**CRITICAL REVIEW** 



# Spark plasma sintering of ceramic matrix composite of TiC: microstructure, densification, and mechanical properties: a review

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### Abstract

The incessant quest in fabricating enhanced ceramic materials for use in aerospace, chemical plants, as a cutting tool, and other industrial applications has opened the way for the fabrication of ceramic-based composites with sintering additives which have been experimented to influence sinterability, microstructure, densification, and mechanical properties. The current research practices for the consolidation of ceramic matrix composite (CMC) have been in the utilization of metallic and non-metallic additives as a reinforcement for the ceramic matrix. The use of additives has a promising influence in ensuring the achievement of good microstructures and excellent properties. The use of metallic additives enhances the sinterability of CMC but it has a debilitating effect on its intrinsic mechanical properties, especially at high-temperature applications. Hence, its uses in a hightemperature application environment under high impact load are limited. Thus, the types and amount of additives to be added to a ceramic-based matrix composite depends on the type of application and properties desired to be achieved from the composites. One of the critical issues that have affected the properties of CMC is the type of powder metallurgy (PM) used for consolidation. PM has been experimented with to be efficient in manufacturing ceramic-based composites. Although, past review works have pinpointed diverse PM methods, viz, hot press, pressureless sintering, hot isostatic press, and spark plasma sintering (SPS), for manufacturing ceramics-based composites. Amidst these diverse methods, SPS has progressively been applied for the consolidation of ceramics, owning to its possibility of achieving a good sintered compact in a relatively short time with enhanced properties. This review focuses on the synthesis of TiC reinforced with sintering additives, with more attention on carbides as sintering additives. Carbide additives have the potential to improve microstructure, densification, and mechanical properties. In addition, future works on the consolidation and characterization of TiC are included in this review.

Keywords SPS · TiC · Microstructure · Densification · Mechanical properties

### 1 Introduction

When Ti (a transition metal) combines with nitrogen, boron, and carbon, they form a candidate of material regarded as

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ultra-high temperature ceramics, owning a melting point greater than 3000 °C [1, 2]. Since TiC has a melting point of 3160 °C, hence, it has high proficiency to be applied in an environment where alteration of temperature and load are austere, such as in the aerospace industry, high-temperature electrodes materials, cutting-tools, nuclear reactors, and highspeed cutting tools [1, 3, 4]. However, monolithically, these ceramics are not suitable for all applications as a result of their poor thermal resistance, brittle-like nature, and low fracture toughness [5–7]. More also, poor sinterability as a result of low self-diffusion coefficient has also stood as one of the factors that have limited its wider application [7, 8]. TiC ceramic matrix composite (CMC) has been reported to have enhanced properties in comparison to a monolithic TiC ceramic [4, 9, 10]. CMC is a component involving a combination of a dispersed ceramic phase (carbides, oxides, silicides) with a ceramic matrix. CMC can be reinforced by the

introduction of fibers which can be short/discontinuous fibers or long/continuous fibers. Short/discontinuous fiber composites are manufactured via conventional ceramic techniques from a non-oxide (BN, ZrC, SiC, WC, etc.) and oxide (silica, alumina, zirconia, etc.) ceramic matrix, which can be further reinforced with whiskers of SiC, AlN, and TiB<sub>2</sub>. Fibers of SiC have a high modulus of elasticity (stiffness) and high strength; these properties have made it the most common reinforcement for CMC. For long/discontinuous fiber composites, these are produced by the reinforcement of long monofilament or long multifilament fibers. The dispersion phase in the formation of continuous mono/multifilament fibers created the best strengthening impact. Generally, CMC is processed to solve the challenges of toughness in conventional ceramics [11, 12].

Lately, powder metallurgy (PM) techniques have proven to be effective for the consolidation of CMC, owning to its efficiency in producing materials made from powders with diverse chemical compositions, textures, sizes, and morphologies. This consequently produces materials that have good microstructural characteristics and enhanced properties [13, 14]. PM entails the production of net shape materials via the techniques of preparing, forming/powder shaping, consolidation, firing/sintering, and finally post-sintering [15]. Previous works have indicated that this method is suitable for the manufacturing of CMC. This is a result of its pliability and potentiality to lower the sintering temperature; it also aids the regulation of the interfacial reaction between the component of the composites [16]. Furthermore, it encourages the attainment of excellent properties via the judicious densification of the as-received powder and the successive forming/ compaction and sintering processing [17–19].

