

Research Article

Densification Behavior of Fused Silica: Effects of Particle Size, Compaction Pressure, Sintering Temperature/Time, and Boron Oxide Addition

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ABSTRACT

This study systematically investigates the optimization of processing parameters for 400-mesh Chinese fused silica ceramics, focusing on milling, blending, and sintering strategies to achieve high-density components. Twenty-hour attrition milling with zirconia balls (40:1 ball-to-powder ratio) produces a homogeneous powder with an average particle size of $\sim 5 \mu\text{m}$, enhancing sinterability compared to unmilled powder. Excessive milling, however, reduces green density due to increased interparticle friction. A 70:30 blend of milled/unmilled powder provides an optimal balance between enhanced diffusion kinetics (from milled powder) and improved particle packing (from unmilled powder). This blend achieves 94% theoretical density after sintering at 1250°C for 5 hours, while reducing milling energy by 30%. Prolonged sintering ($> 5 \text{ h}$) promotes cristobalite formation, grain growth, and density reduction, underscoring the importance of precise thermal control. B_2O_3 additions (0.5–3 wt.%) hinder densification by promoting premature crystallization and disrupting diffusion pathways, making it unsuitable as a sintering aid. SEM analysis confirms that the 70/30 blend yields uniform grains ($1.5 \pm 0.3 \mu\text{m}$) with minimal porosity ($< 6\%$), outperforming both fully milled and unmilled powders. These findings provide a practical framework for industrial production, emphasizing cost-effective blending and optimized sintering protocols to achieve high-performance fused silica ceramics.

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1. Introduction

Fused silica is one of the most technologically important ceramic materials, offering an exceptional combination of properties that make it indispensable for advanced engineering applications. It exhibits an ultra-low thermal expansion coefficient ($\sim 0.5 \times 10^{-6} \text{ K}^{-1}$), outstanding thermal shock resistance (> 30 cycles between $25\text{--}1100$

$^\circ\text{C}$), low thermal conductivity ($2.09 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$), and excellent dielectric characteristics ($\epsilon = 3.1\text{--}3.8$; $\tan \delta = 0.0004$) [1-5]. Along with its high volume resistivity ($10^{15} \Omega\cdot\text{m}$ at room temperature) and superior corrosion resistance, these attributes enable wide use in high-purity crucibles for semiconductor processing, microwave-transparent components for RF systems, and thermal

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protection systems in aerospace vehicles [6-11].

However, the extremely high melt viscosity of fused silica requires processing temperatures of 1800-2000 °C, making direct fabrication of complex shapes highly challenging [4, 5]. To overcome this limitation, sintered fused silica (sometimes referred to as "quartz ceramics") was developed by pulverization fused silica cullet followed by conventional ceramic processing techniques [4, 5, 10]. The concept was first demonstrated by Skaupy and Weissenberg [4], who sintered quartz sand at 1650-1680 °C. Modern processing employs finely milled waste silica glass [5, 10] formed through various methods including hot pressing [12], slip casting [5, 10, 13], and thermoplastic techniques [5], with slip density directly influencing final casting density [13]. As reviewed by Budnikov and Pivinskii [10], such techniques allow fabrication of complex geometries unattainable through direct melting.

A critical challenge in sintered fused silica processing arises because its typical sintering temperature (1300 °C) coincides with the crystallization threshold [14-16]. The formation of cristobalite during sintering is undesirable due to its disruptive α - β phase transition at 230 °C, accompanied by 2.8% volume change [14-16]. Even a cristobalite content as low as 5% can cause mechanical failure while inhibiting densification [15, 16]. This creates a critical processing trade-off where elevated temperatures (> 1300 °C) enhance densification but simultaneously promote cristobalite formation, leading to a reduction in flexural strength of up to 40% [14, 15, 17].

Recent research has focused on two primary strategies to overcome these limitations: advanced sintering techniques and crystallization inhibitors. Conventional pressureless sintering, while capable of achieving moderate densities (> 90% theoretical), remains constrained by the temperature-crystallization trade-off [18, 19]. Spark plasma sintering (SPS) has emerged as a promising alternative, with preliminary studies demonstrating its potential for fused silica [20, 21]; however, critical knowledge gaps remain regarding parameter optimization for crystallization control [20, 21]. Concurrently, additive approaches have shown

significant promise, with B₂O₃ (3 wt.%) reported to suppress cristobalite formation while achieving 1.89 g/cm³ density and 52 MPa flexural strength [22], and BPO₄-B₂O₃ composites (9 wt.%) shown to promote glass phase formation between 1200-1400 °C while effectively inhibiting crystallization [23].

