

Article

Mechanical Milling and Cold Pressing for the Fabrication of Porous SiC Ceramics via Starch Consolidation

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Abstract: Silicon carbide (SiC) is a highly valued material in structural ceramics due to its exceptional properties, including low thermal expansion, high mechanical strength, thermal conductivity, hardness, and corrosion resistance. These attributes make SiC suitable for a wide range of applications, from filters and electrodes to refractory and structural materials. In this study, SiC samples were produced under various conditions and characterized through techniques such as diffraction, SEM, TGA, and optical microscopy. The results indicated a band gap of 3.195 eV, an apparent density of 1.317 g/cm³, and Vickers hardness ranging from 1193 to 536 HV. Additionally, the Young's modulus of the sample was found to be 0.4 GPa. These findings demonstrate the potential of starch consolidation for the cost-effective production of SiC ceramics with promising mechanical properties.

Keywords: silicon carbide (SiC); mechanical milling; porous ceramics



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1. Introduction

Silicon carbide (SiC) is a widely used material in structural ceramics due to its exceptional properties, such as low thermal expansion, high mechanical strength, excellent thermal conductivity, hardness, abrasion resistance, and, most notably, its ability to maintain elastic strength at temperatures up to 1650 °C [1–4]. These characteristics have enabled SiC to be employed in a diverse range of applications, including filters, electrodes, refractories, and structural materials [5–7]. Porous SiC ceramics, in particular, have attracted significant attention because of their low density, high surface area, chemical stability, high-temperature resistance, and excellent thermal shock resistance [8–10]. These properties make SiC ceramics ideal candidates for various technological applications, such as gas filters, metal filters, heat exchangers, and catalytic membrane supports [11–13].

However, the manufacturing of SiC ceramics faces challenges, particularly in the sintering and molding processes, which typically require controlled atmospheres with gases such as nitrogen or argon and extremely high temperatures (~2500 °C) to avoid oxidation and overcome the strong covalent nature of the Si-C bond [14,15]. These requirements not only increase production costs but also limit the practical and commercial use of SiC ceramics. Consequently, current research is focused on developing methods for fabricating SiC ceramics at lower sintering temperatures without the need for expensive

additives or controlled atmospheres, offering the potential for more cost-effective and scalable production [14–16].

A promising technique in this context is starch consolidation, which has gained attention for its ability to produce high-quality SiC ceramics at relatively low temperatures in an air atmosphere [8,17]. This method involves mixing SiC powder with starch and water to form a paste, which is molded and dried before starch removal through calcination, followed by low-temperature sintering. Starch consolidation offers several advantages, including process simplicity, the ability to mold complex shapes and the potential to reduce production costs [17,18]. Moreover, this technique allows for better control of the material's homogeneity, enhancing the control of shrinkage during sintering, which is critical for achieving consistent dimensions of the final product [19,20]. However, challenges remain, such as ensuring complete starch removal and achieving uniform distribution of SiC powder and starch during molding, which can affect the final material properties [8,9].

Several studies have explored similar approaches to enhancing the properties of SiC ceramics. For example, Ahmed et al. discussed the starch consolidation process for creating porous ceramics with controllable porosity and microstructure, offering a flexible and cost-effective approach for fabricating SiC components [20]. Additionally, the study of porous SiC ceramics in relation to bending strength and porosity, highlighted by Kalem Tas et al., provides important insights into how modifying the porosity and pore size of SiC ceramics influences their mechanical performance [8]. These studies suggest that adjusting porosity and pore size can improve bending strength, which is crucial for applications requiring high mechanical resilience. Furthermore, investigations into energy-efficient fabrication methods, such as low-temperature sintering and direct consolidation, emphasize the potential to lower production costs and enhance the scalability of SiC ceramics for industrial use, particularly in the aerospace and automotive sectors [20–22].

In light of these advancements, this study aims to explore the use of starch consolidation for the production of SiC ceramics, focusing on improving their mechanical properties while evaluating particle size through mechanical grinding. The study will also investigate the effects of processing on band gap, hardness, tensile strength, flexural strength, and density.

2. Materials and Methods

The raw material used in this study was silicon carbide (silicon carbide powder RA-0050, Naniwa Abrasive MFG Co., Ltd., Osaka, Japan). Additional materials included decahydrate borax (Galvanoquímica Mexicana S.A. C.V., Mexico City, Mexico), corn starch (Unilever Manufacturera, Tultitlán, Mexico), kaolin (Molecular Cuisine Supplies MCS, Mexico City, Mexico), and distilled water.

A high-energy planetary mill (Retsch model PM100, Retsch GmbH, Haan, Germany) was used to reduce the particle size of silicon carbide to the micrometer scale. Milling conditions were based on a modified protocol reported in [2], with the aim of identifying the optimal conditions for achieving the smallest possible particle size. For sample fabrication, the following conditions were used: milling time of 12 h with 15 min cycles for direction change, at a speed of 300 rpm, using zirconia (ZrO₂) balls with a diameter of 0.3 cm in a 250 mL stainless steel cylindrical jar. The ball/powder weight ratio was 20:1. The decision to maintain the sample in the vial for 12 h was made to prevent contamination by steel particles, which began to detach from the jar after this time. Under these conditions, no significant difference in particle size was observed with longer milling times.

