

Investigation of Ceramic Dental Prostheses Based on ZrSiO₄-Glass Composites Fabricated by Indirect Additive Manufacturing

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Abstract: Dental prosthesis and restoration technologies have been developed in the past years. Despite the advantages of additive manufacturing, computer-aided design, and computer-aided manufacturing technologies are still the dominant type of method for fabricating prostheses. Therefore, the main goal of this study is to assess the feasibility of using indirect fused deposition modeling to fabricate dental prosthesis made of ZrSiO₄-glass composites. To achieve this goal, filaments were filled by 90% of ZrSiO₄ and 50 μm glass spheres to fabricate prosthesis. Multivariable approach was applied to evaluate the feasibility of the proposed method. Holding temperature, holding time, heating rate, and cooling rate were considered the control factors, while shrinkage, flexural strength, and process feasibility were the study responses. In addition, the flexural strength of materials was found between 25 and 85 MPa, while shrinkage fluctuated between 10 and 25%.

Keywords: Fused deposition modeling ceramic sintering; Additive manufacturing; Ceramics

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Received: September 29, 2020; **Accepted:** October 20, 2020; **Published Online:** November 26, 2020

Citation: Cunico MWM., 2021, Investigation of Ceramic Dental Prostheses Based on ZrSiO₄-Glass Composites Fabricated by Indirect Additive Manufacturing. *Int J Bioprint*, 7(1):315. <http://doi.org/10.18063/ijb.v7i1.315>

1. Introduction

In the past few years, additive manufacturing (AM) technologies have started playing an important role in several industrial and health-care segments^[1]. In addition, development of dental materials and applications signifies the advent of digital fabrication through which the dental implants are mainly fabricated by computer-aided design and computer-aided manufacturing (CAD/CAM) techniques coupled with three-dimensional (3D) scanning^[2,3].

Table 1 shows the most common techniques that are used in accordance with the type of dental prosthesis. Possibly, CAD/CAM is currently the most used technique in prosthodontics^[4]. Although AM is still used in the fabrication of medical/dental models and temporary dentures, its mechanical strength is usually inferior to that of CAD/CAM technologies and no sufficient clinical records regarding its application are available^[2-5]. In

spite of that, CAD/CAM techniques also show some disadvantages, such as requirement of highly trained professional to operate 5-axis computer numerical control, high cost of raw material, excessive waste generation, high cost of maintenance, and short lifetime of tooling. On the other hand, AM could improve the fabrication of dental prosthesis by increasing automation, flexibility, shape freedom, and fabrication speed. Apart from manufacturing and product development, AM is also applied in different areas, such as aerospace, electronics, medical applications, and tissue engineering^[6-15].

After weighing the pros and cons of either technique, several aspects of AM technologies, such as mechanical strength, type of material, bio-reactivity, and cost, still remain as the challenges awaiting to be addressed^[18]. For that reason, AM technologies are widely used for: provisional crown and bridge restorations, casting patterns, dental models, surgical guide, and splints. On the other hand, further development of AM technologies

Table 1. State of the art of prosthodontical technologies

	Classic dentistry technologies					Additive manufacturing technologies				
	Sintering	slip casting and Casting	Hot-pressed/injection molded ceramics	CAD/CAM ¹	SLM ²	SLS ³	Bindjet direct or infiltrated	Multijet	SLA/DLP ⁴ -direct or wax-like castable	FDM ⁵ /extrusion-based
Applications										
	Crown and bridges	[16]	[16,17]	[3,16-19]	[18,19]	[19]		[18,19]	[18,19]	
	Denture	[16]	[17]	[3,16-19]	[18,19]	[19]				
	Denture holder	[16]	[17]	[3,16-19]	[18,19]					
	Copings	[16]	[17]	[3,16-19]	[18,19]					
	Casting patterns/lost wax	[16]	[16]	[3,18]				[18,19]	[18,19]	
	Provisional temporary crown			[18]		[20]		[2]	[2,18]	
	Dental model			[18]				[2,18,20]	[2,18]	[18]
	Surgical guide			[3,18]				[2,18]	[18]	[18]
	Surgical guide plate			[3,18]		[2]	[2]	[2]	[2]	[18]
	Splints			[18]				[2,18]	[2,18]	[18]
	Prosthetic constructions			[18]						
Materials	All-ceramic	[18]	[18]	[18,20]		[18]				
	Porcelain	[18]	[18]	[18]						[18]
	Y-TZP ⁶	[18]	[18]	[18]						
	Metallic	[18]	[18]	[18,20]	[2,18]					
	Glass-ceramic	[18]	[18]	[18,20]		[18]	[18]	[19,20]		
	Polymer-ceramic composite	[18]	[18]	[16,18,20]		[18]	[18]		[2,3,18,19]	
	Metal-ceramic composite	[18]	[18]	[16,18,20]		[18]				
	Polymeric material			[18]		[18]	[18]	[18,19]	[2,3,18,19]	