The current state of research suggests that pure fused silica crystallizes slowly at 1300 °C (0.01-0.1 μ m/min in air) with bulk glass remaining stable for up to 60 hours [16]. Furthermore, certain impurities, particularly glass formers like B₂O₃, can significantly inhibit crystallization rates [16, 22, 23]. These findings highlight the importance of controlled processing conditions and additive formulations in optimizing the microstructure and properties of fused silica ceramics, while also highlighting promising avenues for future research in advanced sintering techniques and composite systems.

Building on these insights, this study systematically investigates the effects of particle size modification via attrition milling, powder blending ratios, sintering temperature/time optimization, and B₂O₃ addition on the densification and microstructural evolution of fused silica ceramics. The novelty of this work lies in identifying the optimal combination of milled/unmilled powder ratios and thermal schedules that maximize density while minimizing energy consumption and avoiding cristobalite-related degradation.

2. Experimental Procedure

2.1. Materials and methods

In this study, fused silica ceramic powder with an average particle size of 400 mesh (produced in China) was used. The as-received fused silica powder had a typical chemical purity of > 99.5% SiO₂. To modify the particle size and size distribution, the powder was subjected to attrition milling using 5 mm zirconia balls with a ball-to-powder ratio of 40:1. Ethanol was added to the ball-powder mixture to fully cover the milling balls. The milling chamber was then sealed and mounted on a dedicated drill-based milling machine. Milling was carried out at 250 rpm for 20 hours. Afterward, the slurry was poured into a beaker, and the ethanol was evaporated

by placing the beaker in an oven at 150 °C for 2 hours to recover the processed powder.

Additionally, the effect of adding different amounts of B₂O₃ (0.5 to 3 wt.%) to the fused silica powder on sintering behavior was also investigated. To improve density, different ratios of 20-hour milled and unmilled fused silica powders were blended in a jar mill with a ball-to-powder ratio of 10:1, using ethanol as the mixing medium for 24 hours. The resulting slurry was then dried in an oven under the same conditions mentioned above to obtain dry powder mixtures.

For the production of green compacts, uniaxial pressing was employed. Disc-shaped samples were prepared by weighing 0.65 g of fused silica powder and loading it into a steel die with an internal diameter of 12 mm. The powder was then pressed at pressures ranging from 60 to 120 MPa.

To study the influence of sintering temperature and holding time on densification, the samples were sintered in air at 1200 and 1300 °C for 5 to 20 hours. The density of the sintered specimens was measured using the Archimedes water immersion method, while the green density of the samples was determined through dimensional analysis with digital calipers (± 0.01 mm accuracy) and an analytical balance (0.1 mg resolution). Particle size distribution was analyzed using a laser diffraction particle size analyzer (Analysette 22 NanoTec, Fritsch, Germany). The morphology and microstructure of the sintered samples were examined using scanning electron microscopy (SEM). Prior to SEM imaging, fracture surfaces were gold-coated to enhance conductivity and improve image quality.

3. Results and Discussion

The particle size distribution (PSD) of the as-received 400-mesh Chinese powder and the 20-hour milled powder is presented in Figs. 1 and 2, respectively. The PSD curves reveal that the 20-hour milled powder exhibits a more uniform particle distribution, with an average particle size of approximately 5 μ m. In contrast, the unmilled powder shows a broad and irregular distribution, including a significant fraction of coarse particles exceeding 100 μ m. Such heterogeneity, particularly the presence of oversized particles, is undesirable to achieving optimal sintering performance.

Fig. 3 presents scanning electron microscopy (SEM) images of the unmilled and 20-hour milled 400-mesh Chinese fused silica powder. The micrographs clearly reveal a substantial reduction in particle size and improved size uniformity in the milled powder compared to its unmilled counterpart, demonstrating the necessity of the milling process. The unmilled powder exhibits large, irregular agglomerates with significant size variation, while the 20-hour milled powder shows a more homogeneous distribution of finer particles with well-defined morphology. This refinement in particle size and uniformity is critical for enhancing sinterability, as it promotes better packing density and more uniform densification [24]. The morphological changes align closely with the particle size distribution data shown in Figs. 1 and 2, confirming that 20-hour milling effectively produces powder characteristics suitable for optimized processing. Such structural modifications are especially important for achieving controlled densification and minimizing defects during sintering.

