



The Effect of TiC Additive with Al₂O₃-Y₂O₃ on the Microstructure and Mechanical Properties of SiC Matrix Composites

M. Khodaei^{a*}, O. Yaghubizadeh^b, S. A. Safavi^a, N. Ehsani^a, H. R. Baharvandi^a, S. Esmaeeli^b

^a Composite Materials & Technology Center, Malek Ashtar University of Technology, Tehran, Iran

^b Department of Materials Science and Engineering, Faculty of Technology and Engineering, Imam Khomeini International University (IKIU), Qazvin, Iran

^c Department of Ceramic, Shahreza Branch, Islamic Azad University, Shahreza, Iran

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ABSTRACT

In this research, the SiC-matrix composite with different amounts of TiC (0, 2.5, 5, 7.5, and 10 wt%) supplemented with additives including 4.3 wt% Al₂O₃ and 5.7 wt% Y₂O₃ were utilized to initiate the required liquid phase. The sintering process was performed using pressureless sintering at 1900 °C for 1.5 hours under argon atmosphere. The composition and microstructure of the obtained composites were analyzed using X-Ray Diffraction (XRD), Field Emission Scanning Electron Microscope (FESEM), and Energy-Dispersive X-ray Spectroscopy (EDX). The results showed that TiC additives improved the densification of samples and impeded the growth of SiC grains. According to the phase analysis, the SiC was the main phase, while the TiC and YAG were characterized as partial phases. Additionally, due to the reaction of TiC and Al₂O₃, the composition of the liquid phase contained YAG and YAM. Assessments revealed that the microstructure and the final properties of composites were affected by density, produced phases and their distribution in the matrix, and grain size. According to the results, upon increasing the TiC up to 5 wt%, all the measured properties including density, hardness, elastic modulus, and fracture toughness improved and reached 97.40%, 26.73 GPa, 392 GPa, and 5.80 MPa.m^{1/2}, respectively. However, with increasing the additives to more than 5 wt%, these properties deteriorated. Microscopic evaluations revealed that crack deflection and crack bridging mechanisms contributed to the fracture toughness of SiC ceramics.

1. INTRODUCTION

Due to high thermal conductivity, low thermal expansion coefficient, and stability of mechanical strength up to 1400-1500 °C, silicon carbide (SiC) can readily tolerate thermal cycles at elevated temperatures [1-7]. The SiC is resistant to thermal shocks [8-11]. In addition, using SiC ceramics is 10 to 50% more energy-efficient than metallic superalloys [11]. Silicon carbide is a hard material (17-25 GPa) with Young's modulus of 400-450 GPa [8,11]. The chemical reactivity of SiC is low at room temperature, which is regarded as one of its inherent chemical properties [8].

Given the mentioned characteristics, SiC ceramics are utilized in industrial heat exchangers, steam and gas turbines, glass industry, metallurgical industries, ceramic

industry, nuclear and thermal power stations, and aeronautical constructions [12-14].

Despite these desirable characteristics, there are also other undesirable ones including low fracture toughness which not only limit the use of SiC in the industry but also encourage scientists to solve this weakness [8,11]. Some researchers argue that reinforcing the structure of SiC ceramics is the best solution to this flaw [15,16].

SiC-matrix composites are manufactured in different ways [8]. Among them, pressureless sintering is the most significant one since it provides the possibility of producing large pieces or complicated shapes [11,13].

In order to reach over 95% of the theoretical density, the methods of solid-state and liquid-state pressureless sintering were employed [17-20]. Liquid-state sintering yields superior mechanical properties to those produced

* Corresponding Author Email: mahdi.khodaei01@gmail.com (M. Khodaei)

by solid-state sintering for the SiC, increasing the employment of liquid-state sintering [11, 20].

The presence of the liquid phase produces improved homogeneity and mechanical properties, especially fracture toughness [20-23]. Liquid-phase sintering depends on the composition of base materials and liquid-phase additives [8,11]. In this respect, the most common additives are Al₂O₃-Y₂O₃ [12,19], Al₂O₃-Yb₂O₃ [10], Al₂O₃-La₂O₃ [24], Al₂O₃-Dy₂O₃ [10], and (SiC-AYC) Al₂O₃-Y₂O₃-CaO [11]. Among these additives, Al₂O₃ and Y₂O₃ have been studied more than others because they can react with SiO₂ which exists on the surface of SiC powders, thereby enhancing sinterability [20,26]. Moreover, it has been proved that adding oxides, carbides, nitrides, and borides such as TiO₂, B₄C, TiB₂, and ZrB₂ as the secondary phase or reinforcement improves the fracture toughness of SiC ceramics [20].