1-CAD, Computer-aided design; CAM, Computer-aided manufacturing; 2-SLM, Selective laser melting; 3-Selective laser sintering; 4-SLA, SLA, Stereolithography apparatus/DLP, Digital light projector; 5-FDM, Fused deposition modeling; 6-YTZP, Ytria stabilized zirconia.

is still need warranted to perfect their applications in the fabrication of dental implants, crown, and bridges denture and prosthetic constructions. Therefore, the main goal of this study is to propose and investigate the feasibility of a novel fabrication method based on $ZrSiO_4$ -glass composite in dental prosthesis applications, such as crowns, coupings, bridges, and dentures. The schematic of this method is illustrated in **Figure 1**.

In this method, the collapsible $ZrSiO_4$ mold of negative of crown or bridge was fabricated by AM based on fused filament fabrication (FFF). The negative cavity was filled by lanthanum glass powder and sintered subsequently. In the end, the pieces were heated to debind the negative structure and then create sintered composite pieces.

It is also important to note that several AM techniques, such as inkjet printing and stereolithography, generate objects with higher resolution than manufacturing by FFF. However, these techniques are restricted by photopolymeric resins and slurry-based techniques. For that reason, they are mainly used for casting patterns, lost wax (resin), and investment casting^[19,20]. In addition, in spite of high resolution, these AM techniques (stereolithography apparatus and Inkjet printing) imply on dimensional and geometrical accuracy equivalent to FFF techniques^[21-23]. In fact, the high resolution has been shown to influence the roughness, rather than the dimensional accuracy^[21-23]. Besides low cost, AM based on FFF is able to fabricate objects with high amount of ceramic and metallic in their composition^[24-26]. Thus,

this technique is suitable for fabrication of collapsible molds made of ceramics or metallic material with high temperature resistance.

To evaluate the feasibility of this technique, we applied multivariable approach to investigate the main effect of control factors on responses. Holding temperature, holding time, heating rate, cooling rate, and shrinkage chamber were the control factors while shrinkage, flexural strength, and process feasibility were the study responses. The fabrication parameters, formulation of materials, flexural testing specimen shape, and crown shape were kept constant. This study helped determine whether the proposed method of indirect AM is feasible to fabricate ceramic dental prosthesis.

2. Materials and methods

To investigate the feasibility of the proposed method in addition to the main effect of control factors on responses, we applied a 2^k multivariable methodology (full design with body central point) where holding temperature (T_h), holding time (t_h), heating rate (R_h), and cooling rate (R_c) were the control factors. In addition, we also defined three screening steps in augmented design approach to minimize the holding time and maximize the densification and mechanical properties.

The levels and values of each control factor are presented in **Table 2**. The values of holding temperature are between the activation temperature of glass and $ZrSiO_4$ (700°C) and the melting temperature of glass (1078°C).

