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Research Article

Microstructural Evolution and Mechanical Properties Enhancement of Ti/SiC Metal Matrix Composites

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Abstract

Titanium's exceptional strength, low density, and outstanding corrosion resistance make it an ideal material for critical applications in power generation, the gas industry, sports equipment, and various industrial sectors. Under high-temperature conditions, titanium alloys must exhibit superior heat resistance and corrosion durability. However, enhancing titanium alloys with silicon carbide (SiC) through conventional powder metallurgy often results in issues such as porosity and the formation of silicides. To mitigate silicide formation, the hot-pressing technique has demonstrated excellent outcomes, achieving near-theoretical density without reaction zones. Nevertheless, increased sintering temperatures typically lead to a reduction in hardness. The highest hardness recorded was 92 HRB for a composite consisting of 70% titanium and 30% SiC at a sintering temperature of 900°C. By optimizing the sintering time, temperature, and applied pressure, denser Ti/SiC composites were produced.

Keywords: Metal Matrix Composite, titanium, silicon carbide, compaction, sintering, powder metallurgy.

INTRODUCTION

Titanium alloys are well-known for their excellent strength-to-weight ratio, outstanding corrosion resistance, and superior mechanical properties, making them indispensable across various industries, including power generation, gas, sports equipment, and aerospace applications. In power generation, titanium must exhibit exceptional heat and corrosion resistance to perform reliably in high-temperature and chemically aggressive environments [1-3].

Enhancing titanium alloys with ceramic materials, such as silicon carbide (SiC), through metal matrix composites (MMC), can significantly improve their mechanical performance. SiC reinforcement enhances the strength, hardness, and wear resistance of titanium, making it ideal for demanding applications [4]. However, powder metallurgy (P/M), a common fabrication method for Ti/SiC composites, presents challenges, particularly the formation of porosity, which can degrade mechanical properties [5].

Optimizing the P/M process to minimize porosity is critical to producing composites with improved mechanical performance [6, 7]. The hot press technique in P/M has shown promising results, yielding Ti/SiC composites with uniform density and enhanced mechanical properties [8]. This research focuses on determining the optimal volume fractions such as 70:30% and 60:40% Ti matrix to SiC reinforcement—under sintering temperatures of 900°C, 1000°C, and 1100°C, and their effects on the mechanical characteristics and microstructure of Ti/SiC composites.

Mechanical testing, including hardness testing and Scanning Electron Microscope (SEM) analysis, was performed to evaluate the mechanical properties and investigate the influence of sintering temperatures on the resulting microstructure [9-11].

MATERIALS AND EXPERIEMNTAL METHODS

Experiments were conducted using titanium (Ti) powder as the matrix and silicon carbide (SiC) powder as the reinforcement. Figure 1 presents a schematic illustration of the fabrication process for the Ti/SiC composite using powder metallurgy, which includes powder preparation, mixing, pressing, and sintering at varying temperatures. The Ti and SiC powders were reduced to a particle size of #320 mesh. Composite volume fractions were set at 70:30 and 60:40 to achieve ideal values. For sample polishing, Al_2O_3 (alumina) was employed alongside abrasive paper ranging from #100 to #1500 grit, with Bakelite used as a mounting material and ethanol as a cleaning agent.

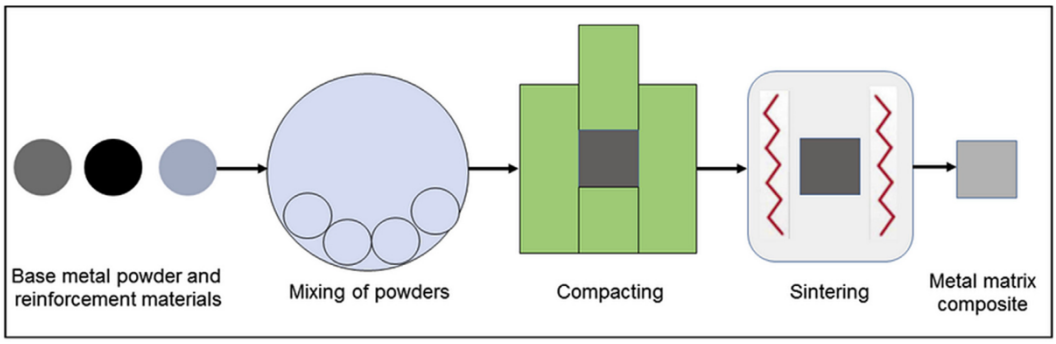


Figure 1. Schematic illustration for the fabrication of Ti/SiC composite by powder metallurgy, encompassing powder preparation, mixing, pressing, and sintering at different temperatures [12].