The related pieces of research have shown that employing TiC particles increases the sinterability of SiC ceramics [8]. Such particles impede the growth of matrix grains and improve the mechanical properties [27]. Further, the existence of TiC particles on the path of crack growth increases fracture energy [8,20] and generates subgrains and microcracks; in addition, the mismatch between the thermal expansions of TiC and SiC plays a significant role in improving the properties [8].

The present study investigates the effect of different amounts of TiC (0, 2.5, 5, 7.5, and 10 wt%) on the properties and microstructure of the pressureless sintered SiC-TiC composite.

2. EXPERIMENTAL PROCEDURES

2.1. Raw materials and Methods

In this study, α -SiC made by Alfa Aesar, with the mean grain size of 0.3 microns, was utilized as the main powder. In addition, 10 wt% α -Al₂O₃ and Y₂O₃, made by Alfa Aesar, with the particle size less than 1 micron and a ratio of 4.3:5.7 were utilized to produce the liquid phase. TiC powder, with the particle size less than 1 micron was produced by Merck Co., and employed as reinforcement.

Compositions analogous to those shown in Table 1 were prepared to assess the impact of TiC additive on the properties of samples. The weight ratio of Y₂O₃ to Al₂O₃ in these compositions was determined to be 57:43 based on the lowest eutectic temperature.

The powders were mixed in a planetary mill in a tungsten carbide chamber and an ethanol setting, whose rotational speed was set to 180 rpm for 3 hours. Then, the obtained slurry was dried at 100 °C for 24 hours. Next, the samples underwent uniaxial pressing (90 MPa) and turned into cylindrical specimens. Finally, the samples

were sintered at 1900 °C under argon atmosphere for 1.5 hours.

The heating rate was 2 °C/min. After sintering, samples were kept at environment temperature inside the furnace.

According to the assessments, 1900 °C was the best temperature for sintering SiC-Al₂O₃-Y₂O₃ system [8, 11, 20, 25].

TABLE 1. Compositions of each sample

Raw materials	wt%				
	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
SiC	90	87.5	85	82.5	80
TiC	0	2.5	5	7.5	10
Al ₂ O ₃	4.3	4.3	4.3	4.3	4.3
Y ₂ O ₃	5.7	5.7	5.7	5.7	5.7

2.2. Measurement of properties

The density and porosity of the sintered samples were measured using the Sartorius LA 230S digital scale (with the precision of 0.0001 g), according to C373–88 standard, through Archimedes' method. Equation (1) was used to calculate the theoretical density:

$$\rho_{com} = v_a \rho_a + v_b \rho_b + v_c \rho_c + \dots \quad (1)$$

where v and ρ are the volume fraction and density of a given phase, respectively. Crystalline phases were characterized by X-ray diffraction. Micro-Hardness was measured by Vickers method according to ASTM C1327. The hardness was also calculated by exerting a force of 5 N in an MXT-al microhardness tester, as shown in Equation (2):

$$H = (1.854)P/d^2 \quad (2)$$

where P and d represent the applied force and indentation diameter, respectively.

The indentation fracture toughness of samples was measured based on the ASTM C1421-10 standard. In this method, ESE WEY microhardness tester, DVRB–TV model, was utilized to exert a force of 300 N on the surface of samples so as to develop cracks on their surface. Then, the length of the produced surface cracks was measured, and the toughness of samples was determined using Equation (3):

$$K_{IC} = 0.0752 P/C^{3/2} \quad (3)$$

where P and C represent the applied force and length of the produced surface crack, respectively. In order to study the microstructure, samples were etched by Murakami's reagent using a chemical method. Microstructural studies of the polished and etched surfaces of the sintered samples were undertaken by applying a voltage of 15-20 kV using a scanning electron microscope called Tescan model.

3. RESULTS AND DISCUSSION

3.1. Phase analysis of samples

In Fig. 1, the X-ray diffraction pattern of the samples containing 0, 5, and 10 wt% TiC additives is depicted. In the sample devoid of TiC additives, there can be observed SiC as the dominant phase as well as a negligible amount of the $Y_3Al_5O_{12}$ phase known as the Alumina–Yttria–Garnet (YAG) that exists as the grain-boundary phase. However, in the samples containing TiC additives, other than the SiC phase SiC (JCPDS card 01-073-1663), which is the dominant phase, YAG (JCPDS card 01-088-2048), YAM ($Y_4Al_2O_9$) (JCPDS card 00-014-0475) phases, and TiC (JCPDS card 065-7994) phases are obtained.