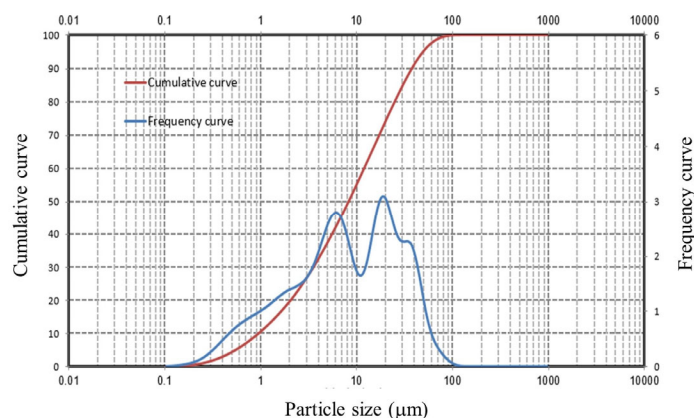


Fig. 1. Particle size distribution (PSD) of the as-received 400-mesh Chinese fused silica powder before milling.

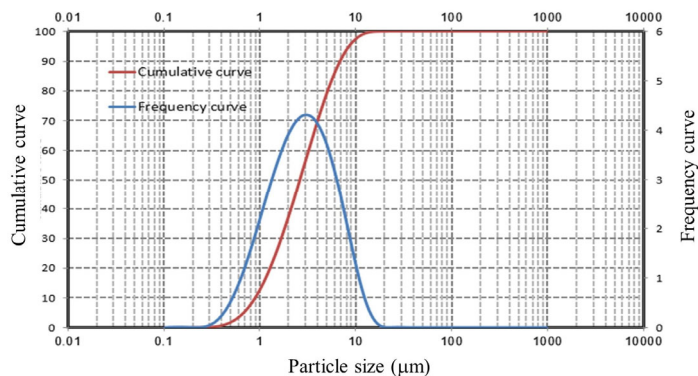


Fig. 2. Particle size distribution of 400-mesh Chinese fused silica powder after 20 hours of attrition milling.

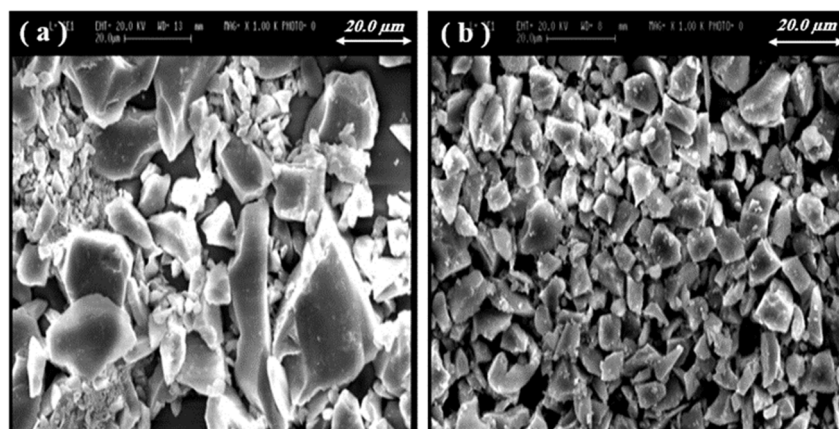


Fig. 3. SEM images of 400-mesh Chinese fused silica powder at 1000× magnification: (a) as-received (unmilled) and (b) after 20 hours of milling.

The green density data presented in Fig. 4 demonstrates key trends in the compaction behavior of both unmilled and 20-hour milled 400-mesh Chinese fused silica powder. As the compaction pressure increases from 60 to 120 MPa, the green density of the samples improves progressively, reaching an optimum before additional pressure leads to mechanical failure of the green bodies due to particle fracture. However, the 20-hour milled powder consistently shows lower green density values compared to the unmilled powder at equivalent pressures, despite having a more uniform particle size distribution. This apparent contradiction arises from the higher interparticle friction in the milled powder, caused by its finer particle size and increased specific surface area, which hinder efficient particle rearrangement during compaction. The reduced compactibility of milled powders represents a well-known trade-off in ceramic processing, where particle refinement enhances sintering potential but compromises green density [24]. These findings emphasize the need to

carefully balance milling parameters, compaction pressure, and powder characteristics to optimize the processing of fused silica ceramics.

3.1. Analysis of sintering temperature effects

The densification behavior of pressed fused silica samples (120 MPa compaction pressure) was investigated as a function of sintering temperature and milling time, as shown in Fig. 5.

