Global Journal of Medical Research: J
Dentistry & Otolaryngology
Volume 20 Issue 7 Version 1.0 Year 2020
Type: Double Blind Peer Reviewed International Research Journal
Publisher: Global Journals Inc. (USA)
Online ISSN: 2249-4618 & Print ISSN: 0975-5888

Investigation of Ceramic Dental Prostheses based on Zrsio4 - Glass Composites Fabricated by Indirect Additive Manufacturing

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Abstract- Technologies for dental prosthesis and restoration have been developed during the past years. Despite the advantages of additive manufacturing, CAD/CAM technologies lead prostheses fabrication. Therefore, the main goal of this work is to investigate the fabrication of dental prosthesis based on $ZrSiO_4$-glass composites applying the indirect fused deposition modelling. In order to achieve this goal, we used filaments filled by 90% of $ZrSiO_4$ and 50μm glass spheres in order to fabricate prosthesis. We also applied multivariable approach to scan the feasibility of the proposed process. Holding temperature, holding time, heating rate and cooling rate were considered the control factors while shrinkage, flexural strength, process feasibility were the study responses. It was possible to see the proposal feasibility for holding temperatures between 700 and 800°C and holding time between 1 and 4 hours. Additionally, the materials flexural strength was found between 25 and 85MPa, while shrinkage fluctuated between 10 and 25%.

Keywords: FDM ceramic sintering, additive manufacturing, ceramics.

GJMR-J Classification: NLMC Code: WU 500

INVESTIGATIONOFCERAMICDENTALPROSTHESESBASEDONZRSIO4GLASSCOMPOSITESFABRICATEDBYINDIRECTADDITIVEMANUFACTURING

Strictly as per the compliance and regulations of:

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I. Introduction

During the last years, additive manufacturing (AM) technologies started playing an important role in several segments (Cunico and de Carvalho, 2016). In addition, development of dental materials and applications points to digital fabrication whereas dental implants are majorly fabricated by CAD/CAM techniques in collaboration with 3D scanning (Sulaiman, 2020, Li et al., 2014).

Table 1 shows the most common techniques which are used in accordance with the type of dental prosthesis. Therefore, it is possible to see that CAD/CAM is currently the most used technique in prosthodontics (Karthick et al., 2019). On the other hand, additive manufacturing is still used for medical/dental models and temporary dentures, whereas there are not long clinical records and mechanical strength is usually lower than CAD/CAM technologies (Sulaiman, 2020, Li et al., 2014, Gali and Sirsi, 2015, Karthick et al., 2019).

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Table 1: Mapping of Prosthodontical technologies

		Classic Dentistry Technologies					Additive manufacturing Technologies					
		Sintering	slip casting and Casting	Hot-pressed/ Injection Molded Ceramics	UV resin molded	CAD/CAM	SLM	SLS	BindJet Direct or infiltrated	Multijet	SLA / DLP - Direct or Wax-like castable	FDM / Extrusion based
Applications	Crown and bridges	,[7]	[2],[7]	[2]		[1], [2], [3],[4],[7],[8]	[3],[8]	[8]				
	Denture	,[7]	[2]	[2]		[1], [2], [3],[4],[7],[8]	[3],[8]	[8]				
	Denture holder	,[7]		[2]		[1], [2], [3],[4],[7],[8]	[3],[8]					
	Copings	,[7]	[2]			[1], [2],[4],[8]	[3],[8]					
	casting patterns /lost wax	,[7]		,[7]		[1], [4]				[3],[8]	[3],[8]	
	provisional / temperary crown					[1]		[6]		[5]	[3],[5]	
	dental model					[1]				[3] ,[5], [6]	[5], [3]	
	surgical guide					[1],[3]				[3] , [6]	[3]	1
	surgical guide plate					[1],[3]		[5]	[5]	[5]	[5]	[3]
	splints					[1]				[3],[5]	[3],[5]	1
	Prosthetic Constructions					[1]						
Material	All-Ceramic	1	1	[2]		1 , [6]		[6]				
	Porcelaine	1		[2]		1 , [6],[7]						, [6]
	Y-TZP	1				1 , [6],[7]						
	Metallic		1			1 , [6]	[1],[5]					
	glass-ceramic	1	1	[2]		1 , [6]		1 , [6]	1	[6],[8]		
	Polymer-Ceramic Composite	1	1			1 , [6],[7]		1	1		[3],[4],[5],[8]	
	Metal-Ceramic Composite	1	1			1 , [6]		1				
	Polymeric material					1		1	1	[3],[8]	[3],[4],[5],[8]	

1 - (Lin et al., 2019) ; 2- (Denry, 1996); 3-(Lin et al., 2019); 4- (Li et al., 2014); 5- (Sulaiman, 2020); 6- (Denry and Kelly, 2014); 7-(Anusavice, 2013); 8-(Torabi et al., 2015)

In spite of that, CAD/CAM techniques also show some disadvantages, such as:

- Requirement of high trained professional to operate 5 axes CNC.
- High cost of raw material
- Excessive waste generation
- High cost of maintenance
- Short life time of tooling

On the other hand, additive manufacturing might bring the fabrication of dental prosthesis to the next level, increasing automation, flexibility, shape freedom and fabrication speed. Nevertheless, material and technology restrictions, such as mechanical strength, type of material, bio reactivity and cost are still challenges to be overcome (Lin et al., 2019).