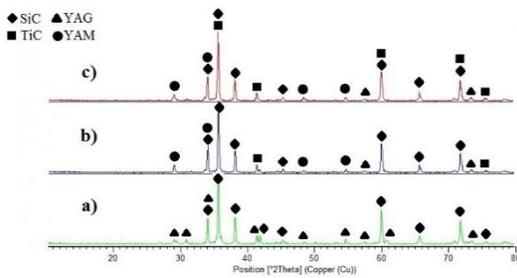
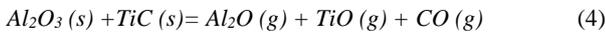


Figure 1. XRD analysis of the sample containing a) 0, b) 5, and c) 10 wt% TiC

According to Fig. 1 (b and c), with an increase in the amount of TiC phase, the intensity and number of TiC peaks increase as well. Additionally, with a rise in the amount of TiC phase, the intensity of YAG peaks decreases and YAM phase reaches the peaks in the XRD of the sintered samples. The probable reaction between the TiC and Al_2O_3 phases (reaction 4), together with the withdrawal of the Al_2O_3 from the composition of the liquid phase, is one of the reasons why the YAM phase is formed [27].



According to Reaction (4), the withdrawal of Al_2O_3 from the composition of the liquid phase and the stoichiometric changes in the YAG phase based on the Al_2O_3 – Y_2O_3 phase diagram (Fig 2.) form the YAM phase. On the one hand, the formation of this phase may be due to the rapid cooling of the samples at room temperature after sintering and decomposing the YAG phase [8,25]. On the other hand, it is possible that with the withdrawal of Al_2O_3 , some excess Y_2O_3 remain in the samples. This Y_2O_3 can react with the SiO_2 existing on the surface of SiC particles, forming Y_2SiO_5 [28]. However, given the XRD patterns of the sintered samples, no Y_2SiO_5 was found.

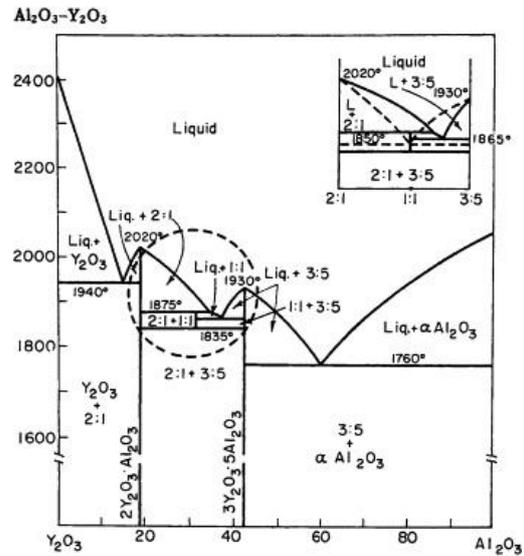


Figure 2. Phase diagram of Al_2O_3 - Y_2O_3 [25]

3.2. Microstructure

In Fig. 3, the line scan analysis of the sample containing 7.5 wt% TiC is depicted. The white-colored regions in grain boundaries contain Al, Y, and O, which somehow confirms the presence of the YAG in the grain boundary of samples. Moreover, given the presence of Ti and C elements in this region, X-Ray diffraction patterns prove the existence of the TiC phase.

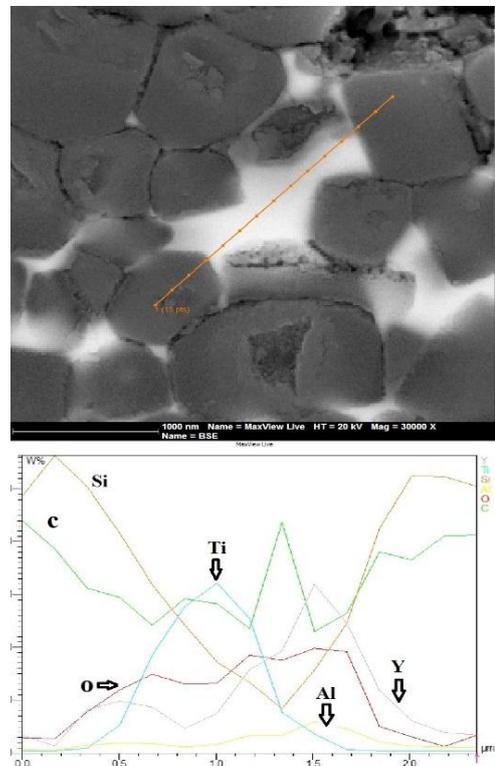


Figure 3. Line scan analysis of sample containing 7.5 wt% TiC

Map analysis of the composition containing 5 wt% TiC additives is depicted in Fig. 4. Clearly, secondary phase particles are homogeneously distributed everywhere.