Furthermore, Figure 2 illustrates the grain refinement process, emphasizing the critical steps in reducing the size of Ti/SiC powder for composite material preparation. According to [13], achieving a homogeneous particle size is crucial for the composite's performance. The process involves carefully balancing the matrix material powder (Ti) and the reinforcing powder (SiC) to ensure uniform particle sizes. This consistency facilitates a uniform microstructural bond during compaction and sintering, resulting in an enhanced bonding strength between the titanium matrix and the silicon carbide reinforcement, thereby improving the mechanical properties of the final composite material.

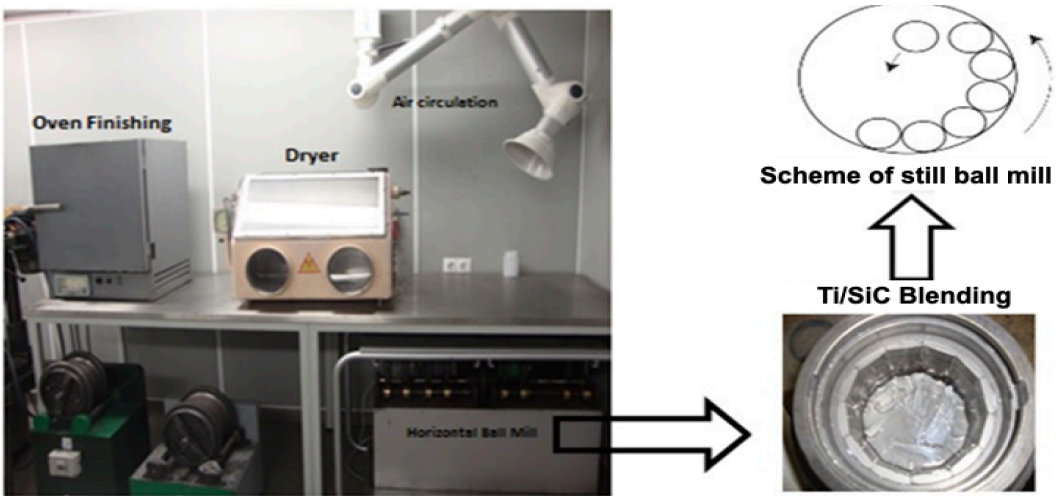


Figure 2. Grain refinement through the use of horizontal ball milling process.

Experimental setup

The experiment involved compacting a Ti/SiC composite powder mixture using a 20-ton press machine. The sintering process was conducted with a muffle furnace, utilizing temperature variations of 900°C, 1000°C, and 1100°C. Hardness measurements were obtained through a Rockwell Hardness Tester (HRB). Microstructural morphology changes between the Ti matrix and SiC reinforcement were analyzed with a Scanning Electron Microscope (SEM), while Energy Dispersive Spectroscopy (EDS) was employed to identify the elemental distribution within the composite. X-Ray Diffraction (XRD) analysis was also performed to characterize compounds formed between the Ti matrix and SiC reinforcement. The muffle furnace, featuring a front-opening door and precise temperature control, ensured uniform heating and supported the controlled solidification of the compacted Ti/SiC composites, achieving the desired material characteristics. Figure 3 depict powder refining process for achieving the particle size in Mesh Ranges that varied from 80 to 360 mesh.

Figure 3. Powder refining machine used to achieve particle sizes in Mesh Ranges (80-360 mesh), KRISBOW 20-Ton powder metallurgy press machine with red frame, and lab-based muffle furnace for sintering Ti/SiC composites

Preparation of Samples

The titanium powder was processed to achieve finer particle sizes and subsequently sieved through #100 mesh sieves, while the SiC powder was sieved to #320 mesh. The powders were then combined in specified ratios (70:30 and 60:40) and precisely measured to the required quantities. The composite powders were compacted using a press machine under consistent force, with stearic acid applied to the mold as a release agent to prevent adhesion during compaction.