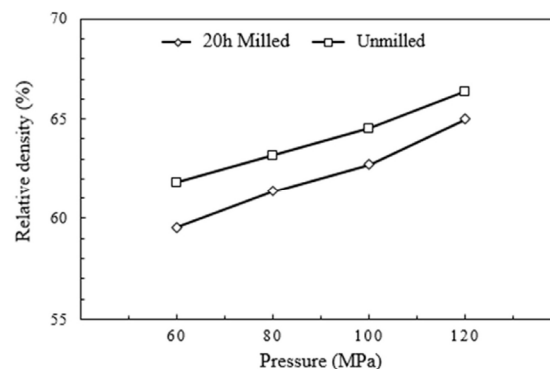


Fig. 4. Variation in green density of pressed samples produced from (a) unmilled and (b) 20-hour milled 400-mesh Chinese fused silica powder at different compaction pressures.

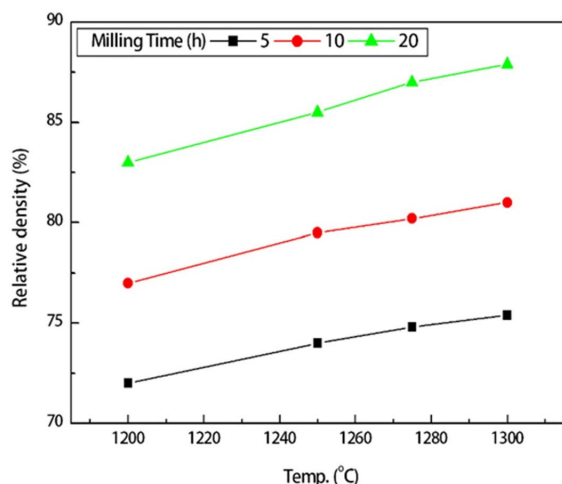


Fig. 5. Density of pressed samples fabricated from 400-mesh Chinese fused silica powder with different milling durations after sintering at various temperatures for 5 hours.

The results indicate that increasing sintering temperature enhances densification up to an optimal range of 1250–1275 °C, primarily due to reduced porosity and enhanced grain boundary diffusion. Notably, samples prepared from 20-hour milled powder achieved higher densification than those from unmilled powder, reaching 88% relative density at 1275 °C, attributed to finer particle size, improved packing uniformity, and more active diffusion pathways.

However, beyond 1275 °C, the conversion of fused silica to cristobalite introduces competing effects. While cristobalite has a higher theoretical density (2.33 g/cm³

vs. 2.20 g/cm³ for amorphous silica), its formation disrupts sintering kinetics by creating heterogeneous grain boundaries and reducing diffusion rates by approximately 40%. As a result, densification reaches a plateau despite the intrinsically higher density of the crystalline phase.

SEM analysis (Fig. 6) confirms that extended milling (20 hours) produces a more uniform microstructure after sintering at 1200–1250 °C. However, at higher temperatures (1275–1300 °C), the apparent 'finer' microstructure is attributed to the surface nucleation and growth of fine cristobalite crystallites, which disrupt the sintering process and lead to the observed plateau in density. While microstructural refinements promote densification at lower temperatures, it simultaneously increases densification at higher temperatures. These results underscore the delicate balance between milling duration, sintering temperature, and phase stability in optimizing the densification of fused silica ceramics.

Fig. 7 presents a comparative SEM analysis of fused silica samples sintered at 1250 °C for 5 hours, prepared from both unmilled and 20-hour milled 400-mesh Chinese powder. The microstructural differences between the two conditions are striking and demonstrate the critical role of powder preparation in sintering behavior.