Ceramics like WC, TiN, SiC, TiB<sub>2</sub>, and ZrC and metals like Ni, Mo, Ti, and Co are among the frequent additive for TiC that have been used for the consolidation of ceramics because they inhibit grain growth, lower sintering temperature, and enhance the materials' fracture toughness [20-22]. The achievement of ultrafine microstructures is highly necessary since enhanced physical and mechanical properties can be achieved from it [23-26]. There is the formation of a solid solution or the elimination of oxide impurities by carbidebased sintering additives which enhance microstructure, wettability, densification, sinterability, and eradicate grain growth. More also, the toughening and strengthening of TiC have been experimented with to be enhanced by the addition of carbide-based additives, viz, SiC, WC, and ZrC [9, 27–32]. Conventional means of consolidating this ceramic, viz, hot press, hot-isostatic press, and pressureless sintering, leads to excessive grain growth, due to long dwelling time, and these consequently have negative impacts on its microstructure and mechanical properties [33, 34].

One of the ways to eliminates grain growth and simultaneously produce dense bulk TiC materials besides exceptional properties is by the application of spark plasma sintering (SPS) technique. SPS is a modern means of sintering that has been channeled for the consolidation of fine-grained ceramics, because of its ability to carry out sintering effectively in a short time, at a higher heating rate with lower sintering temperature [35–37]. SPS uses a direct current that passes through the powder compact and the die directly. The capability to enhance ceramics' sinterability and inhibit grain growth is another important factor of using SPS for the consolidation of a ceramic-based material. Hence, a significantly short sintering time is mostly required to achieve homogenous fine grains and improved mechanical properties [38–41]. Previous works have also discovered that the full densification of ceramic materials can be enhanced by SPS in a very short processing time. In addition, the combinational effects of applied pressure, joule heating, and concurrently local plastic deformation of grains during sintering have been the major reason for the achievement of full densification [42-46].

This article is a review on TiC reinforced with sintering additives consolidated by SPS. An observation will be carried out on how carbide-based material additives have influenced sinterability, densification, microstructure, and mechanical properties of a TiC ceramic-based matrix. More also, the review finally concludes by pinpointing some extra work needed in some areas to completely make TiC CMC suitable for potential industrial utilization.

### 1.1 Structures, properties, and application of TiC

Covalent, ionic, and metallic bonds exist in metal-like carbide structures. Hence, this has made TiC exhibit both ceramic and metallic features, with a typical crystal structure depicted in Fig. 1. Densification of a pure TiC can only be achieved at an elevated temperature under a subjected high pressure [48]. TiC has exceptional properties such as high corrosion, thermal expansion, low thermal expansion, wear resistance, low high thermal conductivity, and high stability in a severe environment. The production of diverse morphologies of TiC



Fig. 1 Crystal structure of TiC, adapted with permission from [47]

ceramics is conditioned by various factors. Generally, inherent factors such as thermodynamic features comprising phase transformation entropy and surface energy as well as intrinsic crystal structures all contribute to the interplay of each particle and finally leads to the final growth morphologies of TiC. Some external factors such as thermal, mass transportation, and growth mechanisms in the melts influence these morphologies (TiC) as well [49, 50]. All these attributes have made TiC a high candidate for nuclear reactors and aerospace materials, turbine blades, etc. [51].

### 1.2 Powder metallurgy

PM is an incessant and quick emerging technology on the fabrication of huge diverse shapes of ceramics and its composites materials [52]. The development of PM industry in the past decades is hugely accountable to the saving costs connected to the production of near-net shape of materials in contrast to other fabrication techniques especially forging or casting. The efficient application of powder metallurgy is primarily due to its capability to manufacture materials with complex shapes, and the efficient way in which ceramic matrix with its reinforcement can be combined for both finished and partial finished products. Four essential stages are required for this techniques, viz, production/preparation of powder, mixing/blending of powders along with other compositions (binder or sintering additives), shaping of the powder in the die at ambient temperature, and lastly accompanied with sintering operation to manufacture into desired form and dimension so as to minimize post-machining operation and for large production of materials [53]. Mechanical alloying is a metallurgical processing technique that entails cold welding and breaking of powder particles by the high impact energy of the milling ball. This technique is a practice that involves the mixing of material in order to obtain uniform composites. The mechanical alloying techniques start with powders blending in the accurate amount and filling the mill with the blended powders besides with the grinding media. The desired extent of time is determined by the balance state of the organization of various powder particles and also by the size of powder particles needed to be achieved. The ball milling is a method by which a mixture of the powder is subjected to high impact with the milling media, and this causes the breakage of the preliminary materials into a smaller size. To obtain an enhanced milled material, researchers have made it known that wet milling operation is more superior to dry milling. The wet milling operation eliminates cold welding and the coalescence of powder particles. Generally, the blending of powders via powder metallurgy methods cannot produce a uniform mixture. The incorporation of mechanical alloying to manufacture ceramic matrix composites remains an excellent method in obtaining enhanced uniformity. Usually, the disparity in size of these particles happens between the ceramic matrix and the reinforcing additives [54, 55]. The mixed and blended powders particles are consolidated via various consolidation processes, viz, hot pressing, conventional sintering, hot isostatic pressing, and spark plasma sintering. Nevertheless, in the obtainment of some definite features, some secondary techniques are engaged on the CMC, e.g., hot rolling and hot extrusion. Hence, the fabrication of materials via PM eliminates secondary operations because the products from PM have near-dimensional tolerances in contrast to other conventional techniques. A representation of the processes involved in PM is shown in Fig. 2; each process is cost-effective, flexible, efficient, and environmentally friendly [52].