For that reason additive manufacturing technologies are widely used for:

Provisional crown and bridge restorations, casting patterns, dental models, surgical guide and splints. On the other hand, AM technologies still need to be developed in order to apply in dental implants, crown and bridges denture and prosthetic constructions.

Therefore, the main goal of this work is to propose and investigate the feasibility of a novel dental prosthesis fabrication method which is based in $ZrSiO_4$-glass composite, as illustrated in Figure 1.

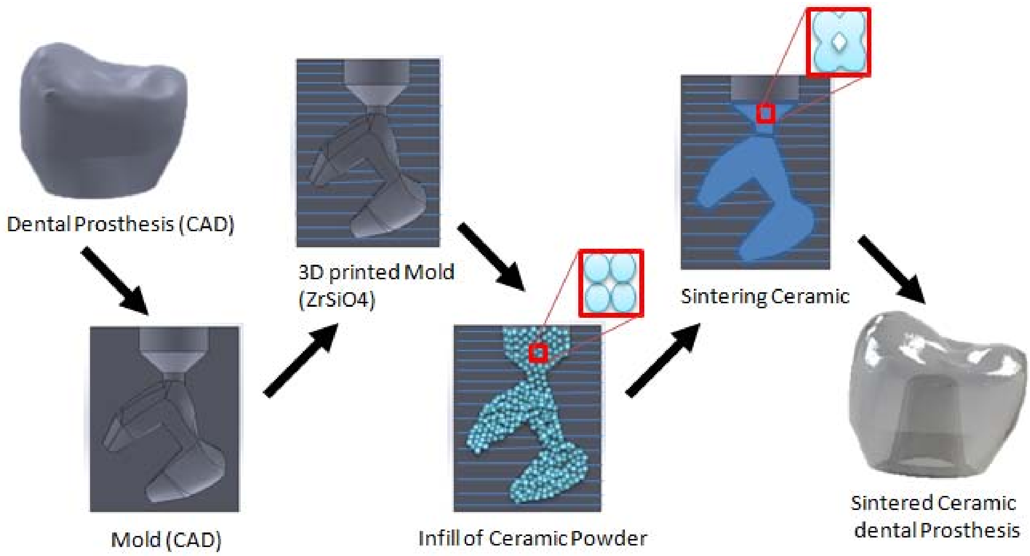

Figure 1: Schematic of ceramic dental prostheses based on ZrSiO4 – Glass composites fabricated by indirect Additive manufacturing

In this method, the collapsible ZrSiO4 mould of negative of crown or bridge is fabricated by additive manufacturing based on fused filament. Therefore, the negative cavity is filled by Lanthanum glass powder in order to be subsequently sintered. By the end, the pieces are heated in order to debind the negative structure and then create sintered composite pieces.

In order to evaluate the feasibility of this technique, we applied multivariable techniques 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, process feasibility were the study responses. We have also kept the fabrication parameters, materials formulation, flexural testing specimen shape and crown shape constant.

As a consequence, it was possible to identify whether the proposed process has potential feasibility to make dental prosthesis.

II. Material and Methods

In order to investigate the feasibility of the proposed process 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 3 step screening in augmented design approach in order to minimize the holding time and maximize densification and mechanical properties.

The levels and values of each control factor is presented in Table 2, where it is possible to see that the values of holding temperature are between the activation temperature of glass and ZrSiO4 (700°C) and the melting temperature of glass (1078°C).

It is also possible to indicate that the cooling time enhance two types of heat treatment (Quenching for fast cooling and annealing for slow cooling). Therefore, it is possible to see the effect of such treatments on, crystallization level, mechanical strength and geometry distortion.

On the other hand, holding time and heating rate are expected to affects the sinterization parameters, such as nucleation, grain growth and diffusion.