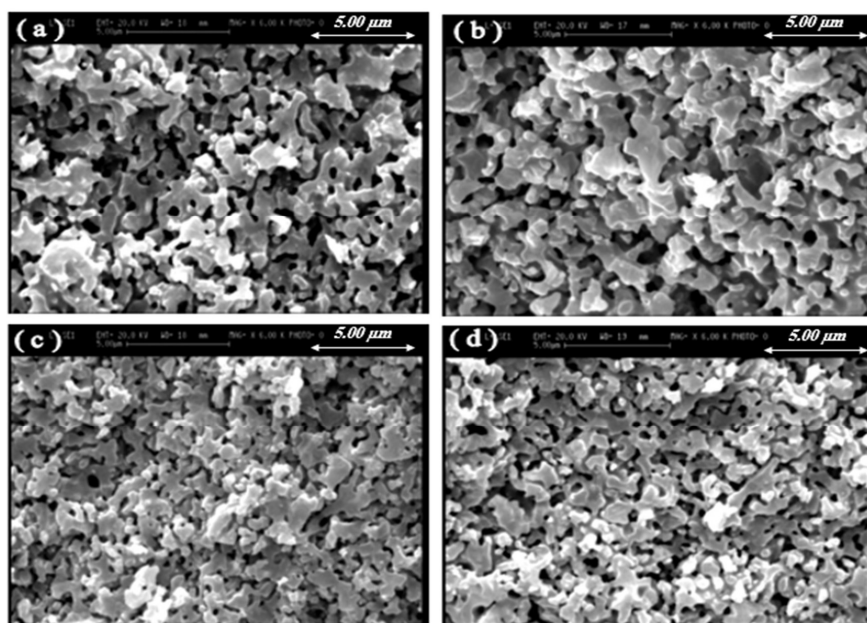


Fig. 6. SEM micrographs (6000× magnification) of pressed samples prepared from 20-hour milled 400-mesh Chinese fused silica powder after sintering for 5 hours at: (a) 1200 °C, (b) 1250 °C, (c) 1275 °C, and (d) 1300 °C.

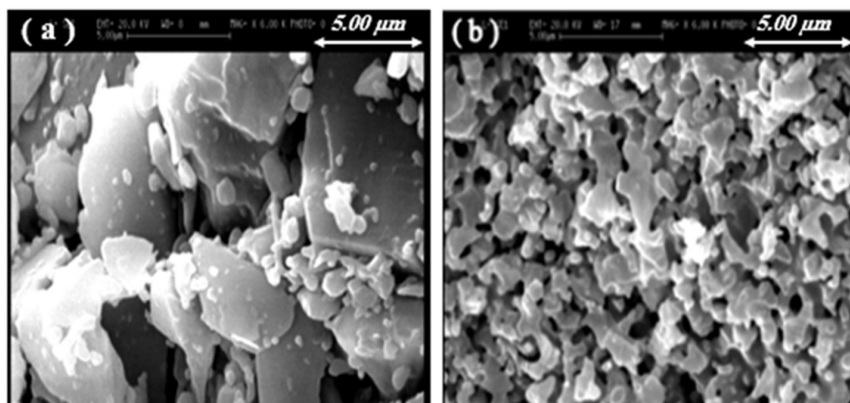


Fig. 7. SEM images (6000×) of fused silica sintered at 1250 °C for 5 hours: (a) unmilled powder and (b) 20-hour milled powder.

Fig. 7 demonstrates the critical importance of powder milling for achieving optimal sintered microstructures in fused silica ceramics. The 20-hour milled powder produces a homogeneous microstructure with uniform grain size ($2.1 \pm 0.3 \mu\text{m}$) and minimal porosity ($< 10\%$), compared to the irregular structure (1-7 μm grains, 15-20% porosity) of unmilled samples. This improvement results from three key effects of milling: (1) breakdown of agglomerates for better powder packing, (2) increased surface area enhancing diffusion, and (3) prevention of abnormal grain growth.

3.2. Effect of boron oxide addition on fused silica sintering behavior

To investigate the effect of boron oxide on the sintering conditions of pressed samples made from 400-mesh Chinese fused silica powder, B_2O_3 was added in amounts of 0.5, 1.5, and 3 wt.%. The mixture was homogenized using jar milling for 24 hours with a ball-to-powder ratio of 10:1. The mixed powders pressed into samples using a hydraulic press. The sintering process was conducted at 1250 °C for 5 hours. The results of this experiment are presented in Fig. 8.

The results demonstrate a critical optimal level for B_2O_3 addition near 0.5 wt%, where it acts as an effective sintering aid by creating an active liquid phase that enhances densification. However, beyond this optimal amount, further addition negatively impacts sintering. Although B_2O_3 is a classic network former known to increase chemical stability and inhibit devitrification in melt-forming processes, its role in the solid-state

sintering of pure silica powder is distinct. Our results show that even small additions lower the viscosity of the glassy phase and significantly reduce the activation energy for cristobalite nucleation. This promotes premature surface crystallization at temperatures below 1200 °C, acting as a catalyst rather than an inhibitor in this specific context. This early phase transformation, coupled with the formation of an excess boron-rich glassy phase at grain boundaries, creates diffusion barriers that hinder atomic mobility and impede uniform densification. As corroborated by the figure, the relative density peaks at 0.5 wt.% before progressively declining due to this combination of premature crystallization and restricted diffusion. This behavior is consistent with previous studies on sintering [22, 23], making B_2O_3 an unsuitable aid beyond trace amounts for applications requiring high-density fused silica components.

