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Enhanced Densification and Mechanical Performance of Fused Silica via Gel Casting

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ABSTRACT

The sintering behavior, phase evolution, and mechanical properties of fused silica ceramics were systematically investigated for samples fabricated by gel casting and dry pressing. Gel-cast samples were prepared using an optimized aqueous formulation containing 58 wt.% solids, whereas dry-pressed compacts were produced at 100 MPa. All specimens were sintered in air between 1100 °C and 1300 °C for 5 h. Dilatometric analysis revealed distinct densification characteristics for the two forming methods. Despite their lower initial green density (~30–33% of theoretical), gel-cast bodies exhibited faster and more complete shrinkage than dry-pressed samples (~57–60% of theoretical), achieving near-full densification above 1150 °C. This enhanced sintering efficiency is attributed to the superior microstructural homogeneity of gel-cast green bodies, whose fine and interconnected pore network provides higher capillary driving forces and more efficient mass transport during viscous sintering. In contrast, heterogeneous particle packing in dry-pressed compacts produced isolated pores that impeded densification. Flexural strength increased with sintering temperature but decreased with higher polymer content due to residual porosity formed during burnout. The maximum strength (~85 MPa) was observed for gel-cast specimens sintered at 1250 °C, while a subsequent drop in 1300 °C corresponded to extensive cristobalite crystallization, as confirmed by XRD and SEM. Crystallization induced microcracking and limited further densification. These results demonstrate that microstructural uniformity is more critical than initial green density for achieving dense, high-strength fused silica. Gel casting thus represents a superior forming technique for producing defect-free amorphous silica ceramics below the cristobalite formation threshold.

1. Introduction

Fused silica, a high-purity amorphous form of silicon dioxide (SiO_2), is one of the most critical materials in modern engineering applications owing to its exceptional thermal stability, low coefficient of thermal expansion, optical transparency, and chemical inertness [1–3]. These attributes make it indispensable in semiconductor lithography, precision optics, aerospace thermal protection systems, and advanced refractory components [4, 5]. Nevertheless, fabricating dense and mechanically reliable fused-silica components remains

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challenging because the material exhibits extremely high viscosity and limited atomic mobility during sintering [6, 7]. As a result, densification typically requires high temperatures above 1300 °C and long dwell times, which not only increase energy consumption but also promote undesirable crystallization into cristobalite, leading to microcracking and strength degradation [8–10]. Developing forming and sintering routes that accelerate densification while preserving the amorphous structure is therefore of continuing interest.

Among the various shaping routes available for oxide ceramics, gel casting has emerged as a particularly attractive approach [11–14]. It involves dispersing ceramic particles in a monomer solution that polymerizes in situ to form a rigid, uniform gel network. The technique provides near-net-shape capability, excellent particle homogeneity, and low defect concentration, giving it clear advantages over conventional dry pressing or slip casting [15, 16]. By contrast, dry pressing, though industrially mature, often results in anisotropic compaction and local density gradients that translate into non-uniform sintering behavior and residual porosity in the final body [17, 18]. Comparing these two forming methods—gel casting and dry pressing—offers valuable insight into how initial green-body microstructure governs densification kinetics and the ultimate mechanical performance of amorphous silica ceramics.

The sintering of fused silica proceeds predominantly by viscous-flow densification rather than solid-state diffusion as in crystalline ceramics [8, 19]. The process involves rearrangement and coalescence of glassy particles under capillary forces generated by surface tension. Because of silica's high viscosity, densification is sluggish and easily interrupted by crystallization to cristobalite, a high-temperature polymorph of SiO_2 [9–12]. Cristobalite formation is particularly detrimental because its reversible α–β phase transformation near 270 °C generates about 2.5 % volume change, producing internal stresses and microcracks upon cooling [10, 12]. Understanding and controlling this balance between densification and crystallization are essential for optimizing the microstructure and mechanical integrity of fused-silica ceramics.