#### 1.2.1 Spark plasma sintering

Spark plasma sintering (SPS) is also regarded as pulsed electric sintering (PECS), plasma-assisted sintering (PAS), and field-assisted sintering (FAS). SPS is a technique that allows the passage of pulsed DC through the powders (conducting) and the die concurrently during sintering. The spark that is initiated at the particle boundary is understood to provide quick heating, which in consequence promotes the sintering rate [56, 57]. There is attainment of maximum densification of powder via the spark effect pressure, electric field diffusion, and joule heating [58, 59]. This technique is most appropriate for the consolidation of nanopowders because it inhibits grain growth as a result of its short sintering time compared to hot pressing and other conventional sintering that make uses of long dwelling time to achieve their consolidation. SPS has predominance in application over conventional consolidation technique due to enhanced densification of materials due to short sintering cycles, high reproducibility, simplicity of operation, dependability and safety, precise control of consolidation energy and also quick sintering speed, high heating rates, and reduce sintering temperature that is achievable [60, 61]. The non-conventional technique via SPS involves pouring the starting powders into a mold made of graphite, and the sintering process is achieved through the concurrent application of electric current and external pressure [16, 62].



Fig. 2 Powder metallurgy method

Nevertheless, restricted sample geometries and high costs are the main drawbacks of SPS [63, 64]. Hence, for large production of diverse geometries with a satisfying performance at inexpensive prices, the sintering routes cannot be applied [65].

### 1.3 Limitations and challenges of TiC

The difficulty in densification as a result of the poor sinterability and high covalent nature of TiC has created some challenges in its consolidation. Monolithic application or singlephase nature of TiC is limited owning to poor fracture toughness, brittle-like nature, and poor thermal shock resistance [4, 6, 7, 21, 66, 67]. These challenges have limited its wider application in chemical and high-temperature conditions. The oxide scale present on the surface of ceramic powder surfaces lowered the surface energy which consequently decreases its densification [68]. Some undoped TiC has some peak densification of 99.4% via SPS [69], but its combined mechanical properties make it unfitting for some application. Hence, the use of sintering additive in addition to the use of modern techniques of sintering viz SPS has been observed to minimize these challenges [28, 67, 70–72].

### 2 Microstructure, densification, and mechanical properties of TiC

### 2.1 Pure TiC without additives

Monolithic TiC has yielded certain promising densification and occasionally good properties; however, it has some deficiency in properties when compared with a doped TiC which has been experimented with to produce enhanced densification and excellent mechanical properties (most especially fracture toughness) [21]. The validation of these claims is stated below in the outcome of previous works.

Namini et al. [73] attained a densification of 99.23% of a monolithic TiC via SPS at 1900 °C for 7 min under 40 MPa; the densification and mechanical properties (hardness) of TiC were reported to be enhanced when doped with WC. Song et al. [4] stated a density of 98.2% for the undoped TiC when it was hot-pressed at 2100 °C; the microstructural observation of the monolithic TiC indicated the formation of nonstoichiometric  $TiC_{1-X}$  besides graphitized carbon phases which had some negative effects on the ceramics sinterability owning to the oxide trapped in the powder sample. Murthy et al. [74] achieved densification of 97.5% for the TiC matrix but got improved to 98.2% when doped with WC. Sribalaji et al. [30] observed that the densification for a monolithic TiC at 1600 °C for 5 min under 50 MPa could attain a value of 89%. Pazhouhanfar et al. [75] observed a monolithic TiC could achieve a densification value of 95.5%. Babapoor et al. [69] reported a near theoretical density of 99.4% when it was consolidated by SPS at 1900 °C at 7 min 40 MPa; this achievement was credited to the usage of finer powder particles, but the relative density dropped to 92% at 2000 °C due to porosity increment by the irregular grain growth of TiC at the elevated sintered temperature. This irregular grain growth was also noticed by Cheng et al. [36]; they observed that through the use of pressureless sintering at 2300 °C, densification of 96% could be attained for the undoped TiC.

Monolithic TiC or single-phase components of TiC can be used rarely in some less corrosive and high-temperature environments such as in the chemical plant and thermal plant. However, their usability in these areas is limited as a result of poor fracture toughness and microstructure. This has prompted the introduction of dopants/sintering aid/sintering additives in the TiC ceramic matrix, which have been observed to possess a combination of good mechanical properties and microstructure [27, 29, 30, 76]. The attainment of the different relative densities of monolithic TiC using SPS, hot press, and pressureless sintering is graphically depicted in Fig. 3.