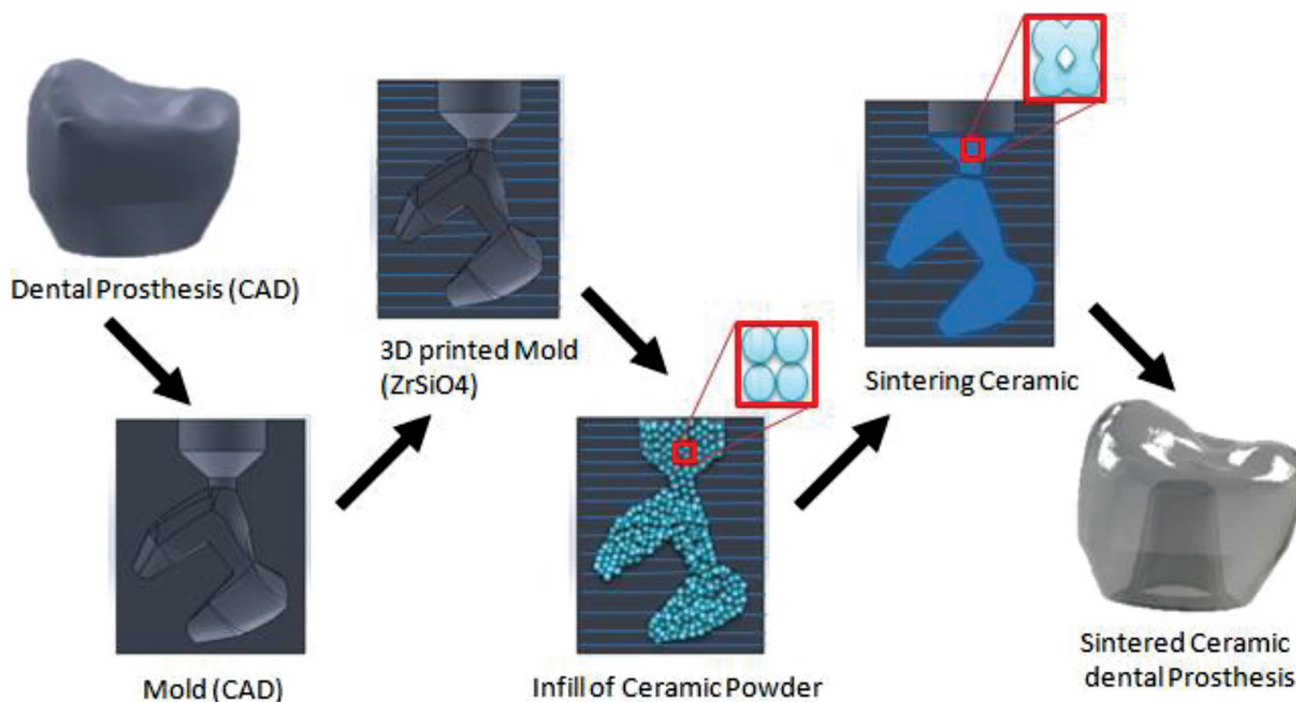


Figure 1. Schematic diagram of the fabrication of ceramic dental prosthesis using indirect additive manufacturing.

This also indicates that the cooling time enhanced two types of heat treatment (quenching for fast cooling and annealing for slow cooling). Therefore, the effect of such treatments on crystallization level, mechanical strength, and geometry distortion can be evidenced. On the other hand, holding time and heating rate are expected to affect the sinterization parameters, such as nucleation, grain growth, and diffusion.

For the sintering process, we used a 2000 W electrical Furnace PID controller with 4 ramps curves and insulation muffle. The main control parameters and schematic sintering temperature curves that we applied in this study are illustrated in **Figure 2**.

The flexural testing was performed in accordance with ISO 6872 in an EMIC DL10000 universal testing machine. We used MATLAB software for the image processing, and the image acquisition was performed using

optical microscope Digital Avangard Optics AN-E500 (AVANGARD 2011), which a magnitude of up to $\times 500$ of amplification. For the gravimetric analysis (drying monitoring), we used a 0.005 g error scale. To identify dimensional distortions of the specimen, we used both 0.05 mm caliper and computational image processing to measure the specimen dimensions. After measuring, we used MATLAB software to perform statistical analysis and evidence the geometrical variation of object external contour. To measure feasibility response, we established a scale from 0 to 1 in which level 1 indicates that the object has no significant distortions and is feasible to be used; level 0.75 exposes minor distortion which corresponds to 5% of distortion; and level 0.5 indicates that the object has 10% of distortion. It is important to note that the measurement of feasibility response was used to analyze non-volumetric distortions and compare CAD models

Table 2. Experiment design

		Level		
Control factors		-1	0	1
1 st step	Heating rate (Hrate) (°C/min)	2	3.5	5
	Holding temperature (Thold) (°C)	700	800	900
	Holding time (thold) (h)	1	2.5	4
	Cooling rate(Crate) (°C/min)	2		30
2 nd step	Holding temperature(Thold) (°C)	700	750	800
	Holding time(thold) (h)	1	1.75	2.5
3 rd step	Holding temperature(Thold) (°C)	700	725	750
	Holding time(thold) (h)	2.5	2.875	3.25

Where: T_{hold} is the holding temperature – period of time where the temperature is kept constant; t_{hold} is the holding time or plato time – temperature which is kept constant during a period of time; H_{rate} is the heating rate of sintering furnace; C_{rate} or k is the cooling rate of object.