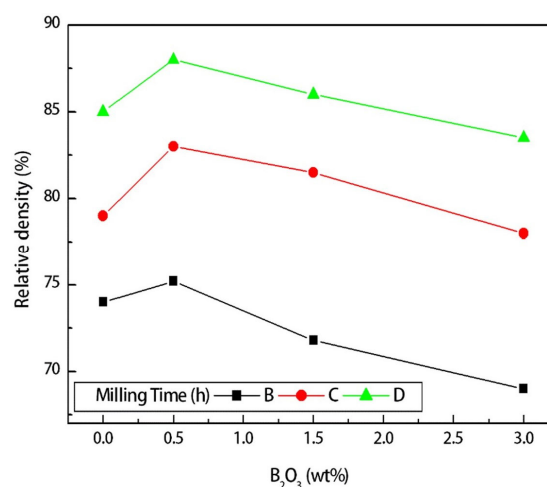


Fig. 8. Density variation of sintered samples prepared from 400-mesh Chinese fused silica powder with different B_2O_3 additions (0-3 wt.%), sintered at 1250 °C for 5 hours.

3.3. Investigating the effects of blending ratios between unmilled and 20-hour milled 400-mesh fused silica powder

Fig. 9 presents the relative density of sintered samples prepared from blended powders consisting of unmilled and 20-hour milled 400-mesh Chinese fused silica, sintered at 1250 °C for 5 hours, while Fig. 10 shows the corresponding SEM micrographs. The results demonstrate that blending the powders in different ratios significantly affects the final density and microstructure. The experimental results indicate that blending 70% 20-hour milled powder with 30% unmilled powder produces optimal sintering characteristics for the 400-mesh fused silica system. This specific ratio achieves near-theoretical density ($\sim 94\%$) while offering significant processing advantages over fully milled powder. The success of this formulation stems from its balanced microstructure, the 70% milled component provides sufficient active grain boundaries to enhance diffusion kinetics, while the 30% unmilled fraction maintains adequate particle packing density and flow characteristics. Microstructural analysis confirms this blend yields uniform grain growth ($1.5 \pm 0.3 \mu\text{m}$) with minimal porosity ($< 6\%$), avoiding both the incomplete densification of unmilled powder and the abnormal grain growth tendencies of fully milled material. From a manufacturing perspective, this optimized blend reduces energy consumption by approximately 30% compared to fully milled powder while simultaneously improving powder handling during pressing operations. The 70/30

ratio represents an effective compromise between sintering performance and practical processing considerations, making it particularly valuable for industrial applications where both product quality and production efficiency are critical. These findings indicate that strategic powder blending can optimize sintered density while reducing milling time and energy consumption, with the 70% milled blend offering the best balance between processing efficiency and final product quality. The microstructural evolution correlates directly with the density measurements, confirming that controlled blending effectively minimizes porosity while maintaining microstructural homogeneity.

3.4. Investigation of sintering time effects

Based on the results reported in this study, to examine the effect of sintering time on pressed samples, a powder blend consisting of 70% 20-hour milled powder and 30% unmilled 400-mesh Chinese fused silica was used. The optimal sintering temperature, as determined by the study's findings, was set at 1250 °C. To evaluate the influence of sintering time, durations of 1 h, 3 h, 5 h, 10 h, and 15 h were investigated. Fig. 11 presents the density variation of samples sintered at different holding times (1-15 hours) at 1250 °C, while Fig. 12 shows the corresponding SEM micrographs of the sintered microstructures.

The study reveals a clear progression in densification behavior with sintering time for the 70/30 blended fused silica composition at 1250 °C.