The preparation of the samples involved several sequential processes to ensure the desired characteristics of the final products. Particle size analysis was conducted on the milled silicon carbide to evaluate its distribution. Before analysis, the material underwent

ultrasonic treatment for 15 min. A 0.1 g sample of the powder was dispersed in 100 mL of distilled water, and the particle size distribution was measured using a Shimadzu model SALD-201V, (Shimadzu Corporation, Kyoto, Japan) particle size analyzer.

To achieve powder homogenization, the milled silicon carbide (55.93 wt%), was mixed with decahydrate borax (18.06 wt%), corn starch (23.49 wt%), and kaolin (2.5 wt%) in a container. A small amount of distilled water (0.5 mL) was added to facilitate mixing. The total weight of the homogenized powder mixture was 2.5 g.

Two different methods were employed to manufacture the samples: uniaxial compaction and mold cast. In the uniaxial compaction process, the homogenized powder was formed into tablets with a diameter of 2 cm and a thickness of 0.3 cm. Each tablet weighed approximately 2 g. Compaction was performed using a Montequipo LAB-30-T hydraulic press (Montequipo, Mexico City, Mexico), applying a pressure of 250 MPa. The powder was compacted in a steel die with a cylindrical cavity of 2 cm diameter, where the die remained stationary while the punch moved to compress the material in a uniaxial direction. For the mold cast method, a silicone mold was used to create samples with a diameter of 2.4 cm and a thickness of 0.35 cm. The homogenized powder mixture was poured into the mold in a wet state and dried in a dehydrator at 70 °C for 24 h.

Sintering was conducted on both compacted and molded green bodies using a Thermolyne model FD1535M electric furnace (Thermo Fisher Scientific, Waltham, MA, USA), as illustrated in Figure 1. The sintering temperature was set at 635 °C, based on thermogravimetric analysis indicating the degradation of borax between 600 °C and 720 °C (this is further discussed in the Results and Discussion section). The heating rate was controlled at 1 °C per minute, followed by a 1 h isothermal hold at the maximum temperature. Upon completing the sintering cycle, the samples were allowed to cool gradually within the furnace. Both types of samples, uniaxially compacted and cast-molded, were subjected to identical sintering conditions in terms of temperature and time.

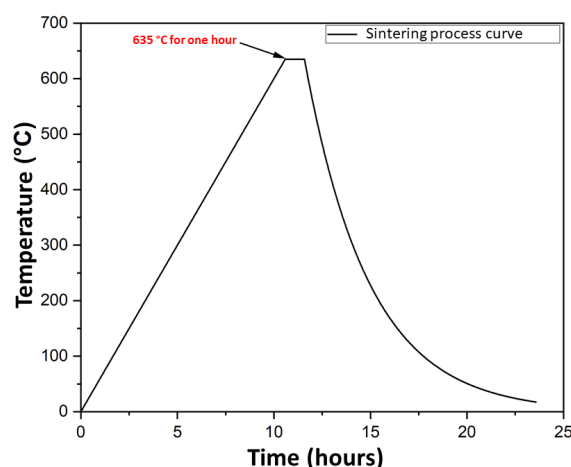


Figure 1. Sintering process for all samples.

Microstructure characteristics were analyzed by scanning electron microscopy (SEM) using a JSM-IT100 model from JEOL (JEOL Ltd., Tokyo, Japan). SEM analysis was carried out at voltages ranging from 1.5 kV to 4.0 kV. Additionally, X-ray diffraction (XRD) analysis was performed using a PANalytical X'Pert PRO model (Malvern Panalytical, Almelo, The Netherlands) equipped with Cu-K α radiation in order to identify crystalline structures in the resulting material. Thermogravimetric analysis (TGA) was used to investigate mass changes, and phase transitions of the samples and individual components, such as borax and kaolin. These analyses were performed with a SHIMADZU model DTG-60H Simultaneous DTA-TG Apparatus (Shimadzu Corporation, Kyoto, Japan). The mechan-

ical properties were also thoroughly evaluated. Vickers microhardness (HV) tests were conducted using a Wilson Instruments model 402 MVD (Wilson Instruments, Norwood, MA, USA) microhardness tester with a maximum load of 2 kgf. Four measurements per sample were taken, and the indentation diagonals (d1 and d2) were measured using the tester's integrated microscope. Compression testing was performed using a Gunt WP300 universal testing machine (Gunt Gerätebau GmbH, Hamburg, Germany) with a maximum capacity of 20 kN. A WP 300.20 data acquisition unit was employed to record the sample behavior under compressive loads, generating force–displacement graphs that provided quantitative data in kN. Both the density and porosity of the consolidated pellets were evaluated using the Archimedes principle. The band gap measurement was conducted using a quartz cuvette containing various samples dissolved in 3 mL of distilled water. The setup included a Thorlabs CCS200 spectrometer (Thorlabs Inc., Newton, NJ, USA) connected to a BFL200HS02 optical fiber and a deuterium lamp (NYP003) (Thorlabs Inc., Newton, NJ, USA). The band gap values were determined using the Tauc plot method. The density and porosity of the samples were also determined using the Arquimides method.

3. Results and Discussion

3.1. Particle Size Distribution

The milling process yielded a significant reduction in the particle size of silicon carbide (SiC), decreasing from an average of 50.20 μm to 0.42 μm , as shown in Figure 2. This reduction is attributed to the high-energy impacts and shear forces inherent in the milling process. Trials were conducted under both dry and wet conditions and for varying durations, highlighting the influence of the milling environment and time on particle refinement. Figure 2a shows the particle size distribution of the raw materials, exhibiting an average size of 50 μm . In contrast, the proposed milling process (Figure 2b) achieved an average particle size of 450 nm. Smaller particles are advantageous for sintering, as they promote densification due to their higher surface area and enhanced reactivity, which can improve mechanical properties such as hardness and compressive strength. These results align with the findings reported in reference [23].