Sintering Procedure

Table 1 shows the compacted samples subjected to sintering in a muffle furnace at three distinct temperatures: 900°C, 1000°C, and 1100°C. Each sample was held at the designated sintering temperature for one hour. Following sintering, the samples were allowed to cool to ambient temperature before further testing.

Table 1. Comparison of mechanical properties of Ti/SiC composites at different volume fractions (Vf)

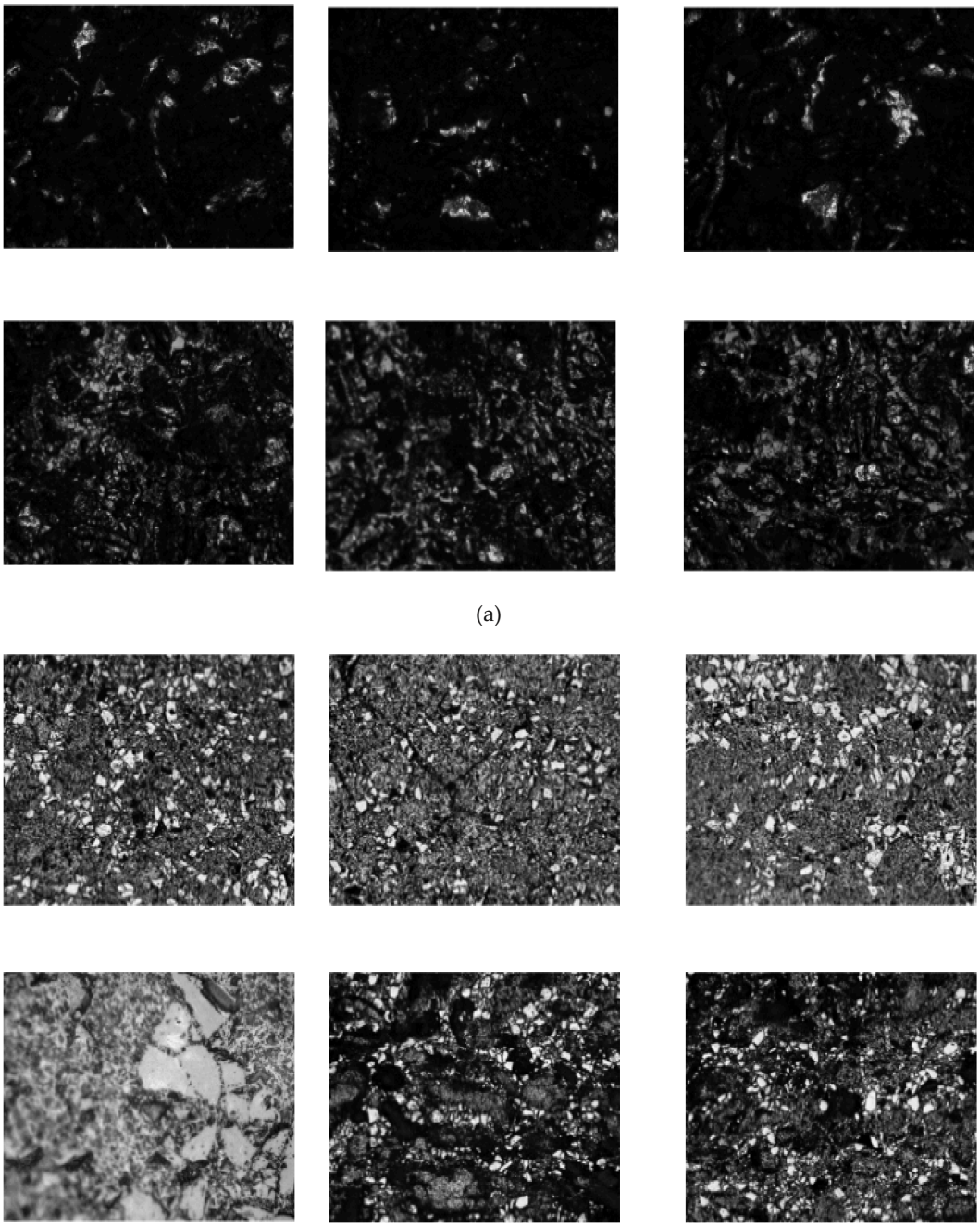
Characterization	**Volume Fraction 70:30 %**			**Volume Fraction 60:40 %**		
	900°C	**1000°C**	**1100°C**	**900°C**	**1000°C**	**1100°C**
Hardness (BHN)	92	-	18	54	50	61
Porosity (%)	2.3	3.3	4.1	4.1	5.9	4.3
Density (gr/cm^3)	4.3	4.5	4.3	4.3	4.2	4.4