Several strategies have been proposed to enhance silica sintering—such as particle size reduction, the use of sintering additives, or careful adjustment of heating profiles—but these approaches can introduce impurities or compromise optical properties [6, 8]. A more intrinsic and sustainable route is to tailor the green microstructure through the forming process itself. The initial packing uniformity determines pore size distribution and inter-particle contact, which in turn control the driving force for viscous flow and the overall densification kinetics [17, 19]. Studies have shown that microstructural homogeneity plays a more decisive role in achieving full density than initial compact density alone [1, 2, 11, 17]. Consequently, understanding the relationship between forming method, sintering trajectory, and resulting properties is fundamental for process design.

The mechanical behavior of sintered fused silica is strongly governed by residual porosity and crystallization. In gel casting, the polymer concentration directly affects both green strength and burnout behavior: higher polymer content improves handling but leaves more volatile residues, increasing pore formation and decreasing final density and strength [15, 16]. Optimizing polymer content is therefore crucial for balancing processability and mechanical performance. Prior work on alumina, zirconia, and silicon-nitride ceramics has demonstrated that optimized gel-casting formulations yield highly uniform microstructures and superior mechanical properties [11, 14, 20]; however, systematic investigations of this effect in amorphous fused silica remain scarce.

The present study aims to provide a comprehensive assessment of the sintering behavior and mechanical performance of fused-silica ceramics prepared by gel casting and dry pressing. Using an optimized gel formulation from previous work, we evaluate how forming technique, sintering temperature, and polymer content affect densification kinetics, microstructural evolution, and cristobalite formation. Dilatometry, X-ray diffraction (XRD), and scanning electron microscopy (SEM) are employed to elucidate how the green-body microstructure influences viscous sintering and crystallization. Flexural-strength testing establishes the link between microstructure and mechanical integrity.

Through this approach, the work seeks to clarify the fundamental processing–microstructure–property relationships governing amorphous-silica ceramics. The findings demonstrate that microstructural homogeneity achieved via gel casting can surpass the densification efficiency of dry pressing, even when the latter yields higher initial green density. The study thus highlights gel casting as a promising route for producing dense, high-strength, and cristobalite-free fused-silica components for advanced optical and structural applications [1–20].

2. Experimental Procedure

2.1. Sample fabrication

The starting fused silica powder was a commercially available material with a nominal submicron particle size, as specified by the supplier (China). The same powder batch was used for all gel-cast and dry-pressed samples to ensure a consistent comparison. Detailed information on the particle size and size distribution of the silica powder has been reported in our previous study [21]. Gel-cast samples were prepared using an optimized formulation established in our earlier study [22], with a solid loading of 58 wt.%. The solid loading was selected because it provides a balance between slurry castability, sufficient green strength, and defect-free gelation, as determined from preliminary optimization studies. For comparison, samples were also prepared by uniaxial dry pressing at a compaction pressure of 100 MPa, as it represents a commonly used industrial pressure for dry pressing of silica-based ceramics, providing sufficient green strength without inducing excessive density gradients or particle fracture.

2.2. Sintering protocol

All samples were subjected to a controlled burnout cycle (5 °C/min to 300 °C, followed by 1 °C/min to 600 °C with a 1 h dwell) to remove organics. The burnout schedule was designed based on established gel-casting literature and our prior work to ensure complete removal of organics [11, 22]. Sintering was performed in an electric furnace at temperatures of 1100, 1150, 1200, 1250, and 1300 °C for 5 h with a heating rate of 5 °C/min.

2.3. Characterization methods

- **Density measurement:**

Green density was calculated from the measured mass and geometric volume of dried green bodies prior to burnout. The density of sintered specimens was measured by the Archimedes method. Theoretical density was taken as that of amorphous fused silica, and relative densities are reported as percentages of this value.

- **Dilatometric analysis:**

Sintering shrinkage was monitored using a dilatometer. Comparative tests were performed on specimens fabricated by both dry pressing and gel casting to evaluate differences in densification behavior.

- **Mechanical testing:**

The flexural strength was evaluated using a ring-on-ring test according to ASTM C1499-2003. To ensure statistical reliability, three specimens were tested for each condition, and average values were reported.

- **Phase analysis:**

X-ray diffraction (XRD) analysis was conducted to investigate phase evolution, particularly the crystallization of cristobalite during sintering.