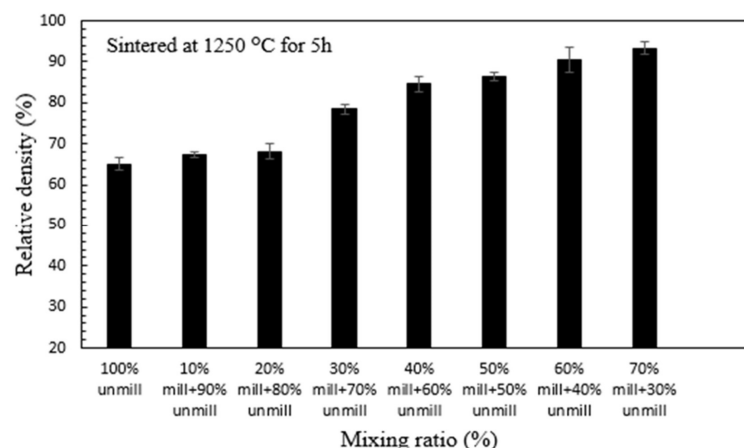


Fig. 9. Relative density of samples sintered at 1250 °C for 5 hours, prepared from blended powders of unmilled and 20-hour milled 400-mesh Chinese fused silica.

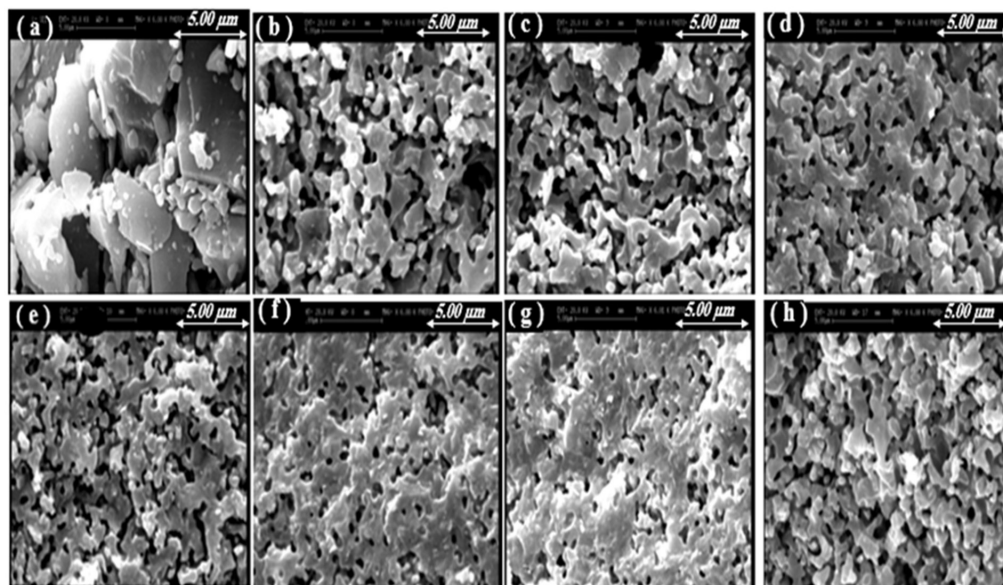


Fig. 10. SEM micrographs (6000 \times) of samples sintered at 1250 $^{\circ}$ C for 5 hours with varying ratios of unmilled to 20-hour milled 400-mesh Chinese fused silica powder: (a) 100% unmilled, (b) 80/20, (c) 70/30, (d) 60/40, (e) 50/50, (f) 40/60, (g) 30/70, and (h) 100% milled.

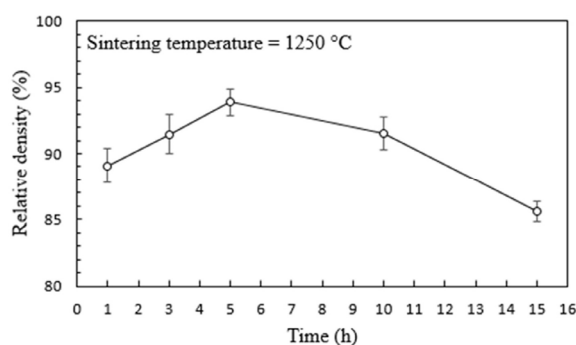


Fig. 11. Density variation of samples (prepared from 70% 20-hour milled + 30% unmilled 400-mesh Chinese fused silica) sintered at 1250 $^{\circ}$ C for different holding times (1-15 hours).