EXPERIMENTAL RESULTS

Density and Porosity Analysis

The density of the Ti/SiC composites, as seen in Table 1, was affected by the sintering temperature and volume fraction. At elevated sintering temperatures (1100°C), the density markedly increased, especially in the 60% Ti and 40% SiC volume fraction, where the maximum density observed was 446 gr/cm^3. The rise in density is due to enhanced compaction of titanium particles and the capacity of SiC reinforcement to occupy the interstitial voids within the matrix. The sintering process facilitates diffusion and densification, hence decreasing porosity and enhancing total density [14, 15].

Figure 4 illustrates the impact of sintering temperature on the density and porosity of Ti/SiC composites. The photos depict varying volume fractions (60% Ti: 40% SiC at 1100°C and 70% Ti: 30% SiC at 900°C), emphasizing the distinctions in density, porosity, and the arrangement of SiC particles. The visual comparison of ideal density and diminished porosity at lower sintering temperatures highlights the correlation between compaction and void development at elevated temperatures [16].



(a)

(b)

Figure 4. Impact of sintering temperature on the density and porosity of Ti/SiC composites. A comparison of (a) 60% Ti: 40% SiC at 1100°C and (b) 70% Ti: 30% SiC at 900°C.

Porosity increases at high sintering temperatures. at 1000°C sintering, the volume fraction of 60%: 40% shows increased densification at high temperatures, based on experiments conducted by Pramono, et al [17] continuous heat reaching critical

temperatures in titanium, can result in gas trapping which results in localized microstructural changes, thus creating gaps in the matrix (voids).

To achieve an ideal balance between temperature and volume fraction, the ideal volume fraction composition follows the temperature uniformly and evenly, as has been done in [18, 19]. Based on ideal experiments, 900°C sintering is optimal for a composition of 70:30, %, because the use of *V*f and sintering is able to reduce the porosity value and increase the density.

Microstructural Evolution and Oxide Formation in Ti/SiC Composites at 1000°C: XRD, EDS, SEM of Optical Analysis

SEM, XRD, and EDS analyses were specifically performed on samples that failed, namely those sintered at 1000°C with a volume fraction of 70:30%. Under these conditions, significant microstructural evolution was observed, resulting in the oxidation of all elements in both the matrix and reinforcement, forming TiO_2 and SiO_2 oxides, as evidenced by XRD and EDS results. XRD analysis showed a dominant percentage of TiO_2 with a tetragonal structure, attributed to oxidation from ambient oxygen exposure in the furnace, as illustrated in Figures 5 until 7. Oxygen was rapidly absorbed, dissociating into ions that reacted with titanium in hot air to form oxides. In contrast, at sintering temperatures of 900°C and 1100°C, intermetallic phases such as TiC and Ti_5Si_4 were observed, alongside stable hexagonal Ti and SiC, indicating these phases were not entirely degraded by oxidation. Elevated temperatures also facilitated crystal formation, suggesting that 1000°C may represent a critical threshold temperature for titanium in Ti/SiC-based composites, where oxide formation substantially compromises structural integrity.

After experiencing an increase in sintering temperature reaching 1100°C, the TiO_2 phase decomposes back into titanium (Ti) and other compounds, as indicated by the XRD peak. EDS analysis describes the composition of the main elements formed are: iron, with nickel, chromium, aluminium, titanium, and sulphur, indicating the possibility of an iron-based alloy suitable for high temperature and corrosive environments. Sulphur can raise concerns about sulphide inclusions [20], which can reduce toughness [21]. SEM analysis at a temperature of 900°C shows optimal mechanical properties, with well-dispersed SiC particles and strong adhesion in the titanium matrix. It can be used as a reference that the ideal temperature for the formation of Ti/SiC-based composites is 900°C. At this temperature, the bond between the matrix and the reinforcement has optimal adhesion properties between the two phases.