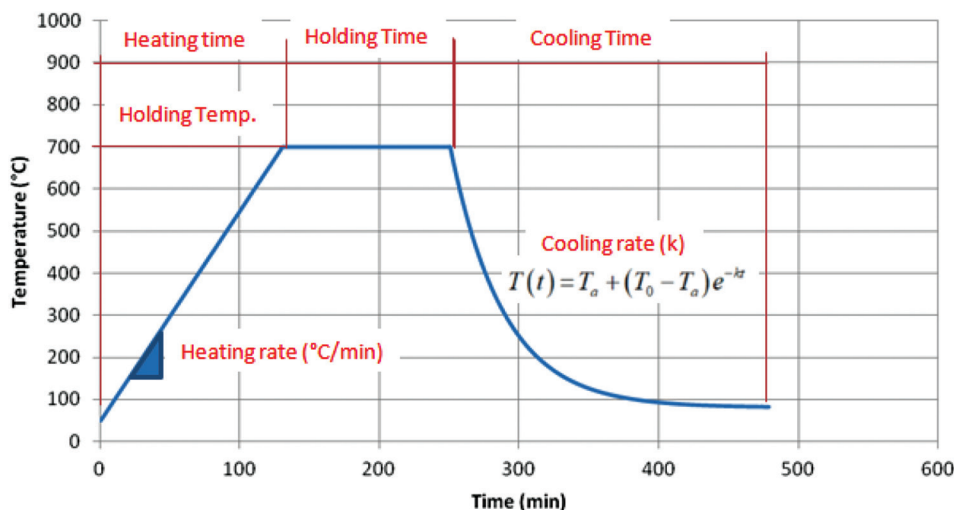


Figure 2. Illustration of sintering temperature curves and control factors: Holding temperature (T_{hold}), holding time (t_{hold}), cooling rate (k), and heating rate (H_{rate}).

adjusted by shrinkage factor, although shrinkage is a sort of volumetric distortion.

For specimen's fabrication, we used a fused deposition modeling process and filament filled by 90% of $ZrSiO_4$. The main process parameters remained constant, where extrusion temperature was 220°C, layer thickness was 0.1 mm, distance between filaments was 0.2 mm, and nozzle diameter was 0.4 mm. In addition, we have also considered no support material and no retract to build the specimens. In all the cases, the extrusion temperature and chamber temperature were also kept constant, while no bed temperature was established. The fabrication environment was also controlled at 25°C (room temperature) and 50% of relative humidity.

The repeatability and error of the AM-generated mold were also identified before performing the study. Ten samples of the mold were fabricated, and their external and internal geometry was measured. The internal geometry was evaluated by transversal cuts, which helped to ensure that the divergence between CAD- and AM-generated molds was up to 0.15 mm besides an error of 2% in the small dimension of mold.

3. Results and discussion

In general, the evaluation of concept feasibility was satisfactory, whereas a feasible process window was identified. From geometric point of view, the crown was obtained in low sintering temperatures because high levels of temperature distorted the geometry due to excessive melting and high shrinkage.

With regard to the main effect of control factors on the feasibility, mechanical strength and shrinkage, holding temperature had the strongest effect on the feasibility and flexural strength in comparison with the other control factors (**Figure 3**). On the other hand, holding time affects shrinkage the most. It shows that holding time and holding temperatures are the most relevant factors for the augmented design. Therefore, the second design of experiments screening increased

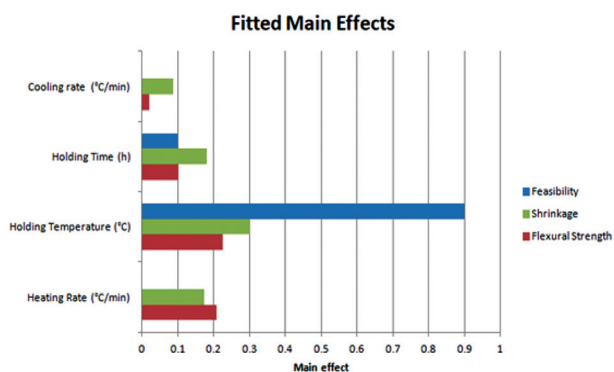


Figure 3. Main effect of control parameter on feasibility, flexural strength, and shrinkage.

the experiment resolution for holding temperature and holding time. The flexural strength was also affected by heating rate, indicating that densification of material might have reduced the strength of material.