## 2.2 The influences of carbide additives on the consolidation of TiC ceramic matrix

Carbide additives such as SiC, WC, ZrC, and B<sub>4</sub>C have proven to enhance the quick solid solution of TiC CMC and this increase in solid solution enhances mechanical properties and ensures the achievement of uniform microstructure [77]. Carbide additives eliminate the coalescence of TiC grains and usually, at post sintering examination, carbide additives remain smaller in size than TiC grains. With a regulated increasing sintering temperature, carbide additives decrease porosity and are favorable to the densification of the microstructure. The use of nano-sized carbide-based additives usually prompts the attainment of finer microstructure, and the fracture mode is mostly intergranular in this typical microstructure [68]. The examination of the fracture surface depicted that there is usually a location of fine carbide-based additives (CBA) grains on the grains of coarse TiC grains. After the cracks spread and extend to a sub-micro grain of CBA or TiC, there will be difficulty for the cracks to cross via the particles. Hence, crack propagation and deflection will occur along the grain boundaries of TiC and SiC which causes improvement in the fracture toughness as stated by Wang et al. [21] and Luo et al. [35]. Other researchers have also credited the deflection of crack and its propagation to the residual stress created by the alteration in elastic modulus and thermal expansion coefficient between TiC and CBA [78, 79]. Therefore, the promotion of the toughening mechanism of TiC is enhanced by the crack deflection, particle dispersion toughening, solid solution toughening, and crack bridging by carbide-based sintering additives [80]. Crack propagation is inhibited by the enhancement of the grain boundary and this

**Fig. 3** The densification of monolithic TiC



consequently enhances the fracture toughness of the ceramic composites [81, 82]. As a result of the good wettability between TiC and carbide-based sintering additives, the bonding strength of the grains increases hence, ensuring good compatibility for solid formation [21].

## 2.3 Spark plasma sintering of TiC matrix composites using carbide-based material as sintering additive

Cabrero et al. [27] carried out an observation of different volume contents of SiC (0-50%) on the microstructure and mechanical properties of TiC. The composites were sintered at 1600–1900 °C for 5 min using SPS. From the microstructural analysis, there was no indication of reaction between TiC and the reinforced SiC during sintering. A synonymous observation was made in past studies when the binary system of TiC-SiC was consolidated (indicating no solid solution) [35, 83, 84]. The incorporation of SiC into the TiC ceramic matrix was observed to inhibit grain growth. The relative density of the composite increases with temperature; hence, the maximum densification was reached at 1900 °C (with a relative density of 96–98% for the entire composites). But the relative density was lowered at sintering temperatures of 1600 °C and 1700 °C, specifically for the samples comprising of large SiC content. The composites consolidated at 1800 °C produced a slim increase in the Vickers hardness in correlation with the increase in SiC content. The enhancement of the Vickers hardness of TiC-SiC composite was attributed to the volume fraction of SiC and because SiC possesses higher hardness than TiC [28]. In contrast, the samples consolidated at 1600 °C and 1700 °C experienced a reduction in Vickers hardness because the density of similar SiC content decreases under the same sintering condition. The fracture toughness for the composites (TiC-SiC) when 50% of SiC was introduced into it got to a peak value of 5.6 MPa  $m^{1/2}$  (Fig. 4), while the undoped TiC was about 3.7 MPa.m<sup>1/2</sup>. Therefore, the introduction of the second phase in TiC was observed to enhance the properties of undoped TiC. This is in concordance with various studies [78, 85, 86]. The improved fracture toughness was credited to crack deflection by the distribution of various particles [78].

Wang et al. [21] examined diverse nanosize of SiC as reinforcement in TiC ceramic matrix composite when it was spark plasma sintered at 1600 °C for 12 min under 70 MPa. The undoped TiC and the matrix incorporated with 10-20wt% of SiC all have their relative density greater than 99% but the composites with 40wt% of SiC resulted in the reduction of the relative density with a value of 92%. This reduction was attributed to the intrinsic low sinterability and a large amount of SiC content. The composites' fracture toughness firstly increased with the SiC content but it could only reach a peak value of 5.76 MPa.m<sup>1/2</sup> when 20wt.% of SiC was added to it. The attainment of this value is 90% greater in value than that of the undoped TiC. The enhancement in the fracture toughness was credited to the dispersion of diverse particles which causes deflection of cracks which is presented in Fig. 5. The Vickers hardness of the composites increases in the similitude



Fig. 4 The correlation between fracture toughness and SiC composition of TiC-SiC consolidated at 1800  $^{\circ}$ C under 75 MPa for 5 min, adapted with permission from [27].