The microstructures of the samples containing 0, 5, and 10 wt% TiC are represented in Fig. 5. Based on the microstructures of the samples devoid of TiC additives shown in Fig. 5 (a), most of SiC grains are relatively equiaxed and only a negligible amount of elongation in SiC grains is observed.

As observed in Fig. 5 (b-c), in the sample with 5 wt% TiC, the amount of porosity was lower than that in the sample with 10 wt% TiC. Therefore, the relative density of the former is higher than that of the latter. Additionally, according to the images of both samples and presented data in Table 2, it can be concluded that adding up to 5 wt% TiC impedes the excessive growth of SiC grains and refines the microstructure. Nonetheless, Adding up to 10 wt% TiC results in grain growth and based on the obtained results, it reduces the density as well as attenuates mechanical properties.

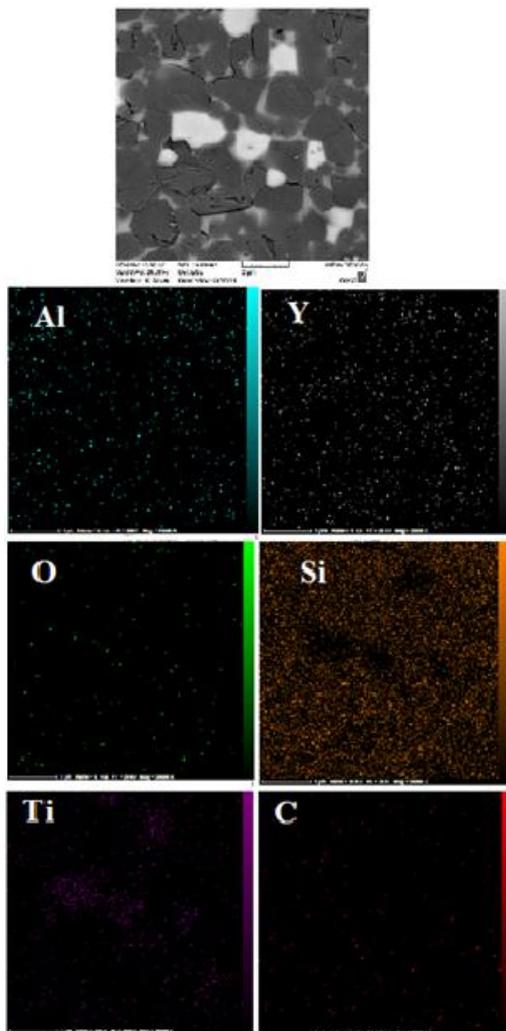


Figure 4. Map analysis of sample containing 5 wt% TiC

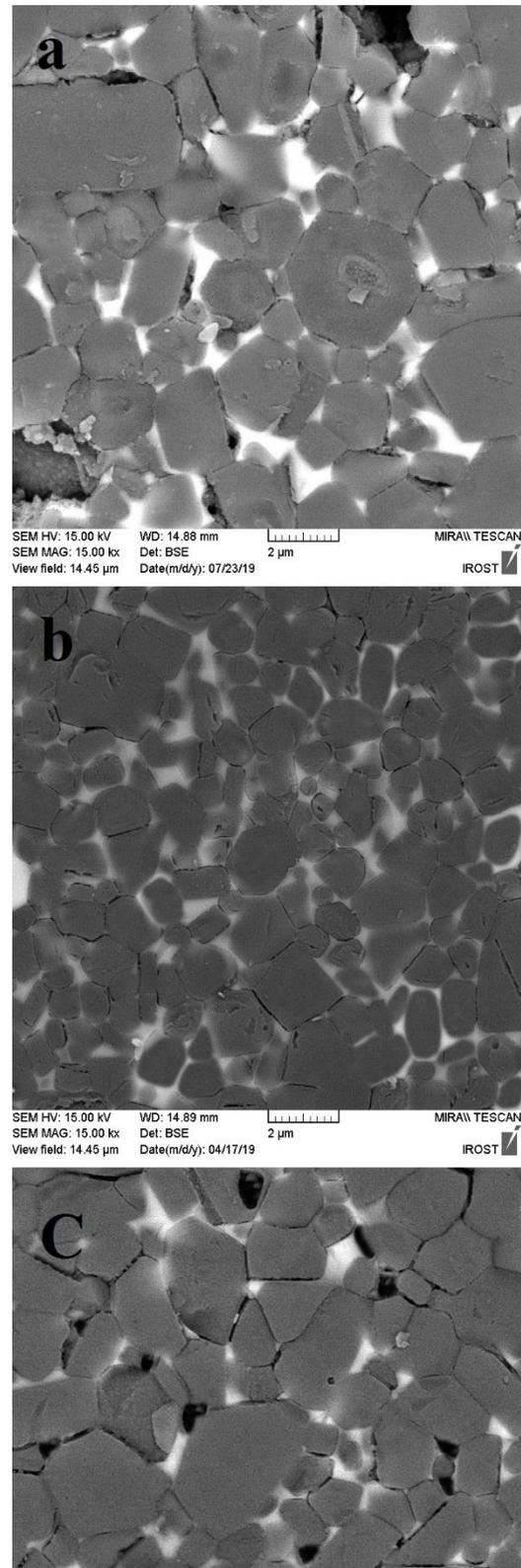


Figure 5. Microstructure images of samples containing a) 0, b) 5, and c) 10 wt% TiC

TABLE 2. The effects of different TiC additives on the grain size of composites

TiC (wt. %)	0	2.5	5	7.5	10
Grain size (μm)	2 ± 0.04	1.5 ± 0.3	1.36 ± 0.07	1.5 ± 0.6	1.8 ± 0.13

Given the presence of TiC particles on the boundaries of SiC grains and their homogeneous distribution in the SiC matrix, a microstructure with fine equiaxed grains is attained in the samples containing 5 wt% and 10 wt% TiC. This suggests that adding TiC impedes the excessive growth of SiC grains and refines the microstructure. Researchers have found that according to Zener's model, the secondary phase impedes the growth of matrix grains [29]. The grain size of the matrix can be calculated using Equation (5).

$$G = 4r/3f \quad (5)$$

where r and f present the radius and volume fraction of the secondary phase, respectively. Equation (5) shows that with an increase in the fraction of the secondary phase, the grain size of the matrix decreases [8,29].