The results of the EDS spectrum are the elements formed in the Ti/SiC composite, where the presence of several key elements are as follows: Titanium (Ti) and Silicon (Si) WHICH are the main constituents, confirming the Ti/SiC composition. The peak for Oxygen (O) indicates oxidation on the surface, while the smaller peaks are: Iron (Fe), Nickel (Ni), Chromium (Cr), and Aluminium (Al) are alloying elements or contamination in the process.

SEM investigations revealed substantial microstructural changes in the Ti/SiC composite sintered at 1000°C, with a volume fraction of 70% Ti and 30% SiC. These changes

can be attributed to the transformation of the composite material, namely the oxidation reaction that occurs at high temperatures. At this temperature, the interaction between titanium (Ti) and silicon carbide (SiC) with oxygen (O_2) results in the formation of titanium dioxide (TiO_2) and possibly silicon dioxide (SiO_2). The SEM image clearly shows the modified surface morphology and the presence of oxide-rich regions. The formerly distinct borders between the titanium matrix and SiC reinforcement are now indistinct, signifying the passage of oxygen into the matrix, which facilitates the synthesis of these oxides. The reaction at this temperature can be summarized as follows:

$$Ti+SiC+O_2 \rightarrow TiO_2+SiO_2 \text{Ti+SiC+O}_2 \rightarrow \text{TiO}_2+\text{SiO}_2$$

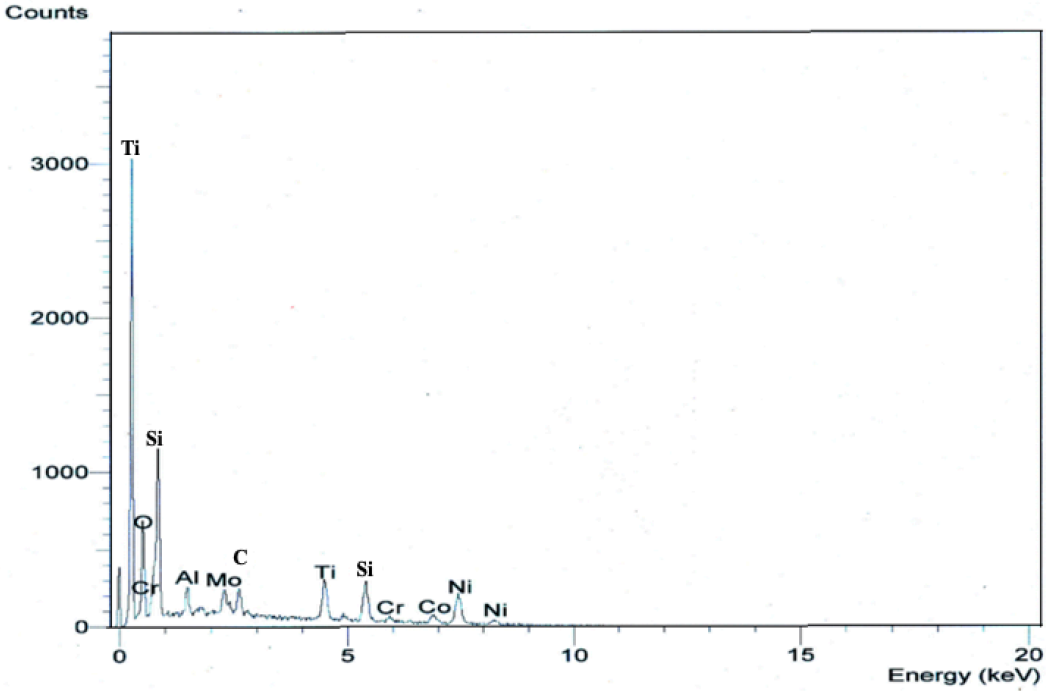


Figure 5. EDS spectrum analysis of Ti/SiC composites.

The cracks and voids observed in the images further confirm the onset of internal stresses due to oxidation. As oxide layers develop, they apply pressure on the composite matrix owing to the disparate thermal expansion coefficients of the oxides and the substrate, resulting in microcracking. This is a prevalent result in high-temperature oxidation procedures. The production of TiO_2 and SiO_2 modifies the material's characteristics, diminishing its overall structural integrity. Although TiO_2 offers a measure of surface protection in specific applications, the oxide layers may also increase the brittleness of the composite, resulting in diminished mechanical strength and wear resistance.