Fig. 5 A scanning electron micrographs, depicting a crack path created by the indentation of Vicker in TiC composites. **a** Undoped TiC, **b** TiC+20wt.%SiC, **c** and TiC+30wt.%SiC, adapted with permission from [21]

of the relative density; the maximum Vickers hardness was achieved for TiC-30wt%SiC composite, but it got reduced at 40wt%SiC. The low relative density (92%) that was achieved for the same reinforcement content in the TiC matrix was the main reason for the reduction [67].

Cheng et al. [87] examined the importance of submicron SiC as a sintering additive in the TiC matrix; the composite was consolidated at 1600 °C under 50 MPa for 5 min. The different percentage volumes of submicron SiC (14.6, 27.7, 39.7, and 50.6 vol%) were used to determine its impact on the microstructure, densification, and properties of the TiC matrix. From the microstructural observation, no sintering reaction was observed, similarly observed by [84]. The grain size of SiC and TiC reduced in proportion with the increasing SiC reinforcement and the optimum microstructure was attained when 27.7 vol%SiC was reinforced. It was understood that the pinning effect of SiC particles in eliminating the grain boundary movement contributed to the retardation of TiC grain growth in some of the samples [88]. However, the porosity of the composites got elevated at increasing SiC. The peak fracture toughness of the composite was attained for the samples with 14.6% SiC; this was a result of the even distribution of SiC and its higher densification (as depicted in Fig. 6). The composites with <30 vol% SiC have a higher fracture toughness in contrast to the undoped TiC. Hence, the most enhanced fracture toughness was achieved for the sample containing 14.6 vol% SiC (5.2 MPa. m<sup>1/2</sup>); this improvement accrued by 21% in contrast to a pure TiC [36].

The composites' fracture toughness depreciated incessantly with the increment of SiC, particularly when the quantity is > 30 vol%, and this occurrence may be ascribed to the elevated porosity. In contrast to the outcomes of Ti/submicron-SiC composites achieved by Wang et al. [89], the composites' (TiC-14.6 vol% SiC) fracture toughness got enhanced by 44.4%. The improvement in fracture toughness was a consequence of the deflection and crack bridging, and more also the fracture system alteration from intergranular to transgranular form all contributed to the toughening mechanism [90].

Asl et al. [91] studied the impact of silicon carbide whiskers 0-30vol%(SiCw) on the microstructure and mechanical properties of TiC. The composites were sintered at 1900 °C for 7 min under 40 MPa. It was observed that the increment of SiCw nearly yields a linear improvement in the densification of the TiC matrix composite, excluding the composite with 10vol% SiC<sub>w</sub> reinforcement. A relative density >100% was attained by the samples having 20 and 30vol%SiCw. These densification achievements indicated that the higher the quantity of SiC<sub>w</sub> in the TiC matrix, the denser the material becomes. This is as a result of the formation of diffusion bonding at high diffusivity between TiC particles accompanied by filling of the pores. Microstructural observation revealed that the existence of SIC<sub>w</sub> in the composites contributed to the elimination of grain growth. More also, the uniform distribution of SIC<sub>w</sub> in the microstructure initiated the removal of residual stresses (owning to the disparity of thermal expansion coefficients of SiC<sub>w</sub> and TiC) and this resultantly led to the



Fig. 6 The relative density and fracture toughness of TiC-SiC composites as a function of SiC content, adapted with permission from [87]

nullification of compressive and tensile stresses in adjacent regions. The reduced densification of the samples with 10vol% SiC<sub>w</sub> was as a result of its insufficiency in the matrix, particularly in the areas where they have coalesced. More also, an increase in the disparity of thermal contraction and expansion between the adjacent TiC matrix and SiCw prohibited the full densification. The Vickers hardness varies as the SiC<sub>w</sub> increases which were reported to be in the same manner for the densification. The hardness of the monolithic TiC was 25.08 GPa, but the introduction of 10 vol%  $SiC_w$  has its hardness reduced by  $\sim 2\%$  (with a value of 24.54 GPa). Kachenyuk et al. [92] reported a contrasting result in hardness by the increasing volume of the reinforcement (SiC) in the TiC matrix, 10 vol%SiC in the composites produces the highest hardness because it contained the lowest porosity of all the sample. However, Asl et al. further stated that the hardness of the samples improved significantly when the reinforcement increased from 10 to  $30\text{vol}\%\text{SiC}_w$  and a peak hardness of 29.04 GPa was acquired. Overall, different defining roles contributed to the hardness of TiC components such as the grain size, composition of the sample, and porosity. The slim decrease in the hardness of the TiC matrix by the inclusion of 10vol%SiCw was related to the occurrence of fine pores and lowered densification [93–95]. The peak flexural strength was attained for the TiC matrix composite when reinforced with 20 vol%SiC (644 MPa). The interfacial bond features between the SiC<sub>w</sub> reinforcement and TiC matrix activated the strengthening mechanism for the flexural strength [96–98].