- **Microstructural analysis:**

Fractured surfaces of sintered samples were examined using scanning electron microscopy (SEM).

3. Results and Discussion

The transition from a porous green body to a dense, high-performance ceramic component is governed by the complex interplay of the initial microstructure and thermal history. The results of this study reveal that the shaping method—gel casting versus dry pressing—imprints a distinct microstructural signature that dictates the entire sintering trajectory and ultimately determines the final phase composition and mechanical properties of fused silica.

3.1. The genesis of sintering behavior: green microstructure dictates densification kinetics

The most critical finding of this study is the superior sintering performance and final density achieved by gel-cast samples, despite their significantly lower initial green density (~30-33% of theoretical density) compared to dry-pressed samples (~57-60% of theoretical density). This apparent paradox is resolved by analyzing the fundamental differences in particle packing and pore structure inherent to each forming method.

Dry pressing, while effective in achieving high initial density, often produces a heterogeneous microstructure. The application of uniaxial pressure can induce density gradients, particle alignment, and the formation of hard agglomerates. These inhomogeneities create a network of larger, irregularly shaped pores and regions of varying sinter activity. During sintering, denser regions sinter first, constraining less dense areas and leading to the formation of isolated, stable pores that are difficult to eliminate. This is clearly evidenced by the dilatometry data (Fig. 1(b)), which shows that the dry-pressed sample undergoes a more prolonged densification period (approximately 340 minutes, compared to ~220 min) and achieve a lower total shrinkage.

In contrast, the gel-casting process produces a highly uniform, colloidal-based particle packing. The gel network immobilizes the particles in a homogeneous state, resulting in a green body characterized by a fine, uniform, and well-interconnected pore structure. This "ideal" microstructure provides two major advantages for sintering:

Enhanced driving force: The finer pore size in the gel-cast body corresponds to a higher curvature and thus a significantly larger sintering stress (or driving force), as described by the Laplace equation ($\Delta P = \gamma/\kappa$).

Efficient mass transport: The uniform, interconnected pore channels provide short-circuit paths for mass transport via surface diffusion and viscous flow, which are the dominant densification mechanisms during the sintering of amorphous silica.

As shown in Fig. 1(a), the gel-cast body exhibits faster and more complete shrinkage, achieving near-full densification in a shorter time. Ultimately, it surpasses the density of the dry-pressed body above approximately 1150 °C (Fig. 2). These results demonstrate that, for fused silica, microstructural homogeneity plays a more critical role than initial green density in achieving ultimate densification.

These findings align with established literature on oxide ceramics, which emphasizes the critical role of homogeneous microstructures in enhancing densification [3, 23]. From a processing standpoint, gel casting offers clear advantages for fabricating complex-shaped fused-

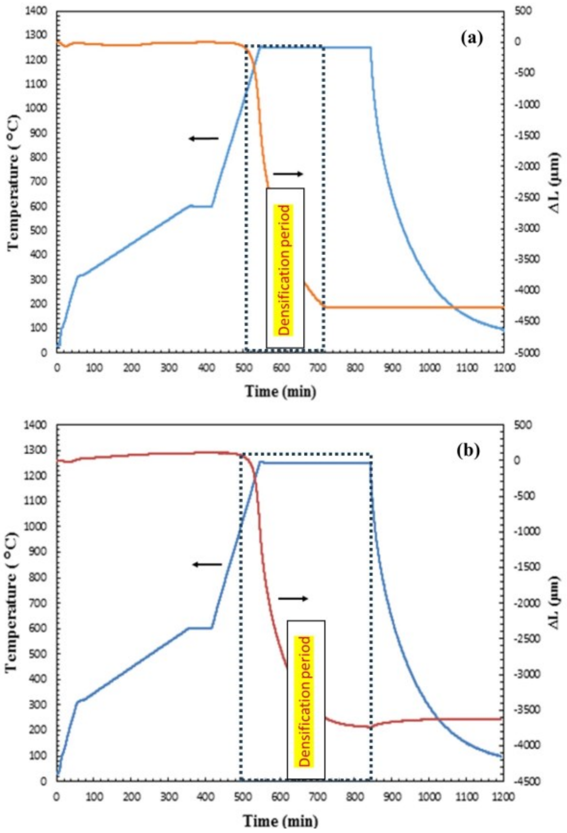

Fig. 1. Dilatometric analysis of (a) the gel-cast and (b) the dry-pressed samples sintered at 1250 °C for 5 hours.