Table 2: Experiment Design

	Control Factors	Level -1	Level 0	Level 1
1st step	**sintering/kiln**			
	heating rate(Hr) (°C/min)	2	3.5	5
	Holding temperature (Th) (°C)	700	800	900
	holding time(th) (h)	1	2.5	4
	cooling rate (Cr) (°C/min)	2		30
2nd step	**sintering/kiln**			
	Holding temperature (Th) (°C)	700	750	800
	holding time(th) (h)	1	1.75	2.5
3rd step	**sintering/kiln**			
	Holding temperature (Th) (°C)	700	725	750
	holding time(th) (h)	2.5	2.875	3.25

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

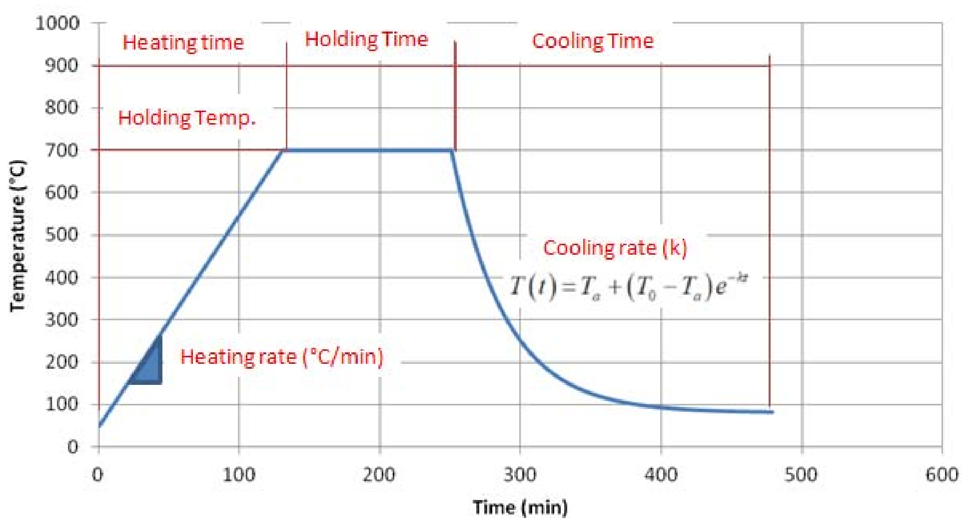

Figure 2: Illustration of sintering temperature curves and control factors: Holding Temperature (T_{hold}), holding time (t_{hold}), cooling rate (k) and heating rate (Hr)

The flexural testing was performed in accordance with ISO 6872 in an EMIC DL10000 universal testing machine.

For the image processing, we used the software MATLAB, while the image acquisition was performed by the optic microscope Digital Avangard Optics AN-E500 (AVANGARD 2011). This optical microscope provides until 500x of amplification magnitude. For the gravimetric analysis (drying monitoring), we used a 0.005g error scale.

In order to identify the specimen dimensional distortions, we used 0.05mm calliper besides computational image processing and MATLAB software to evidence the geometrical variation of object external contour.

In order to measure feasibility response, we established a scale from 0 to 1. In this scale, 1 level indicates that object has no significant distortions and is feasible to be used. In addition, 0.75 levels expose minor distortion which corresponds to 5% of distortion. Likewise, 0.5 feasibility level indicates that the object has 10% of distortion.

It is important to note that although shrinkage is a sort of volumetric distortion, feasibility response only analysed non volumetric distortions.

For specimens fabrication, we used a FDM process and filament filled by 90% of $ZrSiO_4$. The main process parameters were remained constants, where extrusion temperature was 220°C, layer thickness was 0.1mm; distance between filaments was 0.2 mm; and nozzle diameter was 0.4mm. Additionally, 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 constants, while no bed temperature was established. The fabrication environment was also controlled in 25°C of environment temperature and 50% of relative humidity.

III. Results and Discussions

In general lines, 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 while high levels of temperature distorted the geometry because of excessive melting and high shrinkage.

With regards to the main effect of control factors on the feasibility, mechanical strength and shrinkage, Figure 3 indicates that holding temperature causes the strongest effect on the feasibility and flexural strength in comparison with the other control factors. On the other hand, holding time is the factor that affects shrinkage the most.

In this diagram, it is possible to see that holding time and holding temperatures are the most relevant factors for the augmented design. Therefore, the second screening round focused in increase the detail of feasible areas.

The flexural strength was also affected by heating rate, indicating that densification of material might have reduced the strength of material.