Fattahi et al. [71] carried out an observation on the impact of SiC<sub>w</sub> on the microstructure and mechanical properties of a binary TiC-3wt%WC composite. The composites were sintered at 1900 °C under 40 MPa for 7 min. The composition of SiC<sub>w</sub> varies between 0 and 30% and was examined in TiC-3wt%WC. The addition of 10 vol% SiC<sub>w</sub> was reported to have a degrading effect in the densification of the composite in comparison of the sample without SiC<sub>w</sub> content but relative density greater than 100% were achieved with 20 and 30 vol% of SiC<sub>w</sub> content, with the latter having the highest densification value of 103.6% as can be seen in Fig. 7a. A similar trend of results was obtained by Asl et al. [91]. The achievement of relative density greater than 100% was attributed to dense phases of non-stoichiometric TiC that were formed during the sintering operation and the precipitation and dissolution on the TiC grains by WC additive. Additionally, the creation of the (Ti,W)C phase and the introduction of SiC<sub>w</sub> to the TiC-WC composite yield a good bonding diffusion mechanism amidst the matrix powder and ensured the removal of porosity. The hardness values were in the similitude of the relative density such that the addition of 10vol% SiCw to TiC-3 wt% WC dropped the Vickers hardness by ~60% and has a value of 11.63 GPa; this is shown in Fig. 7b. Nevertheless, it was reported that by increasing the additive to 30vol% SiC<sub>w</sub>, the samples' hardness got improved with a value equal to the sample without SiC<sub>w</sub> (27.3 GPa). Shahedi et al. [91] observed a constructive impact of SiCw as an additive on the hardness of TiC composites by the hindrances in the motion of dislocations and these consequently resulted in the achievement of near full densification of the samples and a fine microstructure. Fattahi et al. further stated the effect of diverse addition of 0-30vol% SiC<sub>w</sub> viz on the flexural strength of TiC-3 wt% WC. It was reported that the optimum flexural strength was attained for the sample with 20 vol%  $SiC_w$  with a value of 694 MPa, whereas the weakest value of 368 MPa was achieved with 10vol% SiC<sub>w</sub>. The main attributes of all these mechanical properties were grain size, relative density, and porosity elimination by the interfacial bonding of the reinforcement/matrix [71, 91].

Gu et al. [68] studied the influence of  $B_4C$  on the densification of TiC matrix. It was reported that the consolidation of the undoped TiC via pressureless below 2200 °C revealed poor sinterability. Hence, the relative density of the undoped TiC got up to 72.86% at 2100 °C. However, the densification got enhanced considerably to 96.67% when the temperature was increased to 2300 °C, which inferred that the grains of the



Fig. 7 a, b The graphical representation of the effect of 0-30vol% SiC<sub>w</sub> on the densification and Vickers hardness respectively, adapted with permission from [71]

TiC were developed at the high temperature. The densification of TiC matrix got improved significantly when  $B_4C$  was incorporated into it. This improvement in densification was credited to the removal of oxides which serve as impurities on the powder surface. The same attributes were also observed when ZrC, SiC, and TaC were made as sintering additives or as a matrix [99–101]. The flexural strength and fracture toughness of the TiC matrix doped with  $B_4C$  were said to be enhanced in comparison with the undoped TiC. The core toughening mechanism for the fracture toughness was the crack deflection initiated by the reinforcement [78, 102].

Cheng et al. [67] studied the reinforcement of tungsten carbide (WC) on microstructure, densification, and mechanical properties of TiC ceramic composites. Diverse compositions of WC (0-10 wt%WC) were reinforced in the TiC matrix and they were consolidated at 1450-1600 °C for 5 min under 50 MPa. It was noted that the doped TiC with 3.5 wt% WC yielded increased densification from 98 to 100% as the sintering temperature increases to 1600 °C, but with a further increment of WC to 10%, the relative density decreased to 91%. It was further stated that no abnormal grain growth was observed with the composites of TiC + 3.5 wt% WC in contrast to the single phase of TiC. The achievement of 100% relative density was credited to precipitation and dissolution of WC in TiC grain boundaries, which prompted the diffusion coefficient of TiC grains. The incorporation of WC was also reported to improve the sinterability and wettability of the composites and consequently eliminates grain growth. Qu et al. and Namini et al. made synonymous observations [73, 103]. Cheng et al. further observed that at a lower sintering temperature of 1450 °C, a relative density of 98.2% was achieved for TiC composites when it was doped with 3.5wt.% WC in comparison with undoped TiC ceramic whose relative density was 98.4% at 1600 °C under similar pressure and dwell time [36]. This outcome depicted a reduction in the densification temperature of TiC-3.5 WC composite which was reduced by ~150 °C and no significant drop in the Vickers hardness was observed in contrast to undoped TiC. (Figure 8 revealed how the sintering temperature contributed to the enhancement in densification and hardness.)