In parallel with the feasibility of the proposed concept, high temperature implies on the unfeasibility of concept regardless of time holding, cooling rate, and heating rate (**Figure 4**). In this study, the holding temperature was the most relevant parameter for feasibility, followed by holding time. In contrast, heating and cooling rates were found not to affect the feasibility. From the geometric point of view, our analysis revealed that the shrinkage varied from 13.4% to 27% into the feasible area. The lowest value of shrinkage was found in a condition when the heating rate equals to 5°C/min, holding temperature 700°C, holding time 3.2 h, and cooling rate 30°C/min, whereas the highest value was found in a condition when the heating rate equals to 2°C/min, holding temperature 700°C, holding time 4 h, and cooling rate 2°C/min.

Figure 4 shows the main effect of control parameter shrinkage. The holding temperature was the most relevant factor for the shrinkage, while the heating rate showed the smallest effect among the control parameters. With respect to material mechanical strength, **Figure 4** also presents the main effect of control parameter on flexural strength. The temperature and heating rate were the most relevant factors for mechanical strength, while cooling rate presented the smallest effect among the control parameters. The mean values of flexural strength varied from 25 to 82 MPa, where the lowest values were found in low holding temperatures (700°C) and short holding time (1 h). On the other hand, the highest values were obtained by long hold time (1 h) and 800°C of holding temperature. It is also important to indicate that this process did not evaluate the effects of neither heating treatment nor material. Therefore, further studies are still needed for incorporating the use of stronger materials and heating treatments in this concept.

In addition, **Figure 5** shows a comparison diagram of geometrical concept for feasibility as a function of holding temperature and holding time. In this figure, a feasibility line separates the results which were considered feasible and unfeasible from the geometrical point of view.

In addition, the feasibility was also separated in two areas. High grain growth was obtained at low temperature (700°C) during long holding time (4 h), producing specimen with high densification. On the other hand, low grain growth was obtained at low temperatures (700°C) during short periods of time (1 h), producing specimen with low densification. Of note, the central point of the study indicated a limit of feasibility so that the sintering process can be directly correlated to the absorbed energy as a function of time and temperature. The unfeasible

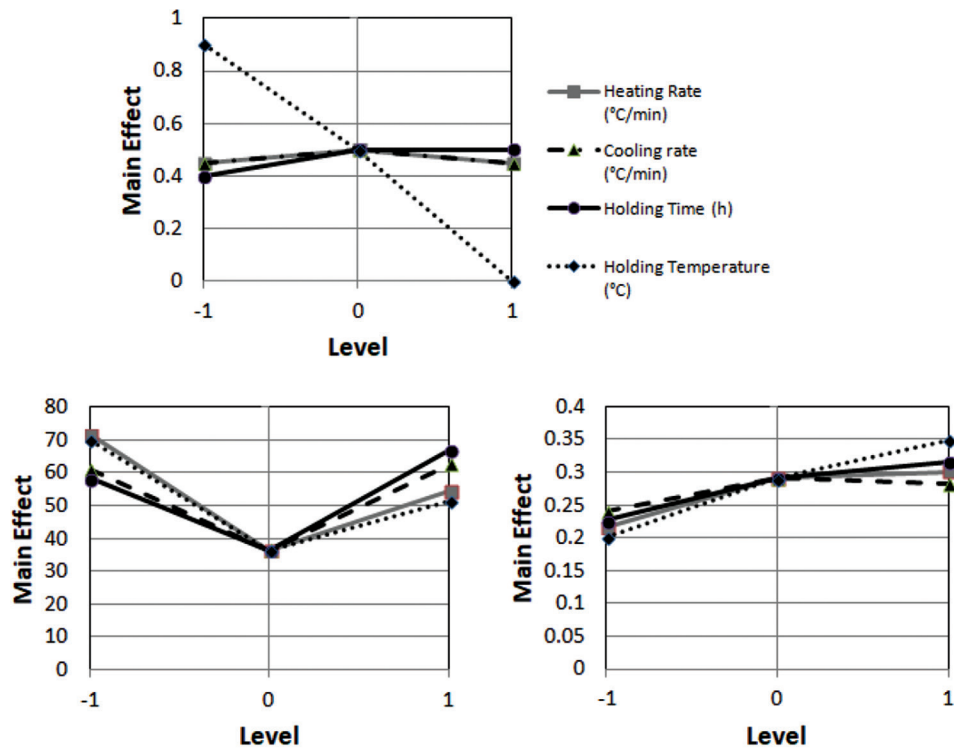


Figure 4. Standardized main effect of control factors on feasibility, flexural strength, and shrinkage.