In addition, in compositions with up to 5 wt% TiC additives, the presence of TiC particles on the grain boundaries of the SiC increases the aspect ratio. These obtained results are significant with respect to toughening since any increase in aspect ratio enhances the fracture toughness of the composite [30].

Therefore, it can be asserted that the presence of TiC particles in the grain boundaries of SiC not only refines the matrix but also makes SiC grains elongated. This may be due to the fact that TiC particles precipitate in the grain boundaries of the SiC, limiting the preferred sites for the growth of equiaxed grains [27].

3.3. Evaluation of density, weight loss, and linear shrinkage

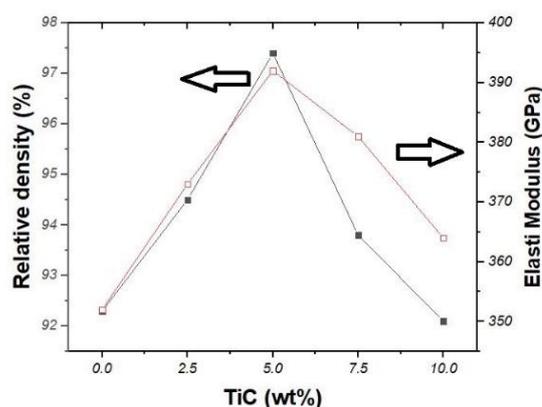
In Fig. 6, the effect of different amounts of TiC additives on densification of the liquid-phased sintered SiC ceramics is illustrated. As observed, by increasing the amount of TiC up to 5 wt%, the density increases, while by adding up to 10 wt% TiC, the density of the composite diminishes. The density of the samples increases due to several reasons: first, the presence of TiC particles on the grain boundaries of SiC reduces the rate of boundary migration, thus minimizing the excessive grain

growth. Second, an increase in surface diffusion and movement of porosities diminish cavities in samples, which increases density during the sintering process [8]. The reason why the density of samples decreases is because there is a reaction between TiC and Al_2O_3 , which is in agreement with the results of XRD patterns. However, if there is not sufficient liquid phase, it cannot wet SiC particles completely, thus creating a great deal

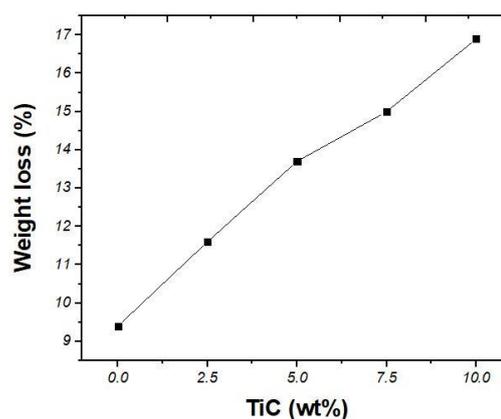
of porosity on the grain boundary phase. This lowers the density and mechanical properties of the sintered samples.

