphene added by hot isostatic pressing at a pressure of 150 MPa at 700 °C and then isothermal forging at a forging rate of 3% at 970 °C. The results indicated that the GNPs can be introduced homogeneously into the titanium matrix. Microstructure examinations showed that the Titanium and Graphene formed TiC structures in the composite after 970°C. The authors observed that the Graphene was homogeneously distributed in the TiC particles. As a result, tensile strength and yield strength were noticeably improved by the reinforcement of 0.5% by weight of GNPs, with almost no loss of ductility.

Song *et at*., (2016) sintered pure titanium powder with multilayer graphene (MLG) with 0.5% - 1.5% by weight of graphene, via pulsed plasma sintering (SPS), at a temperature of 1100 °C, a pressure of 40 MPa, a heating rate of 150 °C/min and an isothermal plateau of 6 min. With the addition of 0.5% by weight of multilayer graphene, an increase was observed in the properties such as modulus of elasticity, hardness and yield strength of the composite produced by the addition of multilayer graphene compared to pure Ti. In this study, the fact that composites with a metallic titanium matrix reinforced with MLG showed greater hardness and modulus of elasticity was attributed to the presence of titanium carbide and titanium oxides formed during the sintering and/or powder mixing process. On the other hand, the addition of 1.5% by weight of MLG caused a slight decrease in hardness and modulus of elasticity in the composites. Multilayer graphene can be used not only as an excellent reinforcing phase, but also as an excellent lubricant phase. The significant improvement in tribological performance is attributed to the self-lubrication of MLG and the easily formed friction layer at the contact interface.

Sadegh Moghanlou *et at*., (2021) investigated the thermal diffusivity and conductivity of TiN-based ceramics with the addition of 6% graphene nanoplates. The authors sintered two samples using pulsed plasma sintering, a monolithic TiN ceramic and a TiN ceramic with added graphene, at 1900 °C for 7 min under a pressure of 40 MPa. The addition of graphene resulted in a 2% reduction in relative density, but led to a fine-grained microstructure. Another interesting fact was that the graphene reacted with the surface oxide impurity (TiO2) during sintering and converted into the transient TiC, which finally led to the formation of titanium carbonitride (TiN0.8C0.2). The TiN-6% by weight of graphene sample obtained lower thermal conductivity compared to monolithic TiN, which can be attributed to the smaller grain size of the graphene-added sample.

Studies have also shown that the addition of graphene reduces the friction coefficient and wear rate of TiAl substrates, which can be attributed to the self-lubricity of graphene and the improvement in hardness of Ti-based composite materials due to the addition of graphene (CHEN, JIANG, 2020) .a new type of transition-metal carbonitride VWCN is proposed by introducing the fourth element W into the ternary V2CN system. The value of valence electron concentration (VEC

Carbon allotrope materials such as graphene, carbon nanotubes (CNTs), carbon black, carbon fiber and graphite have been used as a source of carbide as well as reinforcement in titanium matrix composites in order to improve their mechanical properties (VASANTHAKUMAR *et at* . ., 2019)

Dispersing nanocarbon is always a challenging task due to its agglomeration during processing. There are several techniques, such as mixing, spray drying and surfactant addition for the effective dispersion of carbon sources in metal and ceramic matrix. Among all the techniques used, the ball mill has proven to be a suitable technique for uniform dispersion of nanocarbon reinforcements in the matrix (AWOTUNDE *et at*., 2019; TAPASZTÓ *et at* .., 2011)

The impact force and shear force introduced by the grinding ball can break the van der Waals force in the interlayer between the graphene sheets and break up the agglomerates. At the same time, the high-speed rotation of the grinding jar promotes uniform dispersion and homogenization of the powder. One reason why the ball mill is a popular method of mixing ceramic/metal and graphene powders is that the high shear force generated by the ball mill can be used to exfoliate the GNP from the graphite particles (MICHÁLKOVÁ *et at* .., 2014)

According to Nieto *et at*. (2017), due to Graphene's inherent energy dissipation mechanism, which inhibits crack nucleation and propagation, the addition of Graphene continues to increase toughness and flexural strength in CMC. Due to the grain refinement induced by GNP, the incorporation of the soft Graphene phase has also led to greater toughness in some studies. Graphene's excellent functional properties have a beneficial effect during processing, such as higher electrical and thermal conductivity. This leads to greater heat transfer and uniformity of the resulting microstructure, especially in conductive ceramics processed by SPS.

Graphene's excellent functional properties lead to improved electrical and thermal conductivity of metals and CMC. Due to its considerable electrical conductivity and thermal conductivity, the use of graphene can effectively facilitate the sintering process and suppress grain growth, increasing densification. GNPs can play a role as heat-conducting plates facilitating the heating process by improving current and heat distribution during the heating process, while acting as heat-dissipating plates, hindering grain growth by increasing the cooling rate during the cooling process (SUN; ZHAO, 2018) . Related to its addition to TiCN, it will be allocated to the grain contours, preventing atomic diffusion between the contours, thus preventing grain growth, in addition to preventing carbon loss. In addition, the unique structure and chemical properties of graphene are promising for improving the oxidation resistance and biocompatibility of ceramic matrix composites (NIETO *et at*., 2017).

FINAL CONSIDERATIONS

This study reviewed the state of the art of titanium carbonitride (TiCN), covering its properties, applications and sintering techniques, with a focus on pulsed plasma sintering (SPS). TiCN has high hardness and wear resistance, but its low fracture toughness limits its application in its pure state. SPS has proved to be an efficient technique for promoting high densification, minimizing structural defects and improving mechanical properties, making it a promising alternative for the manufacture of advanced composites.

The analysis of the studies indicated that the addition of Nb, Zr and Ta improves the thermal and mechanical resistance of TiCN, while the use of graphene contributes to increasing toughness, reducing the coefficient of friction and improving thermal and electrical conductivity. Controlling the microstructure and the binder phase is essential for optimizing the wear resistance and thermal stability of the material, allowing it to be applied in harsh environments.

The core-edge structure was identified as a determining factor in improving the mechanical properties of TiCN. SPS proved effective in obtaining homogeneous microstructures, reducing phase segregation and ensuring greater mechanical performance. The addition of graphene proved to be a promising strategy for limiting grain growth, reducing internal stresses and improving resistance to crack propagation, increasing the material's durability.

Improving TiCN is fundamental to expanding its applicability in sectors such as metallurgy, the automotive industry and aerospace. Future research should focus on optimizing sintering, incorporating new alloying elements and improving the use of graphene in order to improve its mechanical and thermal properties for more demanding applications.