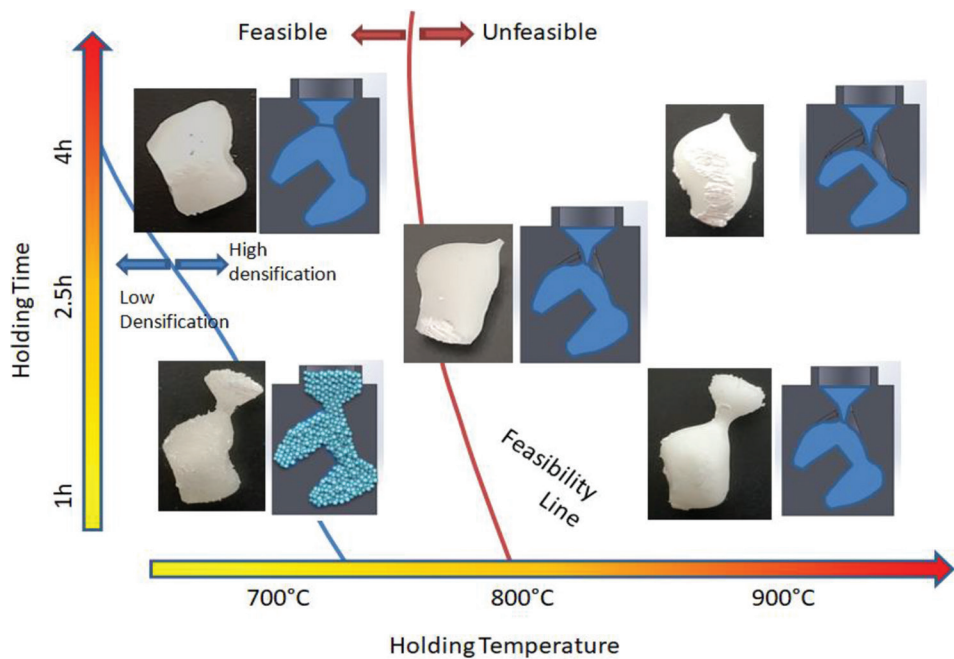


Figure 5. Comparison diagram of proposal feasibility as a function of holding temperature and holding time.

area was highlighted by high densification and excess of deformation due to a decrease in viscosity. In addition, this reduction indicated mold infiltration and subsequent incrustation of collapsed mold on object surface. The materials obtained at high temperature with long holding

time were of high densification and grain size and were bigger than the grain in material produced at low temperatures. This situation is shown in Figure 6, where the comparison of material densification and grain size is presented.

To better understand the behavior of material properties as a function of holding temperature and holding time, **Figure 7** shows the contour diagrams of feasibility, shrinkage, and flexural strength and presents an overlapping diagram indicating the process window from where high values can be obtained.

According to **Figure 7**, high values of feasibility, flexural strength and low values of shrinkage can be obtained with holding time at around 1.5 h and holding temperature at around 750°C. In this case, values of

flexural strength fluctuate around 65 MPa, while the shrinkage values around 13%. The feasibility ratio was considered higher than 0.9. It is important to note that the feasibility level indicates the compatibility between the physical model and the CAD 3D model. Therefore, the distortion between the physical model and the 3D model adjusted with volumetric shrinkage is lower than 10% for the feasibility level which is higher than 0.9. That represents an error up to 0.1 mm in the smallest analyzed dimension of the samples. Another important point on this matter is the potential minimization of shrinkage factor in accordance with the type and formulation of ceramic material and the size of powder grain. Nonetheless, further efforts are still needed to improve material properties and process aspects, such as mechanical strength, biocompatibility, physical, and chemical properties.

Figure 8 shows the comparison in flexural strength between the proposed method and other typical methods that are used for fabricating dental prosthesis. It is important to see that our current results identified values

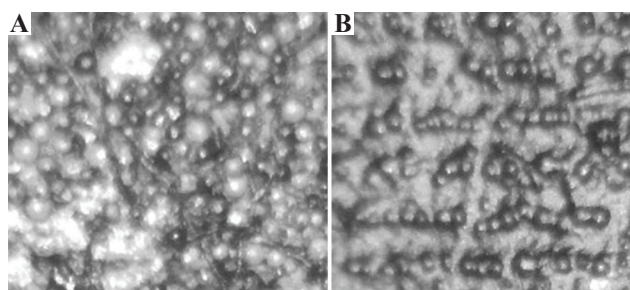


Figure 6. Comparison between low (A) and high (B) densification.