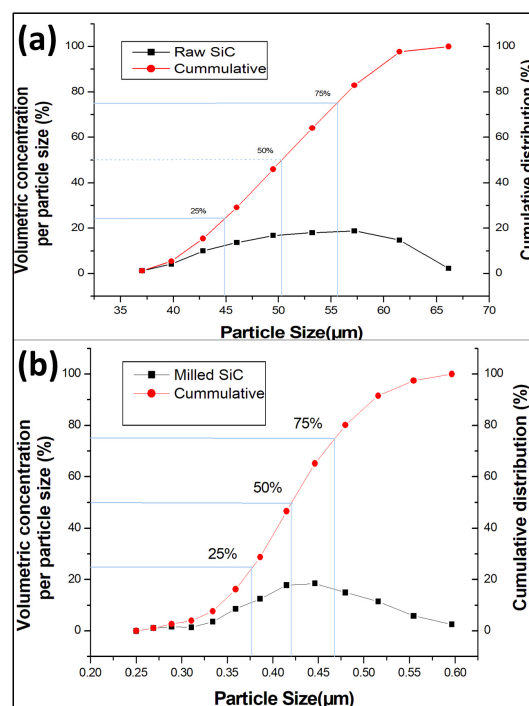


Figure 2. Particle size analysis: (a) raw SiC and (b) milled SiC after 12 h.

3.2. Thermogravimetric Analysis

Figure 3 presents the TGA and DTA results for the SiC samples prepared with borax and kaolin. The analysis was performed with a heating ramp of 1 °C per minute up to 900 °C. The first peak at 120 °C corresponds to dehydration, associated with water vapor release. A subsequent weight loss between 150 °C and 250 °C is attributed to the dehydration of crystalline water in pentahydrated borax. From 250 °C to 350 °C, the burning of amylopectin from the starch occurs, followed by the loss of amylose between 350 °C and 550 °C. At this stage, the starch is entirely removed, preparing the material for sintering. An exothermic peak at 577 °C is associated with the reorganization of pentahydrated borax. Between 577 °C and 680 °C, mass loss is linked to kaolin dehydroxylation, corresponding to the phase transition of $\text{SiO}_2 \cdot 2\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$ to $\text{SiO}_2 \cdot 2\text{Al}_2\text{O}_3$. This transformation corresponds to a weight loss of approximately 13–15% of the kaolin mass. Given the presence of 2.5% kaolin, the expected weight loss from kaolin alone would be around 0.33–0.38%.

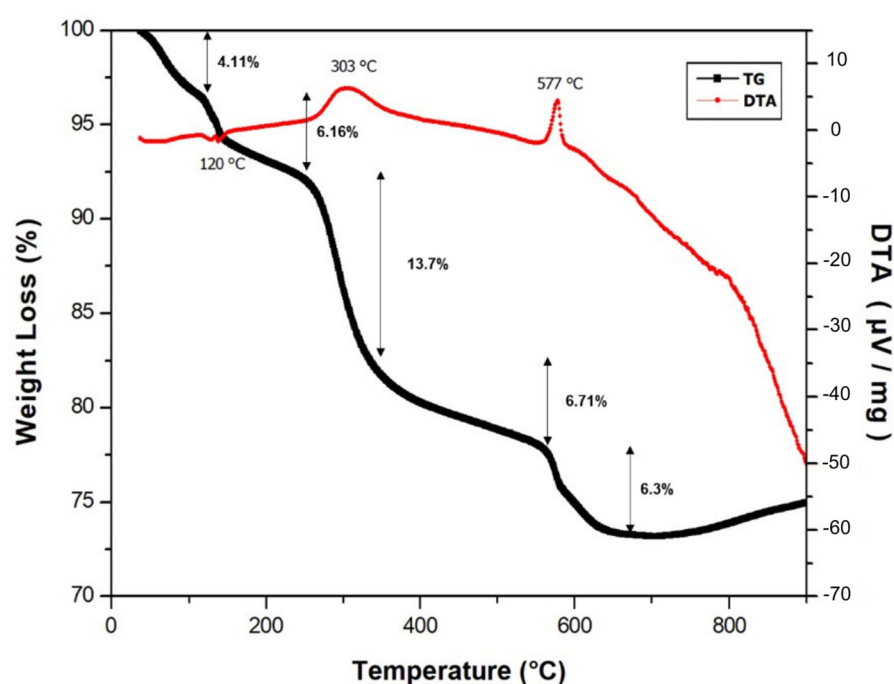


Figure 3. TGA analysis of SiC samples prepared with borax and kaolin, showing weight loss events up to 900 °C.

The discrepancy between the expected and observed weight losses suggests that additional dehydroxylation reactions from other phases (such as borate- or silica-rich compounds) may contribute to the 6.3% loss. Another phase transition occurs at 680 °C as kaolin converts to metakaolin, with a faint peak at 750 °C corresponding to its calcination temperature. No peaks were observed, indicating a liquid phase of borax. Alongside the water loss and calcination of kaolin, an optimal sintering temperature range between 630 °C and 650 °C was identified. The selected temperature for this study was 635 °C, as it was the minimum temperature required to solidify and form the samples.