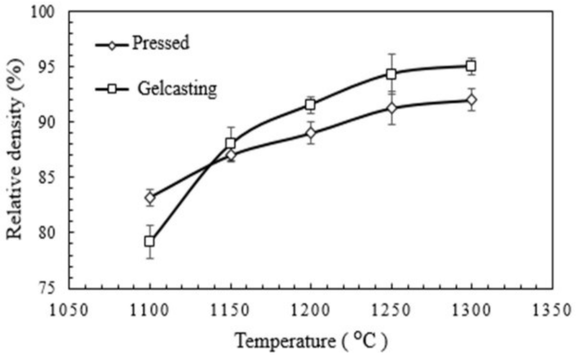

Fig. 2. Effect of the forming method on changes in relative density as a function of sintering temperature, with a holding time of 5 hours.

silica components, where uniformity and defect-free sintering are essential. Although dry pressing provides higher green strength, it is limited in producing intricate geometries and is prone to inhomogeneous densification. Gel casting overcomes these limitations by combining superior shape flexibility with enhanced sintering kinetics, ultimately yielding parts with higher final density and improved mechanical performance. While densification occurs primarily in the amorphous state, the slight increase in bulk density observed at 1300 °C may partly reflect the formation of cristobalite. However, this is accompanied by significant strength degradation and does not represent beneficial densification.

3.2. Effect of polymer content on flexural strength

The results presented in Fig. 3 show that polymer content has a significant effect on the flexural strength of sintered samples. Higher polymer percentage in gel-cast bodies improve green strength but also introduce a larger volume of volatile materials. During sintering, the removal of these volatiles increases porosity, reducing the final mechanical strength. Consequently, at a given sintering temperature, lower polymer content yields higher flexural strength, provided that the polymer is sufficient to ensure successful gelation and avoid casting failure. The observed trend of decreasing flexural strength with increasing polymer content is consistent across all samples, confirming the importance of optimizing polymer concentration for mechanical performance.

Increasing the sintering temperature from 1200 °C to 1250 °C resulted in a general improvement in the final strength, independent of polymer content. This enhancement can be attributed to more efficient sintering at higher temperatures, which promotes better pore elimination, more complete densification, and stronger diffusion bonding (sintering neck growth), thereby resulting in higher flexural strength.

3.3. Microstructural evolution: a window into sintering mechanisms

The SEM micrographs (Fig. 4) provide a visual narrative of the sintering process. At 1100 °C (Fig. 4(a)), the microstructure is largely particulate, with distinct particles and high porosity. As the temperature increases to 1200 °C and 1250 °C (Figs. 4(c, d)), clear evidence of neck growth, particle coalescence, and significant reduction in pore volume and size is observed. The structure gradually evolves into a dense, continuous solid phase with isolated, spherical pores—a characteristic of the final stage. This progression is driven primarily by viscous flow, where amorphous silica behaves like a supercooled liquid, enabling atoms or structural units to rearrange to minimize surface energy.

The observed increase in flexural strength with sintering temperature up to 1250 °C (Fig. 5) follows a classic ceramic sintering trend, where strength (σ) is often related to density (ρ) according to a relationship of the form: $\sigma \propto \exp(-bP)$, where P represents porosity and b is a material-dependent constant. The reduction in porosity and the corresponding increase in interparticle contact area directly enhance mechanical strength [19].

3.4. The Cristobalite conundrum: Crystallization as a performance-limiting phenomenon

The peak in flexural strength at 1250 °C is followed by a dramatic decrease at 1300 °C. This inflection point reflects a fundamental shift in the dominant microstructural mechanism, from densification to crystallization. XRD patterns (Fig. 6) show that samples sintered at ≤1250 °C remain predominantly amorphous, whereas the sample sintered at 1300 °C exhibits almost complete crystallization to cristobalite, as identified according to JCPDS card No. 00-039-1425 (tetragonal structure, space group $P4_12_12$).