The sintering process at 1000°C induces substantial oxidation in the Ti/SiC composite, resulting in the creation of TiO_2 and SiO_2 compounds. The transformation is apparent in both the SEM pictures and the mechanical results, since the oxidation process causes

significant alterations in the microstructure, including observable cracks and porosity. The structural modifications directly influence the mechanical properties of the composite.

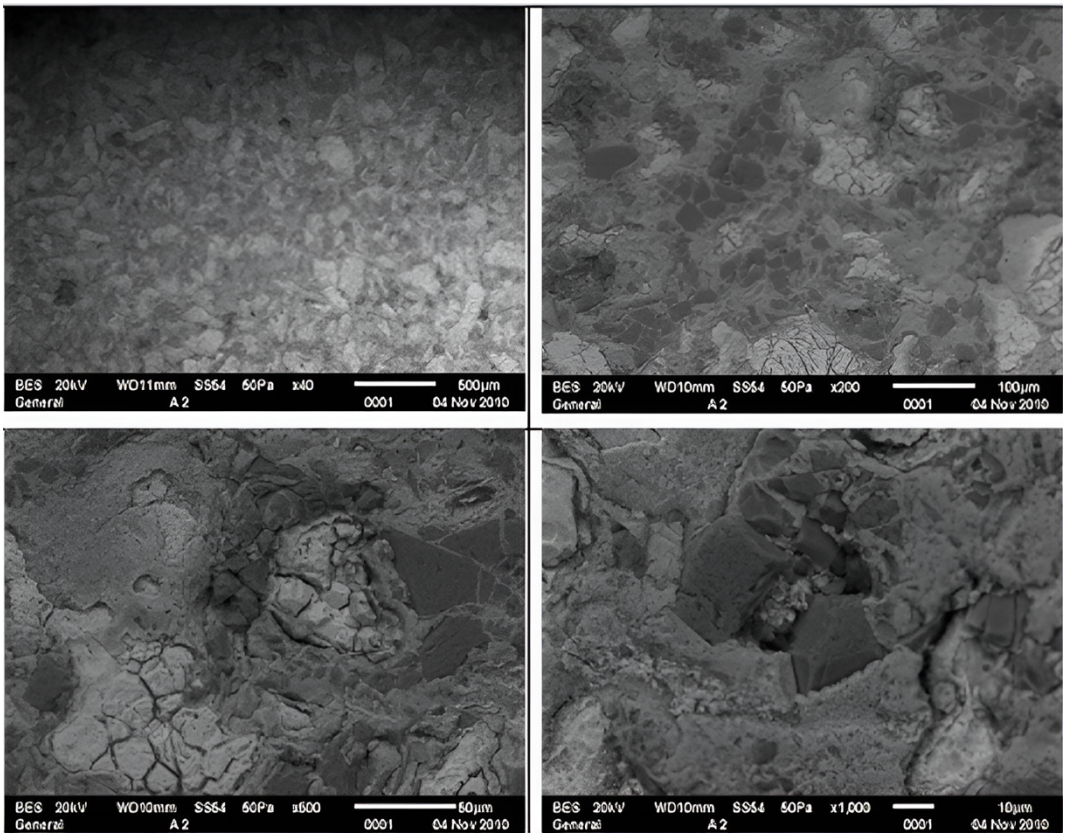


Figure 6. Microstructural evolution and oxidation-induced cracking in Ti/SiC composites at 1000°C through SEM analysis

Data in Table 1 indicates that at a sintering temperature of 1000°C for a 70% Ti : 30% SiC volume fraction, the sample exhibits brittleness, which complicates hardness measurement.

The formation of brittle oxide phases reduces the material's structural integrity, making it unsuitable for high-temperature applications. Brittleness in the sample sintered at 1000°C with a 70:30 volume fraction results from oxidation, evidenced by the formation of TiO_2 and SiO_2 compounds along with fractures and porosity in the microstructure.