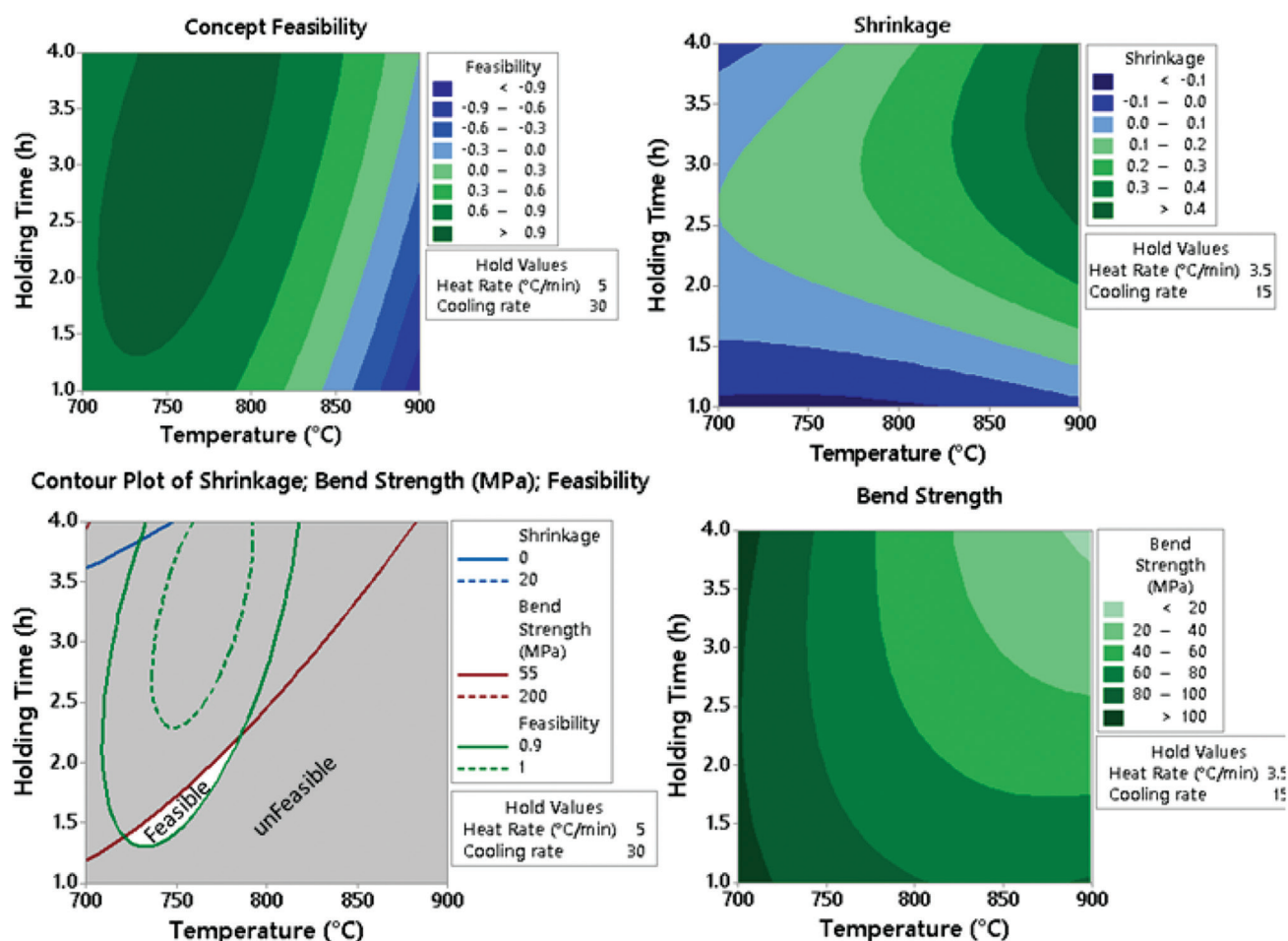


Figure 7. Contour diagrams of flexural strength, feasibility, shrinkage, and the overlapping diagram with combination of high values.