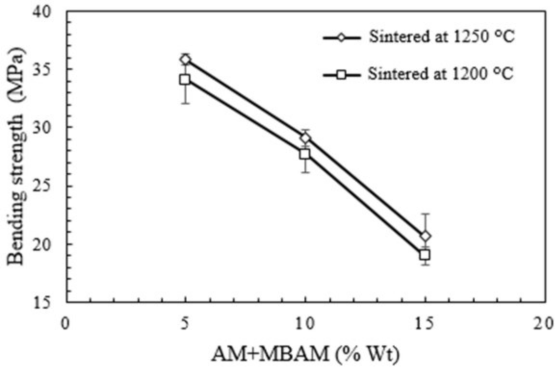

Fig. 3. Effect of polymer content on the flexural strength of samples sintered at 1200 and 1250 °C with a holding time of 5 hours.

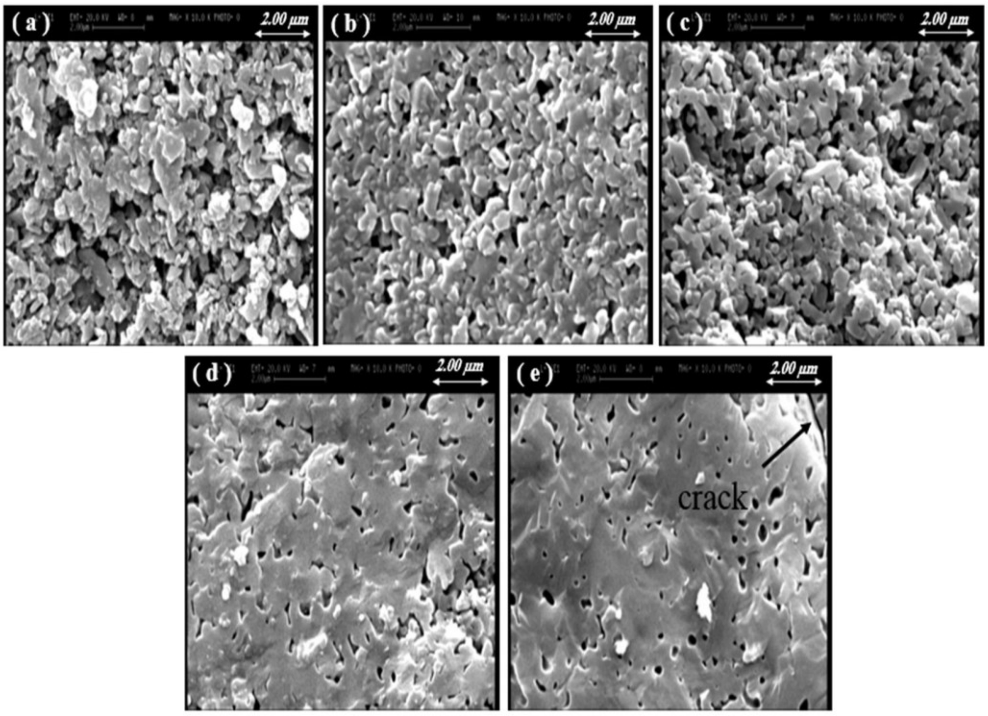

Fig. 4. SEM images of the gel-cast samples at 10,000x magnification, sintered at temperatures of (a) 1100, (b) 1150, (c) 1200, (d) 1250, (e) 1300 °C for 5 hours.

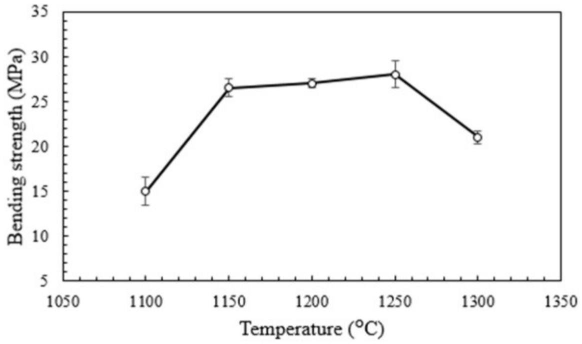

Fig. 5. Relationship between sintering temperature and the strength of gel-cast samples with a sintering time of 5 hours.