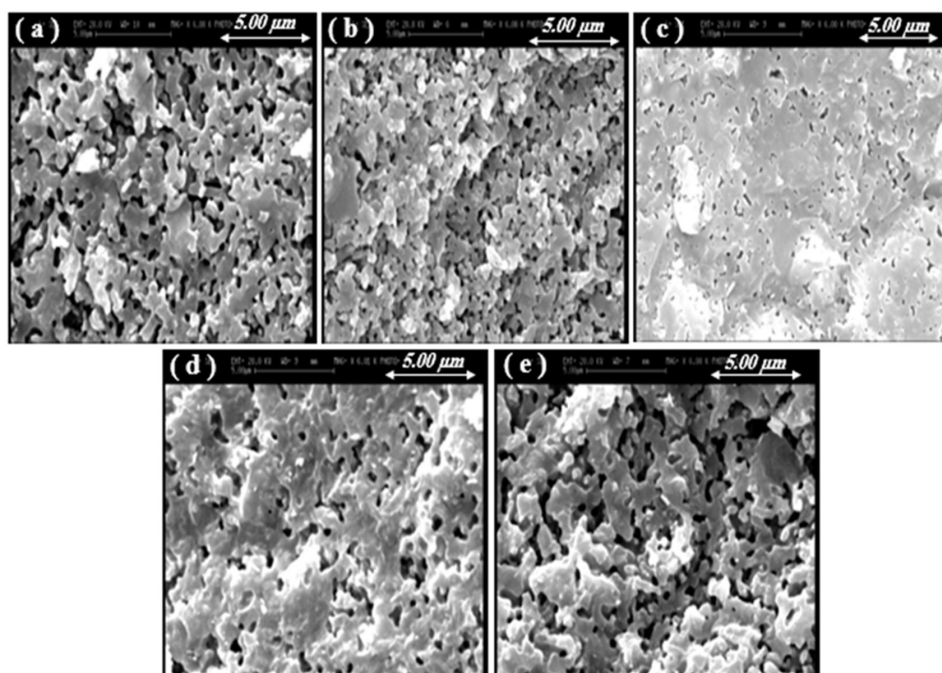


Fig. 12. SEM micrographs (6000 \times magnification) of samples prepared from 70% milled + 30% unmilled powder, sintered at 1250 $^{\circ}$ C for: (a) 1 h, (b) 3 h, (c) 5 h, (d) 10 h, and (e) 15 h.

During the first 5 hours, density increases steadily from 88 to 94% theoretical as grain boundary diffusion dominates, effectively eliminating porosity and developing a uniform microstructure with 1-2 μm grains. This optimal window balances complete densification with energy efficiency. Beyond 5 hours, detrimental effects become pronounced, density gradually decreases to around 91% at 10 hours and then decrease rapidly to around 85 % at 15 hours due to accelerated crystallization [5], causing abnormal grain growth (3-5 μm), pore reappearance, and microcracking from thermal expansion mismatches.

These findings demonstrate that while extended sintering initially improves density, the inevitable cristobalite formation ultimately degrades the microstructure. The 5-hour condition emerges as the practical optimum, achieving near-full density while avoiding crystallization-related defects that compromise longer sintered samples. This balance is particularly crucial given fused silica's strong tendency for surface crystallization above 1200 °C [5], making precise time control essential for quality manufacturing.

4. Conclusions

This study systematically investigated the processing and sintering behavior of 400-mesh Chinese fused silica powder, focusing on optimizing milling, blending, and sintering parameters to achieve high-density ceramic components. The key findings demonstrate that 20-hour attrition milling significantly improves particle size uniformity (reducing average size to $\sim 5 \mu\text{m}$) and enhances sinterability by creating a more homogeneous microstructure. However, milling alone introduces challenges, such as increased interparticle friction, which reduces green density during compaction. The 70:30 blend of milled/unmilled powder emerged as the optimal formulation, balancing sinterability (achieving 94% theoretical density) with practical processing advantages, including $\sim 30\%$ lower milling energy consumption and improved powder flow. Sintering at 1250 °C for 5 hours was identified as the most effective thermal profile, maximizing densification while avoiding detrimental cristobalite formation and grain

growth observed at longer durations ($> 10 \text{ h}$). The addition of B_2O_3 was found to negatively impact densification due to premature crystallization and diffusion barriers, making it unsuitable as a sintering aid for high-density fused silica. Instead, strategic powder blending proved more effective for microstructural control. While the microstructural evolution and densification kinetics provide strong indirect evidence, future work employing X-ray diffraction (XRD) analysis would be valuable for quantitatively confirming cristobalite content and providing a more detailed analysis of the crystallization behavior under these optimized sintering conditions.

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Authors' contributions

M. Afgarazordeh: Methodology, Data curation, Formal analysis, Visualization, Investigation

M. H. Paydar: Conceptualization, Supervision, Validation, Writing original draft, Writing-review and editing

Conflict of interest

The authors declare no conflict of interest.

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