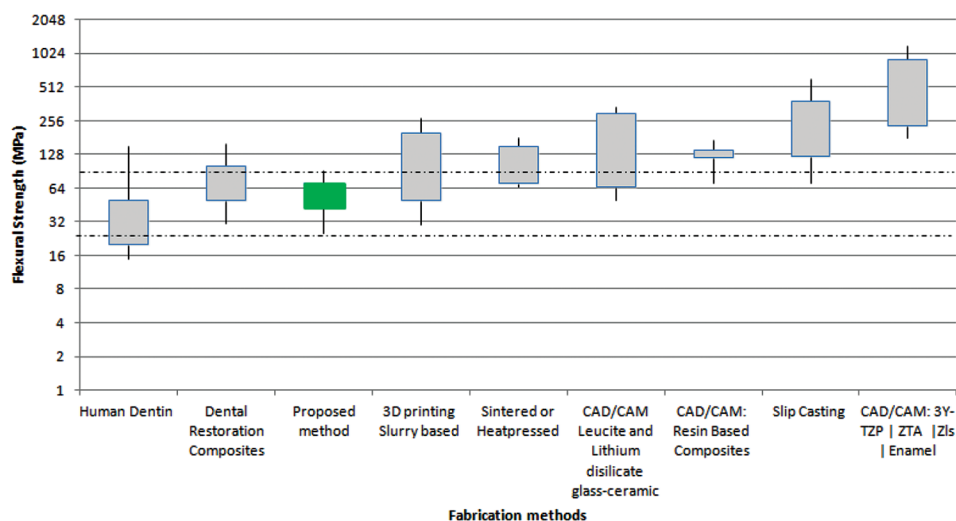


Figure 8. Flexural strength of the proposed method and typical dental prosthesis^[3,17,28-37].



Figure 9. ZrSiO₄-glass composite-based prosthetic crowns fabricated by indirect fused deposition modeling collapsible tool. The crown on the left side is of high grain densification with no finishing and the crown on the right side is of low grain densification with no finishing.

that are comparable to the typical dental applications. Therefore, further studies might increase the flexural strength up to the level of slip casting strength. It is also important to note that the volumetric shrinkage for typical CAD/CAM dental application varies from 5 to 30%^[3,4,17,27]. Therefore, the range of values found in this work (13 – 25%) is compatible with the current state of art, indicating potential application in the field.

This concept has been shown to work and open a new possibility to fabricate glass-ceramic materials by AM technologies using collapsible ceramic mold, as presented in **Figure 9**. In this figure, two prosthetic crowns with no finishing were presented. The left-side crown presents high grain densification while the right-side crown with low grain densification. Both crowns have

no layer marks, as the sintering process merged powder grain and created object superficial tension which inhibits the occurrence of mold layer marks. In addition, it is also known that after the fabrication of the dental prosthesis, the typical dental applications (CAD/CAM, slip casting, *etc.*) are coupled with several stages of finishing and making-up that improve the esthetic appearance of the prosthesis so that they mimic the original human dent^[38]. Therefore, the geometrical characteristics, mechanical properties, and biocompatibility are the most important aspects to be considered in this type of application. Despite the preliminary results on the feasibility of the proposed method, further studies are still needed to improve mechanical strength, diversify the glass-ceramic materials, applications, and biocompatibility.

4. Conclusions

Collectively, this study assessed the feasibility of the glass-ceramic fabrication based on collapsible AM mold of ZrSiO₄. The working proof generates new perspectives to AM in dentistry, ceramics, and medical applications, whereas collapsible AM molds, such as we method used in this study, can support fabrication up to 2300°C.

This study also identified that the holding temperature is the factor that mostly influences the feasibility, strength, and shrinkage, followed by holding time which directly affects the material densification and grain growth. Besides, long time periods and high temperatures increase the densification and soften the material, leading to distorted geometry, and making the method unfeasible. On the other hand, flexural strength fluctuates between 25 and 82 MPa and can be highly affected by heating rate and cooling rate. Further studies are required to investigate the role of heating treatments and increase mechanical and geometrical properties, as

well as incorporate new materials and applications for screening.

Acknowledgment

We would like to thank Concep3D R&D and FAE Centro Universitário for their support and infrastructure.

Conflicts of interest

No conflicts of interest were reported by the authors.

Author contributions

All the stages of this study were performed only by the author.

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