Cheng et al. further reported the achievement of improved fracture toughness when sintered at 1450–1600 for 5 min; it improved from 3.7–4.3 MPa m<sup>1/2</sup> for the undoped TiC to 5–6.3 MPa m<sup>1/2</sup> of the doped TiC with 3.5wt%WC. The enhancement in fracture toughness of the doped TiC was recognized to the crack bridging and deflection around or along with the TiC and WC phase. The bridging or crack deflection beside the phase of (Ti, W)C was influenced by the residual stresses [21].

Namini et al. [73] performed experimentation of the influence of nanosized (0–3)wt% of WC on the mechanical properties and microstructure of the TiC matrix. The composites

were sintered via SPS at 1900 °C for 7 min under 40 MPa. The achieved results indicated that the undoped TiC reached a relative density of 99.23%. In contrast, the introduction of 1.5 wt% WC to the TiC matrix yielded lower densification of 93.05%. However, the increment of WC to 3wt% vielded peak densification of 99.85%, which was credited to the developed phase of (Ti, W)C in the microstructure of the TiC matrix and the precipitation and dissolution of WC on TiC grains [93–95]. The hardness of the composites was observed to alter in the similitude of the densification. The hardness of undoped TiC was lowered by the reinforcement of 1.5 wt%WC; however, it got improved when the WC increased from 1.5 to 3 wt% which was in the same trend as the achieved relative density. The former was a result of increased porosity in the microstructure while the latter was attributed to the higher densification due to the formation of solid solution (Ti, W)C [67, 76, 94, 104]. The flexural strength of the composite with 1.5 wt% of WC got deteriorated from 545 to 418 MPa in contrast to the undoped TiC. With further inclusion of WC to 3wt%, there was a drastic decrement in porosity consequently enhancing the flexural strength via the grain refinement. As noticeable from the microstructure in Fig. 9a, the introduction of 1.5 wt% WC to TiC ceramic matrix, the content of the porosity enlarged to ~7%. The sample containing 3 wt% WC (Fig. 9b) indicated no presence of porosity which agrees with the achieved densification outcome of 99.85%. Generally, the hardness of ceramics composites is a complex function of grain size, composition, and relative density [105].

Li et al. [29] investigated the impact of ZrC and TiC on the consolidation of TiC-ZrC composites via SPS at a sintering temperature of 1500–2200 °C. The effects of compositions and sintering temperature on the densification of the composites were observed as well. It was reported that the densification increases as the temperature increases to 1800 °C; the relative density ranges between 95.9 and 98.9%. This

30

16

100

98

96

94

92

<u>90</u>

Relative density( RD) / %

(a)



**Relative density** 

Vickers hardness

Fig. 9 SEM images of polished surfaces of TiC ceramics reinforced with nano-sized WC: **a** TiC-1.5wt%WC and **b** TiC-3wt%WC, adapted with permission from [73]



achieved densification was compared at high compositions of 80-90mol%ZrC under similar sintering temperature and elevated temperature of 2100 °C; it was observed that the densification reduces below 90%. The relative densities achieved for TiC-ZrC composites improved significantly in contrast to the undoped ZrC. It was further stated that the increment in sintering temperature influences the enhanced Vickers hardness; this enhancement is synonymous with the densification that was achieved. Although the composites whose composition >80 mol% TiC at 1500–1700 °C were completely consolidated with hardness > 26 GPa, the fracture toughness of the composites ranges from 2.5 to 4.5 MPa m<sup>1/2</sup>. The composites sintered above 1800 °C depicted a slight decrease in Vickers hardness; these were attributed to the grain growth increment at high temperatures. Similar related work by Teber et al. [106] produces an improved fracture toughness (6.8 MPa  $m^{1/2}$ ) and a peak Vickers hardness (28 GPa) for 80TiC-20ZrC composite when it was sintered at 1350-1800 °C. The improvement was a result of the alloying impact of ZrC in TiC [107].

Sribalaji et al. [30] study the influence of WC and carbon nanotubes as reinforcement in TiC ceramic matrix via SPS at 1600 °C under 50 MPa. The undoped TiC depicted a value of 89% but got improved to 94% for sintered TiC-3.5%WC. The precipitation of WC along the grain boundaries of TiC was the reason for the enhancement in the densification when 3.5wt%WC was reinforced in the TiC matrix. Cheng et al. [36] also made a similar observation in the densification of WC with TiC. Furthermore, on the addition of 2wt% of CNT in the binary composites of TiC-3.5wt% WC, densification hugely upgraded to ~ 99%. This improvement was a result of the even distribution of CNTs, which consequently promoted even dissipation of heat in TiC-WC composites and the melting of fractional parts of TiC [108]. The introduction of WC and CNTs was observed to impede grain growth. The reinforcement of WC and CNT all contributed to the improvement of the hardness (as shown in Fig. 10). The undoped TiC has its hardness value equivalent to ~23.67 GPa, but it got improved to ~27.79 GPa and ~33.56 GPa when 3.5wt%WC and 2wt%CNTs were added to it respectively. The enhancement was credited to increased relative density and decreased grain size. The fracture toughness of the composites increased significantly with each reinforcement, with ~36% and ~76.2% improvement respectively compared with the undoped TiC. The restriction of grain growth by the reinforcements and by the bridging of crack which created a toughening mechanism for the composites were all observed to be the contributing factor for the improved fracture toughness. Mukherjee et al noticed the same bridging behavior when similar CNT was reinforced in Hf carbide-based composites [109, 110].