The reactivity between SiC and sintering additives during the sintering process is analyzed by measuring weight loss. Thus, in liquid-phased sintering, the reaction between SiC and additives, especially oxide additives, is almost inevitable, and some weight loss is always observed.

In Fig. 7, the weight loss of the sintered samples is shown according to different amounts of TiC. According to Fig. 7, with an increase in the amount of TiC, the amount of weight loss in samples increases, too.

**Figure 6.** The effect of different amounts of TiC on the relative density and elastic modulus

Given that oxide additives, especially Al_2O_3 , are the main causes of the reactions resulting in the weight loss of samples being sintered, by adding TiC, the constant increase in the weight loss of the samples is predictable.

**Figure 7.** Weight loss variations versus TiC content

3.4. Hardness

In Fig. 8, the hardness variations are shown as a function of the amount of TiC additive. With any

increase in the amount of TiC particles up to 5 wt%, the hardness of the samples increases. However, with the further addition of TiC particles up to 10 wt%, the hardness is diminished. This behavior of the samples was completely congruent with the variations in their density and the highest hardness quantity was achieved in samples with the highest density. Therefore, other than the effect of density on the hardness of samples, grain size reduction can also improve the sample hardness [31-34]. According to hardness results of the samples, adding up to 5 wt% TiC enhances the samples' hardness. It is acknowledged that the presence of TiC particles on the grain boundaries of the SiC lessens the rate of grain-boundary migration and minimizes the excessive grain growth, which increases the samples' density and creates a fine-grained SiC composite [25,29]. Based on Hall-Petch relation [29], it can be asserted that with a reduction in the grain size, the hardness of samples increases.

One of the reasons explaining why an increase in TiC additives reduces the hardness is the formation of microcracks on the interface of SiC and TiC grains, which occurs during the cooling process at the sintering temperature as a result of the mismatch between the thermal expansion coefficient of SiC and that of TiC particles [8]. Fig. 9 presents the produced microcracks in the composition of the composites with 10 wt% TiC.

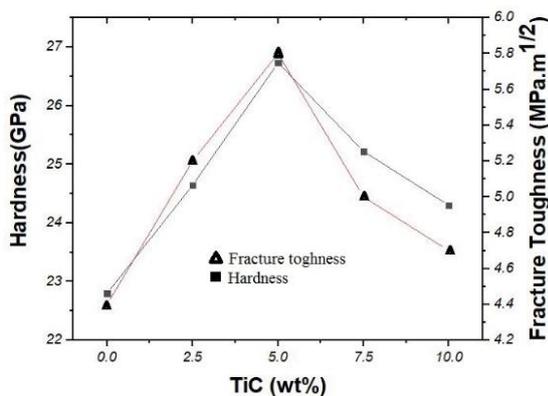


Figure 8. The effect of different amounts of TiC on the Hardness and Fracture Toughness

3.5. Elastic Modulus

Similar to the case of hardness, the elastic modulus is heavily dependent on the porosity and density of samples; therefore, elastic modulus decreases with an increase in porosity or a reduction in the relative density of samples. In Fig. 6, the elastic modulus of the sintered samples with different amounts of TiC additive is represented.

Based on the obtained results, with the addition of TiC, the elastic modulus of the composite went up. However, upon adding more than 5 wt% TiC, the density was diminished and the formation of porosities reduced the sound speed inside the samples. Given that sound waves

are mechanical waves, they need a medium through which they can move. Thus, the lower the amount of matter (less density/more porosity), the slower the speed of sound. Considering the relation between sound speed and Young's modulus, a decline in Young's modulus of samples is expected.

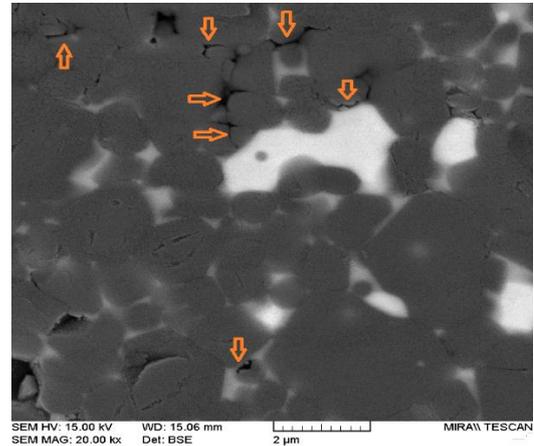


Figure 9. SEM image of the propagation of micro crack in the sample containing 10 wt% TiC

3.6. Fracture Toughness

The variations in fracture toughness are depicted as a function of the amount of TiC additive in Fig. 8. The amount of porosity formed during the sintering process is one of the factors that can affect fracture toughness.