3.3. X-Ray Diffraction

Figure 4 shows the X-ray diffraction patterns of the base SiC and the samples processed through milling and sintering. Specifically, two predominant polymorphs were identified: a cubic phase representing 70.5% of the base SiC sample and a rhombohedral phase accounting for 29.5%. In the milled sample, the proportions shifted to 40% cubic phase and 60% rhombohedral phase. The milling process resulted in a decrease in peak intensity,

attributed to the reduction in particle size from 50.20 μm to an average of 6.232 μm . Calculations indicate that the crystal size in the base sample was 279 nm, which decreased to 9 nm in the milled sample. Furthermore, the microstrain increased from 0.02% in the base sample to 0.363% in the milled sample, as expected, due to the high-energy milling process.

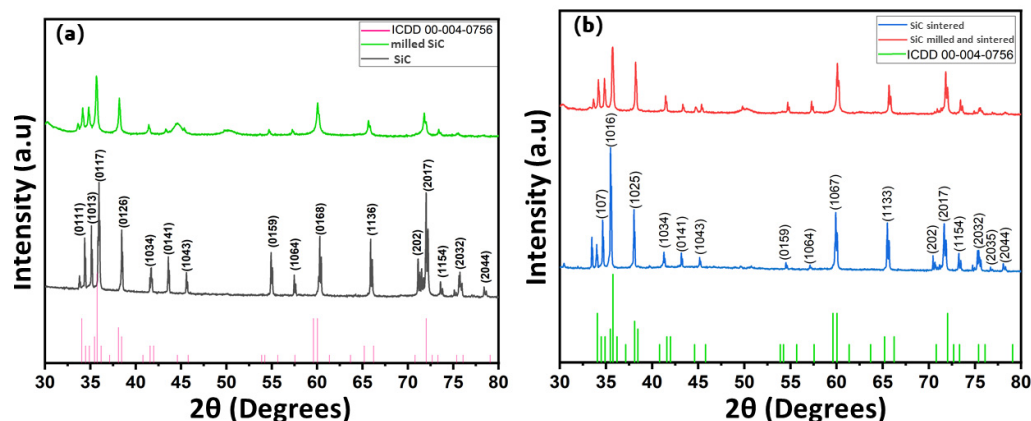


Figure 4. X-ray diffraction patterns highlighting the effects of processing on SiC samples: (a) the impact of milling on the base SiC and (b) the influence of the sintering process on the base and milled SiC samples.

In some samples, the presence of iron oxide was observed in quantities below 5%, resulting from the milling process conducted in a stainless steel vial. The literature suggests that increased milling intensity and duration elevate the likelihood of vial particle detachment and incorporation into the sample. Additives such as kaolin and borax were not detected, likely due to their low concentration in the sample.

Figure 4b illustrates the diffraction patterns of the sintered carbide samples, where the base carbide is shown in blue and the milled carbide in red. Both samples were analyzed post-forming and sintering processes. In the milled and sintered samples, a reduction in peak intensity was observed, consistent with the previously mentioned reasons. Crystal size calculations for the sintered base sample showed a value of 290 nm, while the sintered milled sample exhibited a reduced crystal size of 13 nm. Similarly, the microstrain in the sintered base sample was 0.04%, whereas no significant microstrain was recorded in the sintered milled sample due to the thermal treatment, which reduced the stresses induced by milling.

3.4. Microstructure

The microstructure of both the green and sintered samples, observed through optical microscopy, is presented in Figure 5. The two images correspond to the sample prepared by mixing silicon carbide (SiC) with borax, starch, and kaolin. In the green state in Figure 5a, limited detail can be observed due to the absence of sintering. However, SiC grains are prominent, characterized by their angular, sharp, or square-like shapes. At this stage, no significant porosity is detected since the starch and absorbed water remained within the sample. Upon sintering at 635 °C, as shown in Figure 5b, the starch decomposes, forming porosity, consistent with the thermal gravimetric analysis (TGA) previously discussed.

Post-sintering, the sample was re-examined under the microscope to analyze the resulting changes. Figure 5b reveals a more uniform and less defined structure compared to the green sample. The previously discernible SiC particles, individually identifiable in the green state, appear embedded within a matrix of agglomerating agents. The disappearance of starch is evident through the presence of newly formed pores (appearing as unfocused

regions). Additionally, the oxidation of SiC is observed, facilitated by the borax acting as a neck-forming agent. This process enables the bonding of SiC grains, resulting in a denser, more cohesive structure with improved mechanical properties, including enhanced strength and handling.

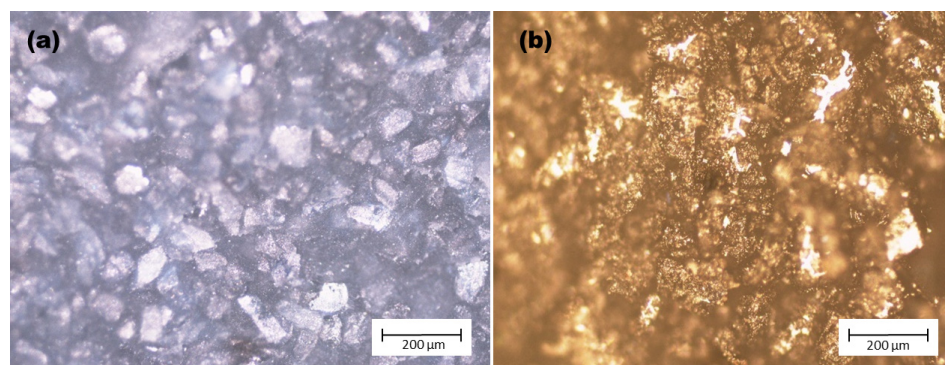


Figure 5. Micrography of (a) green sample prepared without milling and (b) the same sample after sintering.