Figure 8 illustrates the brittleness mechanism caused by oxidation-induced structural damage, leading to diminished mechanical properties. Pramono et al. [16] similarly explored how titanium can produce tricalcium phosphate (TCP) in hydroxyapatite (HAp)-based composites, observing a high-temperature phase transformation that affects the composite's mechanical properties. This phase transition impacts oxide compound formation in Ti/SiC composites, contributing to brittleness. In HAp-based composites, the phase change to TCP also decreases material stability and functionality.

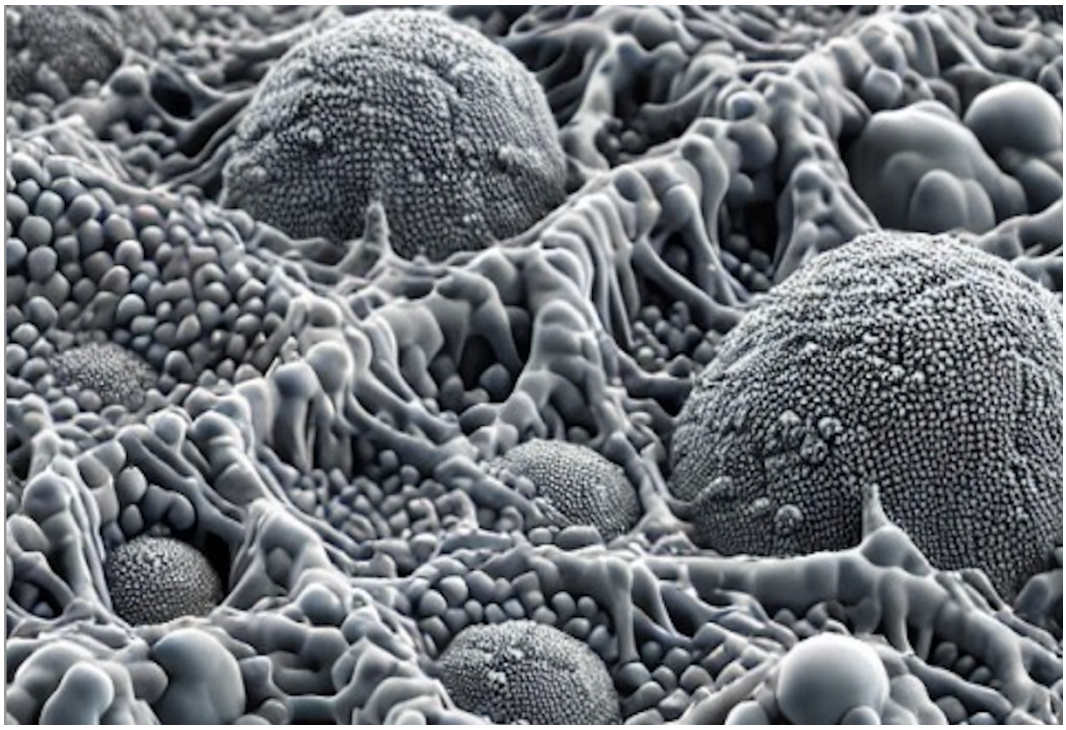


Figure 7. SEM analysis for the effects of sintering at 1000°C on the Ti/SiC composite with a volume fraction of 70% titanium and 30% silicon carbide, inset at 2500x.

CONCLUSION

This research demonstrates that sintering parameters and volume fraction significantly influence the mechanical properties and microstructural stability of Ti/SiC-based composites. The following main conclusions can be drawn:

1. An ideal sintering temperature of 900°C is recommended, particularly for a 70% Ti to 30% SiC composition, as it optimizes the balance between density and porosity, resulting in low porosity and superior hardness.
2. Sintering at 1100°C, while increasing density, leads to higher porosity, the formation of microcracks, and coarse grains due to oxide production (TiO_2 and SiO_2), ultimately compromising mechanical integrity.
3. The study also reveals that a 70% Ti to 30% SiC composition exhibits improved mechanical performance compared to a 60% Ti to 40% SiC ratio, further highlighting the critical role of volume fraction.

These findings emphasize the importance of controlling both sintering temperature and volume fraction to achieve superior composites with enhanced mechanical characteristics and structural stability.

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CONFLICT OF INTERESTS

The authors assert that there are no conflicts of interest pertaining to the publishing of this paper. All procedures were executed with complete transparency, and the research was undertaken autonomously, devoid of any financial or personal affiliations that could improperly affect the data or interpretations offered in this study.

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