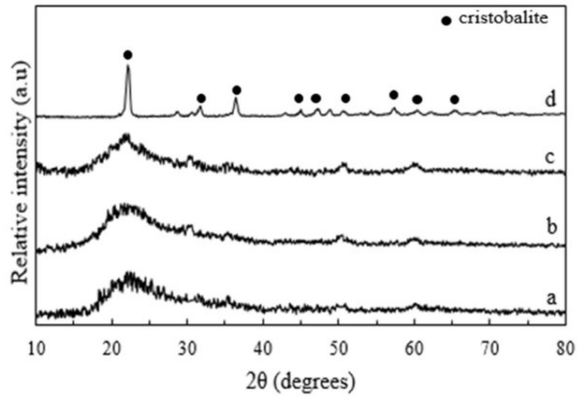

Fig. 6. X-ray diffraction pattern of samples sintered at temperatures of (a) 1150, (b) 1200, (c) 1250, and (d) 1300 °C with a sintering time of 5 hours.

This conclusion is supported by the disappearance of the amorphous halo and the prominence of cristobalite diffraction peaks, consistent with previous reports on silica crystallization under comparable conditions.

The detrimental effect of cristobalite formation on mechanical strength is multifaceted:

Volumetric changes and microcracking: Cristobalite undergoes a reversible α (low-temperature) to β (high-temperature) phase transformation at approximately 270 °C, accompanied by a ~2.5% volume change. During cooling from 1300 °C, the material passes through this transition, generating shear and volumetric strains that create significant localized internal stresses. These stresses readily exceed the local material strength, initiating microcracks that act as critical flaws, drastically reducing flexural strength.

Disruption of sintering: Crystallization also interrupts the last stages of densification. As amorphous silica transforms to crystalline cristobalite, atomic mobility decreases, and the viscous flow mechanism is effectively halted. The newly formed cristobalite grains and their boundaries can pin the microstructure, preventing the elimination of the last remnants of porosity. The effect is evident in the SEM image of the sample sintered at 1300°C (Fig. 4(e)), where the microstructure appears stabilized without further pore removal compared to 1250 °C.

At 1300 °C, the driving force for crystallization overcomes kinetic barriers. Previous densification at lower temperatures reduces the nucleation barrier for cristobalite, while the elevated temperature provides sufficient atomic mobility for rapid crystal growth. Consequently, densification and crystallization occur simultaneously, but crystallization ultimately dominates, terminating densification and degrading mechanical integrity.

4. Conclusions

This study systematically examined the influence of forming technique, polymer content, and sintering temperature on fused silica ceramics, focusing on densification behavior, phase evolution, and mechanical performance. The results demonstrate that gel casting offers significant advantages over conventional dry pressing by producing denser and more homogeneous microstructures. Although gel-cast green bodies had lower initial densities (~30–33% of theoretical) compared to dry-pressed compacts (~57–60% of theoretical), their uniform particle packing and interconnected pore network enabled faster and more complete shrinkage during sintering. Above 1150 °C, gel-cast specimens achieved higher relative densities than dry-pressed ones, confirming that microstructural uniformity is more critical than initial green density for promoting densification. Flexural strength was strongly influenced by both polymer content and sintering temperature. Lower polymer concentrations minimized residual porosity after burnout, resulting in higher mechanical strength, whereas excessive polymer content increased pore formation and degraded strength. Optimal properties were observed at 1250 °C, where gel-cast specimens reached a maximum average flexural strength of approximately 85 MPa. Sintering at 1300 °C, however, induced extensive cristobalite crystallization, which caused microcracking through the α–β phase transformation and markedly reduced mechanical strength. Overall, this study highlights two key factors for optimizing fused silica ceramics: controlling microstructural homogeneity and limiting cristobalite formation. Gel casting provides an effective and versatile processing route, suitable for producing dense, high-strength amorphous silica components for advanced optical, electronic, and thermal applications.

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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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