Figure 6a,b illustrates the effect of sintering at 600 °C on SiC microstructure, with and without milling. In Sample (a), sintered at 600 °C without milling or compacting, the microstructure reveals distinctly separated SiC grains with well-defined edges and significant voids between particles. This indicates that 600 °C is insufficient to promote inter-particle diffusion or neck formation, leading to a lack of particle bonding. The observed rough surface and non-homogeneous structure suggest incomplete sintering, further highlighting the limitations of this temperature for achieving densification. In contrast, Sample (b), which was subjected to high-energy milling but not compacted before sintering at the same temperature, exhibits a finer microstructure due to the reduced particle size achieved during milling. However, the absence of compacting results in a porous structure with poor cohesion between the SiC grains. The presence of visible voids and lower densification in this sample confirms that applying pressure is essential to enhance particle contact and promote sintering. Overall, the comparison between Samples (a) and (b) demonstrates that while milling improves microstructural refinement, the absence of compacting at 600 °C prevents significant densification and particle bonding. This highlights the need for both higher sintering temperatures and compacting processes to achieve improved microstructural characteristics in the SiC samples.

In Figure 6c,d, both samples illustrate the influence of compacting and sintering parameters on the resulting SiC microstructure. Sample (c), which was not compacted prior to sintering, shows partial bonding between particles with significant voids and agglomerations of powders. The surface exhibits roughness with poor crystallization and heterogeneity, likely due to the rapid heating ramp of 10 °C/min, which prevented complete borax crystallization. The voids observed on the surface range from 20 µm to 469 µm, indicating insufficient densification. Internally, the porosity is considerable, with spaces between unsintered crystals mirroring the surface characteristics. These observations highlight the limitations of sintering without compacting, where particle cohesion is minimal, and the resulting microstructure remains porous and heterogeneous. In contrast, Sample (d), which underwent compacting before sintering, exhibits a more favorable and homogeneous structure both on the surface and internally. The particle bonding is noticeably improved, and the porosity is reduced compared to Sample (c). The pores in this sample are more uniform in size, ranging from 3 µm to 15 µm, with smaller, presumably open pores located within larger ones, contributing to the overall porous network. This structure is attributed to the extended sintering time, which allowed for better particle diffusion and

consolidation. Additionally, the precursor powders in this sample were likely subjected to milling and sieving, which further reduced particle size and improved packing density prior to sintering.

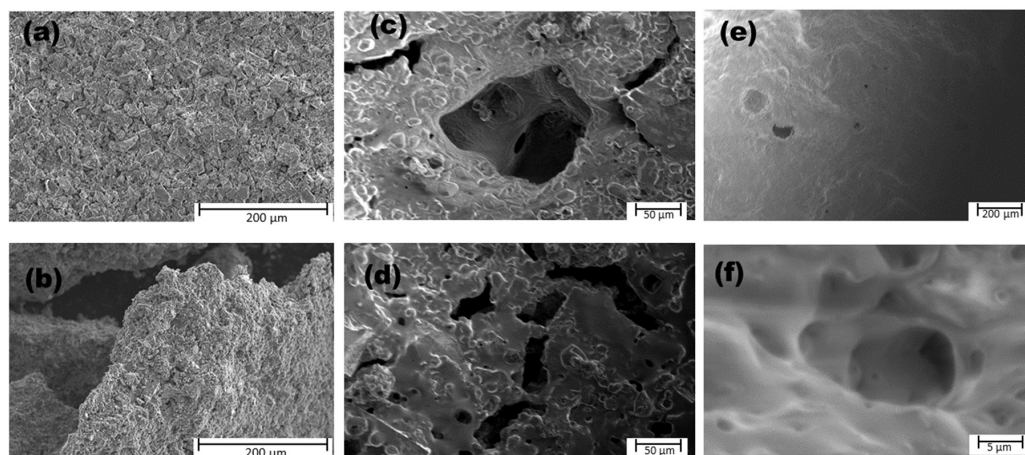


Figure 6. Effect of sintering temperature, milling, and compacting on the microstructure of the SiC samples: comparative analysis at 600 °C and 635 °C. (a) Non-milled and non-compact SiC sample sintered at 600 °C, (b) milled but non-compact SiC sample sintered at 600 °C, (c) non-milled and non-compact SiC sample sintered at 635 °C, (d) non-milled but compacted SiC sample sintered at 635 °C, (e) microstructure of milled and compacted SiC sample sintered at 635 °C, and (f) magnified view (3000×) of the SiC samples (e).

The comparison between Samples (c) and (d) demonstrates the critical role of pressure application during pre-sintering preparation and optimized sintering conditions (heating rate and time). Compacting enhances particle contact, leading to improved densification and more uniform porosity, while rapid heating ramps hinder complete crystallization and bonding, resulting in agglomerated and porous structures. These findings confirm that achieving a homogeneous and well-bonded microstructure in SiC materials requires careful control of both pressure and sintering parameters.