### 2.4 Comparison of the influences of different carbides in terms of properties (microstructure, densification, and mechanical properties) with TiC ceramic matrix composite

The effects of different carbide-based additives have been judiciously studied on the microstructure, densification, and mechanical properties of TiC, and the result (densification and



### Ceramic Materials

Fig. 10 The impacts of WC and CNT on the hardness and elastic modulus of TiC ceramic matrix composite, adapted with permission from [30]

Material composition	Processing condition	Sintered density	Hardness (GPa)	Fracture toughness (MPa $m^{1/2}$ )	Flexural strength (MPa)	References
TiC-(0-50)% SiC	1600–1800°C, 50–75MPa, 5–10min	95.4–97.3	25–32	3.7–5.7		[27]
TiC-	1600°C,	99	5.5	5.76		[21]
20wt.%SiC	70MPa, 12min					
TiC-	1600°C,	~99	5.2			[87]
14.6vol.%SiC	50MPa, 5min					
TiC-(0-30)	1900°C,	99.23-	25.08-		511-644	[91]
vol% SiC <sub>w</sub>	7min, 40 MPa	102.95	29.04			
TiC-(0-10)vol%WC	1450– 1600 °C 50 MPa, 5 min	91–100	22–28.2	5.2-6.3	524–578	[67]
TiC-(0-3)WC	1900 °C 40 MPa, 7 min	99.23–99.85	22–28.6		418-620	[73]
TiC-(10-90)	SPS,	>96				[29]
mol ZrC	2100 °C					
	50 MPa,					
	5 min					
TiC-	1600°C,	99.9	$33.5\pm4.5$	$7.8\pm0.5$		[30]
3.5wt%WC	50MPa					
-2 wt%CNT	5min					

 Table 1
 The influence of various carbides additives in the TiC ceramic matrix using spark plasma sintering for consolidation

mechanical properties) is tabularized in Table 1. More also, the impacts of these additives are graphically shown in Fig. 11. It was observed that WC and CNT served as a good reinforcement to date to provide improvement for TiC ceramic matrix. Hence, good densification and excellent mechanical properties with fine microstructure were achieved.





### **3** Conclusion

Carbide sintering additives have been studied extensively to provide good sinterability, wettability, microstructure, and excellent mechanical properties for TiC ceramic matrix, owing to its ability to eliminate oxygen and also serving as a strong secondary phase. These resulting ceramic matrix composites have had high significance for application in aerospace materials, cutting tools, other high-temperature materials, etc. In ensuring that for a specific purpose one attained the expected mechanical properties of a ceramic material, the amount and type of sintering additives that serve as a reinforcement needs to be taken into cognizance. In respect to this, most researchers have been used to trial by error in determining the amount and sometimes the type of additives needed to be added to a ceramic matrix for the improvement of its resulting composites. This approach occasionally leads to the wastage of materials and time. In circumventing these challenges, a sophisticated approach should be included in research activities in pre-determining the amount and types of sintering additives required to be added to a ceramic-based matrix in achieving the optimum mechanical properties.

More also, the evolvement of sintering reactions has had some contrasting effects on the densification, microstructure, and mechanical properties of TiC CMC. While some (sintering reactions) have had an enhancing capacity, others have produced some depreciating effects. More works are needed before the sintering of the green powder to determine the influence of any possible reactions on the sintered compact. This in consequence will help in having a fundamental knowledge of the effect of the sintering reaction in either to optimize or eliminates it. The optimization and elimination of these sintering reactions can be achieved through the judicious selection of sintering parameters, percentage compositions of each ceramic matrix, sintering additives, and the types of PM used.

Lastly, in the characterization phase of TiC CMC, there are still some voids that are still needed to be filled. Nevertheless, the flexural strength of a few ceramic materials has been determined at elevated temperatures. Hence, the experimentation of the mechanical properties TiC CMC at an elevated temperature is still required at the research stage, with the view to making it applicable at the industrial level.

#### Availability of data and materials Data and materials are available

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**Ethics approval** This review is solely submitted to this journal and has not been published elsewhere. Proper acknowledgement/reference has been accorded to other works.

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