According to the results of the fracture toughness tests such as those of the hardness test, for the samples containing up to 5 wt% TiC, a significant improvement in fracture toughness was achieved. Nevertheless, by adding up to 10 wt% TiC additive to the sintered samples, their fracture toughness steadily decreases. However, the presence of 10 wt% TiC particles in the SiC matrix is beneficial, and the quantities of their fracture toughness are higher than those of the ceramics devoid of TiC additive.

This increase in fracture toughness occurs despite the reduction in the relative density of the samples containing 10 wt% TiC, indicating that the relationship between density and porosity must not be regarded as the only determining factor in the fracture toughness of the samples. Accordingly, it can be asserted that the following elements are influential in the fracture toughness of the SiC-TiC composites: density, reinforcement and its amount, grain morphology, and microstructure [35-37].

The presence of TiC particles on the grain boundaries of SiC enhances the fracture toughness of the sintered bodies due to two reasons: first, the presence of the TiC on the grain boundaries of the SiC refines the matrix grains. The reason why grain refining increases the

fracture toughness can be attributed to the influence of grain boundaries as an obstacle to crack growth [8].

Grain boundaries hinder the movement of cracks. Therefore, a great deal of energy is required for crack nucleation and propagation because it must change its path in order to keep moving on a desirable plane in an adjacent grain.

Another reason for an increase in fracture toughness owing to grain refinement may be related to a reduction in crack length due to the smaller grain size. In such a condition, the crack requires a higher stress for growth and composite failure [8,29].

In addition, using TiC hinders the grain growth and increases the elongation of some SiC grains (Fig. 10), thus improving the fracture toughness of liquid-phase sintered SiC-TiC composites. The elongated SiC grains in the microstructure function as reinforcement and trigger toughening mechanisms such as crack deflection and crack bridging, leading to an improvement in the fracture toughness of SiC bodies. In the following, the toughening mechanisms of this composition will be addressed.

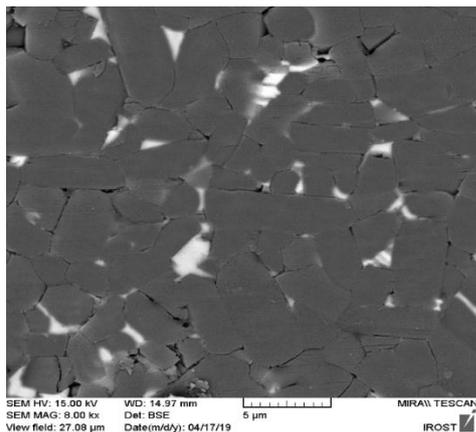


Figure 10. Formation of SiC elongated grains in composite containing 5wt% TiC

3.7. Toughening Mechanisms

The present research utilized Vickers indenter to assess toughening mechanisms and cracks on the polished surface of the samples using a diamond indenter under a 30 kg load. The crack propagation and origin of toughening mechanisms in the SiC ceramics devoid of TiC additive and SiC-TiC composites are presented using an SEM. In Fig. 11 (a), images of crack propagation path in the sample with no TiC additive are shown.

As shown in Fig. 11 (a), the secondary phase is mainly present in the form of a continuous film on the grain boundaries of the SiC. Given that a large proportion of the intergranular phase is comprised of the YAG phase, which is a weak oxide phase, cracks propagate in the form of intergranular cracks. Crack deflection

mechanisms are also observed to a limited extent. In Fig. 11 (b and c), the SEM images of the crack growth path of the ceramics containing 5 wt% and 10 wt% TiC are presented.