Sample (e) exhibited the best results in this series. Milled and compacted before sintering at 635 °C, the sample shows a highly uniform microstructure with improved particle connectivity and significantly reduced void spaces. Milling contributed to a smaller particle size, enabling better packing, while compacting enhanced particle contact, facilitating effective sintering. A magnified view of Sample (f) (3000×) reveals well-defined sink marks embedded within the SiC matrix, indicative of controlled porosity formation. The presence of pores smaller than 5 μm suggests partial closure under the applied sintering conditions. Overall, the samples achieved excellent consolidation, with no visible unsintered particles. Their structural integrity was further confirmed during mechanical handling, where they appeared notably solid and robust, validating the effectiveness of milling, compacting, and the sintering process at 635 °C.

These results confirm that higher sintering temperatures (635 °C), combined with pre-milling and compacting steps, significantly enhance particle bonding and densification in the SiC samples. The comparison between 600 °C and 635 °C clearly demonstrates that the lower temperature is insufficient for complete sintering, while the combination of compacting and milling at 635 °C yields the most favorable microstructural characteristics. Furthermore, based on our experimental results and the theoretical batch composition, the final material consists of approximately 85 wt% SiC and 15 wt% of a bonding phase, as suggested. The estimated composition of the bonding phase is close to (29 mol% Na₂O + 58 mol% B₂O₃ + 6.4 mol% Al₂O₃ + 12.9 mol% SiO₂), indicating a sodium borosilicate-based glassy phase. This phase appears to be more effective in

samples with smaller particle sizes and, when combined with uniaxial pressing, further promotes adhesion between SiC grains, leading to improved densification and microstructural integrity. These sintering results are comparable to those reported in other studies that employed similar methodologies, obtaining SiC samples with porosity through starch consolidation [8,13,17]. This demonstrates that the proposed conditions in this work effectively enable the control of porosity and mechanical properties, making them a viable approach for tailoring the microstructure of porous SiC ceramics.

3.5. Energy-Dispersive Analysis (EDX)

The samples were also characterized using EDX with a JEOL JSM-IT100 system (JEOL Ltd., Tokyo, Japan). Figure 7 presents the energy-dispersive spectroscopy analysis of the base silicon carbide (SiC) material used for all the samples in this study. Figure 7a shows that large, sharp particles are observed, exhibiting varying sizes but similar shapes, with well-defined, striated faces characteristic of a high-hardness material. No evidence of surface porosity is observed. The average grain size exceeds 50 μm . Conversely, Figure 7b: The EDX spectrometer analysis shows the base SiC material, identifying the presence of Si and C. The remaining detected elements, Fe, Al, and O, are trace impurities introduced during manufacturing.

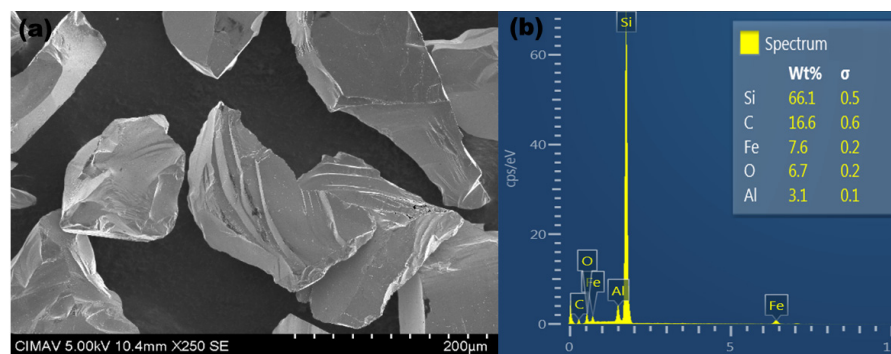


Figure 7. SEM image of raw silicon carbide powders (a) and EDX spectrum (b).

In Figure 8, the SEM reveals the pure silicon carbide subjected to the planetary milling process. Compared to the previous figure, the grain size in this sample decreased to less than 10 microns due to the milling process. These observations align with the particle size distribution analysis, which identified particles even in the nanometer range. The EDX spectrum in Figure 8b shows the same elements as the base material, along with additional contaminant elements introduced during mechanical milling, such as Fe, Cr, and Zr. These contaminants are attributed to the stainless steel container, zirconia balls, and the milling conditions used.

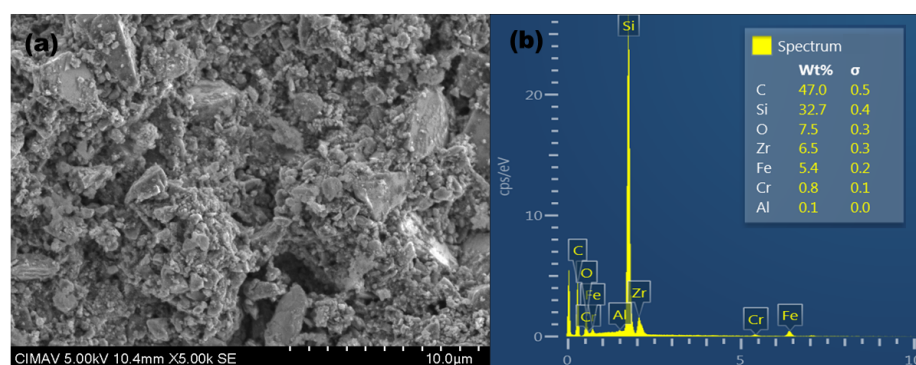


Figure 8. Silicon carbide powders obtained from the planetary mill, sem image (a) and EDX spectrum (b).

3.6. Band-Gap Determination

As shown in Figure 9, a decrease in the band gap was observed in milled samples compared to unmilled ones. This reduction can be attributed to defects introduced during the milling process, as evidenced by the X-ray diffraction (XRD) results, where a higher microstrain was detected in the milled samples. The reduction in the band gap is also closely related to the decrease in crystal structure size and grain size. The band gap of a material is strongly influenced by its crystal size; when subjected to planetary milling, the crystal size decreases, resulting in a reduced band gap. However, milling time plays a critical role in determining the final crystal size. Prolonged milling can lead to particle agglomeration, which increases the crystal size and, consequently, the band gap [24]. In this study, a 12 h milling process was employed, divided into two 6 h intervals, with the milling direction reversed every 15 min. Ethanol was used as the milling medium, effectively preventing particle agglomeration and maintaining a reduced crystal size. This approach resulted in a lower band gap value [24].

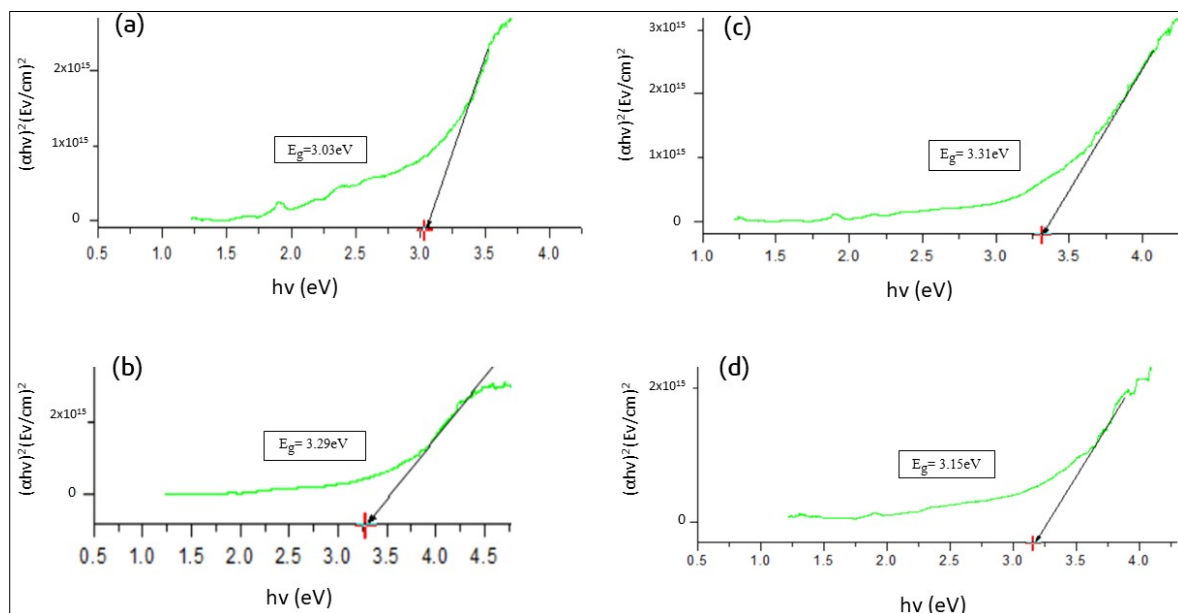


Figure 9. Band gap measurements: graph of (a) milled SiC without sintering, (b) milled SiC after sintering, (c) non-milled SiC after sintering, and (d) non-milled SiC without sintering.

In Figure 9b, the milled and sintered SiC sample exhibited an increase in its band gap to values similar to those of the unmilled and sintered SiC samples (c) and (d). This behavior is attributed to the sintering process, which promoted crystal growth and mitigated the defects caused by milling. Nanoparticles synthesized through mechanical milling often exhibit a broader particle size distribution, lower crystallinity, and higher defect density compared to other methods, such as those synthesized by chemical vapor deposition (CVD), which typically produce nanoparticles with a more uniform size and higher crystallinity [25,26].

3.7. Physical Characteristics

Figure 10 shows optical microscope images of Vickers indentation marks obtained from SiC samples subjected to milling, compaction, and sintering processes. In both images (a and b), the material exhibits low plasticity, as no corner cracks are visible at the edges of the indentations. The indentations observed in Figure 10 are not perfectly defined, particularly for the samples without milling or compaction, due to the uneven surfaces caused by their higher porosity.