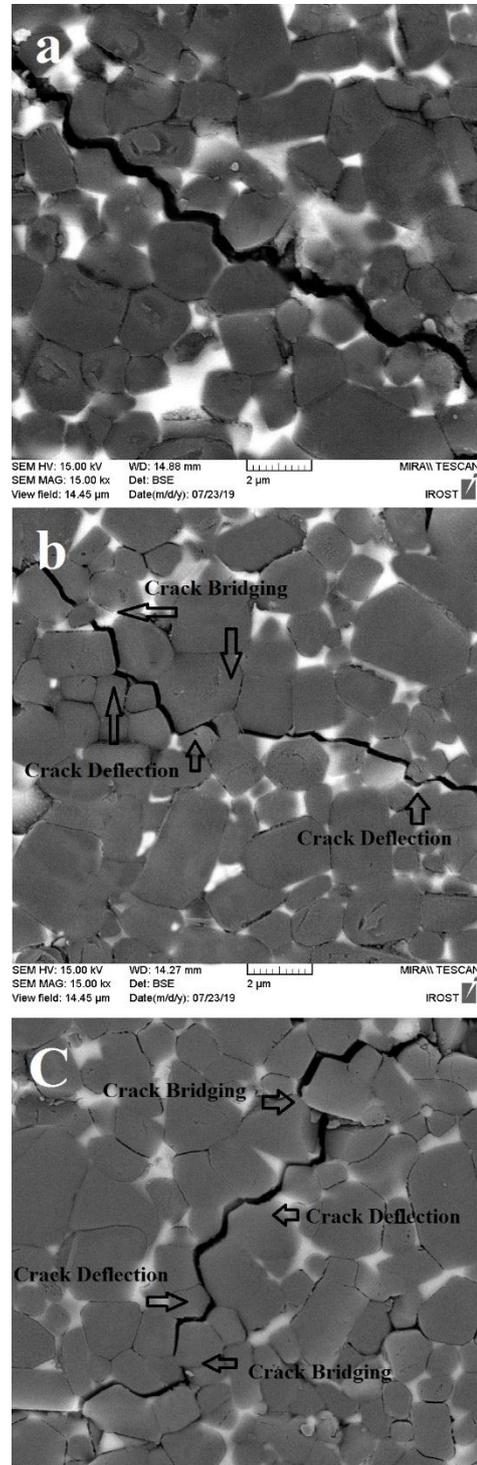


Figure 11. SEM images of the crack path in samples containing: a) 0, b) 5, and c) 10 wt% TiC

According to the evaluations, any enhancement in the fracture toughness of the sintered bodies depends on the grain morphology and formed microstructure [38]. It is probable that in this composition, crack deflection and crack bridging improve fracture toughness. In this regard, the crack deflection mechanism is considered the most significant mechanism in improving the liquid-phase sintered SiC ceramics. The crack deflection mechanism is triggered due to the weak interface between the SiC and the grain boundary phase [8,11].

Another possible mechanism affecting the enhancement of the fracture toughness of these ceramics is the pullout of SiC grains, leading to crack surface bridging.

This mechanism plays a crucial role in the compositions that have elongated grains whose length-to-diameter ratio is high. In the structures containing homogenous (equiaxed) grains, the contribution of this mechanism to the improvement of fracture toughness is not substantial [20,29,39].

A comparison between the results of this study and those of other researchers is presented in Table 3.

TABLE 3. A comparison between the results of this study and those of other researchers

Additive Composition	Relative Density (%)	Hardness (GPa)	Fracture Toughness (MPa.m ^{1/2})	Strength (MPa)	Ref.
Al ₂ O ₃ -Y ₂ O ₃ -TiC	97.40	26.73	5.80	-	This paper
Al ₂ O ₃ -Y ₂ O ₃ -MgO	96.05	26	5.24	440	[32]
Al ₂ O ₃ -Y ₂ O ₃ -TiC	98.1	26.13	4.07	432	[27]
Al ₂ O ₃ -Y ₂ O ₃ -ZrB ₂	98	19.94	6.68	565.70	[39]
Al ₂ O ₃ -Y ₂ O ₃ -TiC	97	-	7.8	-	[40]
Al ₂ O ₃ -Y ₂ O ₃ -TiO ₂	99.2	27.4	4.6	516	[30]
Al ₂ O ₃ -Y ₂ O ₃ -AlN	97	25.7	3.1	582	[41]

4. CONCLUSIONS

1. Based on the results of the present study, the liquid Al₂O₃-Y₂O₃ is a useful additive for the liquid phase sintering of SiC-TiC composites.
2. The sample containing 5 wt% TiC additives exhibits the highest density, Young's modulus, hardness, and fracture toughness. However, with further increasing the amount of TiC additive to 10 wt%, these properties would deteriorate.

3. Introduction of a negligible amount of TiC additive (5 wt%) is sufficient enough to impede the growth of SiC grains and improve the mechanical properties of SiC ceramics.
4. The samples with the highest density and the finest microstructure exhibit higher hardness. The formation of microcracks with an increase in the amount of TiC is one of the reasons why hardness is reduced.
5. Among the parameters that affect the fracture toughness of SiC-TiC composites are density, amount and type of reinforcement, grain morphology, and microstructure.
6. Active mechanisms that improve the fracture toughness of SiC-TiC composite are crack deflection and crack bridging. As a result, crack deflection is the dominant mechanism in the fracture toughness of SiC-TiC composites.

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