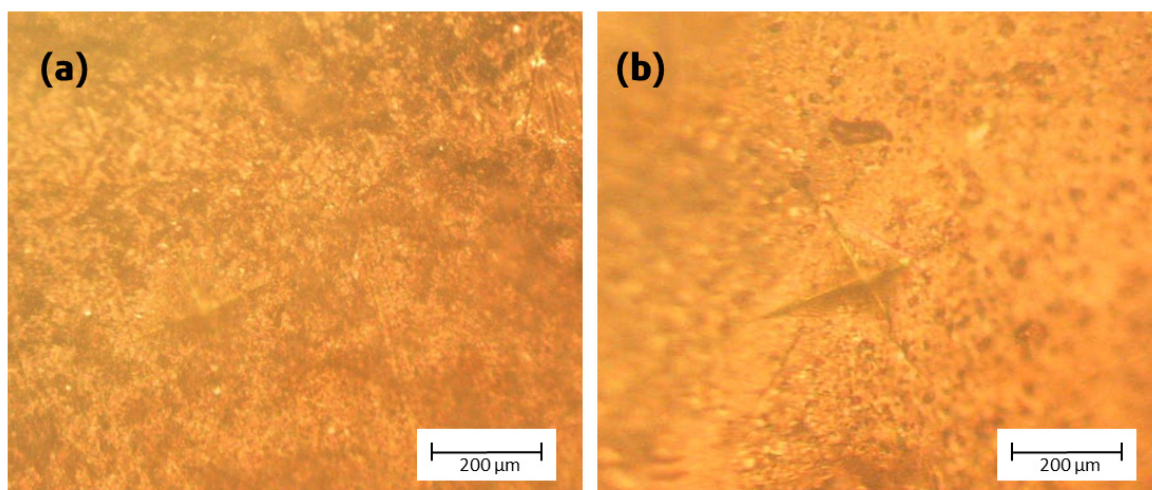


Figure 10. Vickers indentation marks on SiC samples with higher density: (a,b) correspond to samples processed through milling, compaction, and sintering, as described in this study.

In comparison to pure SiC, which has a reported hardness of 2600 HV [23], all processed samples showed significantly lower hardness values. The highest hardness measured was 1193 HV, with a standard deviation of ± 60 HV, for the samples subjected to milling, compaction, and sintering at 632 °C. On the other hand, the lowest hardness was observed in samples without milling or compaction, with a value of 586.6 HV and a standard deviation of ± 100 HV. The large standard deviation and reduced hardness reflect the high porosity and challenges in achieving uniform compaction in these samples.

Table 1 presents the results of relative density, porosity, Vickers hardness, and Young modulus measurements for the evaluated samples. The data indicate that the best densification was achieved in the samples that underwent milling, compaction, and sintering. Conversely, samples without the milling and compaction stages exhibited significantly lower densification.

Table 1. Results of relative density, porosity, and Vickers hardness for SiC samples: (a) raw materials after sintering, (b) raw materials with compaction, (c) milled powders after sintering, and (d) milled powders with compaction and sintering.

Sample	Density %	Porosity%	Hardness (HV)	Young Modulus (Gpa)
a	34.6 ± 7	65.4	536 ± 100	537 ± 100
b	37.4 ± 12	62.6	620 ± 80	0.04 ± 0.2
c	49.8 ± 7	50.2	800 ± 150	0.1 ± 0.3
d	68.5 ± 15	31.5	1193 ± 60	0.2 ± 0.5

Samples (a) and (b) showed no significant difference in densification compared to samples (c) and (d), which underwent milling. The mechanical testing of these highly porous materials revealed considerable challenges, as reflected in the high standard deviation values. Under mechanical stress, many regions within the material collapse, making reliable measurements difficult. None of the samples reached the theoretical density of pure SiC; however, the proposed methodology demonstrated that mechanical milling and uniaxial compaction under the described sintering conditions effectively reduced porosity and improved mechanical properties.

4. Conclusions

Through the proposed methodology, multiple samples were fabricated with slight modifications to the initial procedure to compare the results and identify the optimal approach. Nanoparticles were successfully obtained via mechanical milling, which was optimized by evaluating various milling durations, ball-to-powder ratios, and operational conditions. The reduction in particle size achieved through milling, combined with the compaction process using a hydraulic press, resulted in samples with superior structural properties. This improvement was corroborated by the SEM, optical microscopy, and mechanical testing results.

X-ray diffraction analysis identified two predominant polymorphs of SiC—cubic and rhombohedral. Significant changes in their proportions were observed between the base SiC samples and the milled ones. The milling process substantially reduced the particle and crystallite sizes, impacting the intensity of diffraction peaks and increasing microstrain in the milled samples compared to the base ones. The smaller particle and crystallite sizes contributed to enhanced microstructural uniformity, which improved specific mechanical properties. Additionally, the smaller particles achieved higher temperatures during sintering, promoting greater crystallization and resulting in a more homogeneous and enhanced structure.

SEM analysis confirmed the presence of a homogeneous mixture in the samples, with distinct elemental distributions evident across the different specimens. Impurities were observed due to both the mechanical milling process and the inherent manufacturing contaminants. Moreover, nanoscale particle sizes were confirmed. Variations in pore sizes were also evident, as observed by SEM, leading to differences in the density of the samples. Compared to the control samples, the milled samples exhibited fewer pores, improving their overall properties.

The starch consolidation method was demonstrated to be an effective and cost-efficient approach for fabricating consolidated SiC samples. This technique allows for control over porosity, density, and mechanical properties, making it suitable for a range of applications. The consolidated samples displayed excellent adhesion and structural integrity.

Mechanical testing of the fabricated samples indicated that those subjected to mechanical milling performed best. The milling process significantly enhanced the band gap, with values of 3.03 eV in unsintered samples and 3.29 eV in sintered ones, surpassing the control sample that was not milled. The reduced particle size contributed to greater cohesion and stability, as reflected in the hardness tests, which yielded values of around 1193 HV. Compression testing revealed a maximum load of 3000 N, corresponding to a compressive strength of 40 MPa. The increased density of the milled samples further contributed to their durability, preventing disintegration and enhancing their mechanical performance.

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