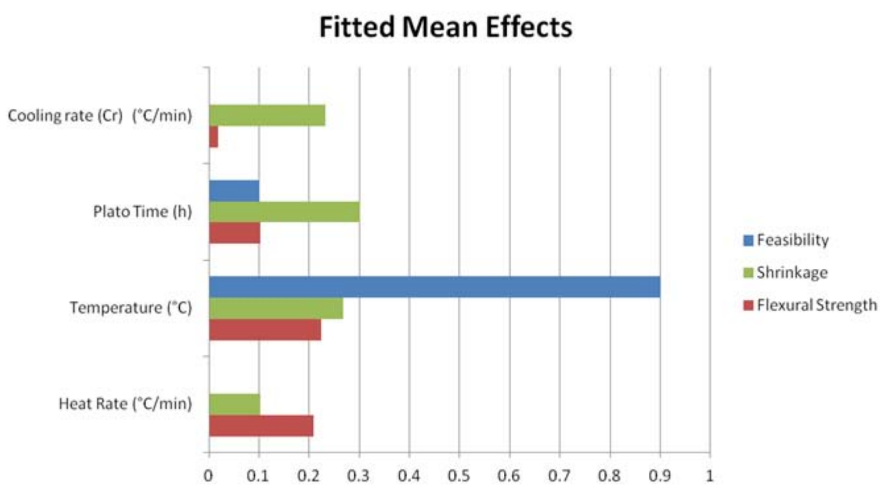

Figure 3: Main effect of control parameter on feasibility, flexural strength and shrinkage

With regards to the feasibility of proposed concept, the main effect diagram (Figure 4) indicated that high level of temperature implies on the unfeasibility of concept apart from time holding, cooling rate and heating rate. It is also possible to see that holding temperature is the most relevant parameter for the feasibility, being followed by holding time. In contrast, heating and cooling rates were found not to affect feasibility.

Continuing analysing the concept from the geometric point of view, it was possible to see that the shrinkage varied from 13.4% to 27% into the feasible area. The lowest value of shrinkage was found for heating rate equal to 5°C/min, holding temperature of 700°C, holding time equal to 3.25h and cooling rate of 30°C/min. Likewise, the highest value found resulted from heating rate equal to 2°C/min, holding temperature of 700°C, holding time equal to 4h and cooling rate of 2°C/min.

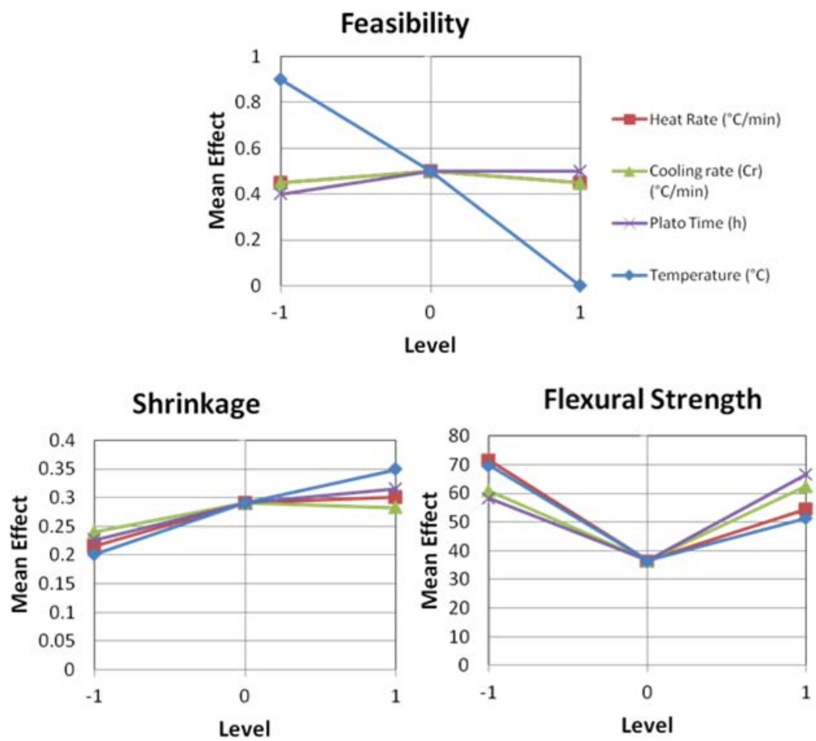

Figure 4: Standardized Main effect of control factors on Feasibility, Flexural strength and shrinkage

In this case, Figure 4 indicates the main effect of control parameter shrinkage. It is possible to see that holding temperature is the factor which is the most relevant for the shrinkage, while heating rate presented the smallest effect among the control parameters.

With respect to material mechanical strength, Figure 4 also indicates the main effect of control parameter on flexural strength. It is possible to see that temperature and heating rate are the most relevant factors for the mechanical strength, while cooling rate presented the smallest effect among the control parameters.

The mean values of flexural strength varied from 25 to 82MPa, where the lowest values were found in low holding temperatures (700°C) and short holding time (1h). On the other hand, the highest values were obtained by long hold time (1h) and 800°C of holding temperature.

It is also important to indicate that this process does not evaluate the effects of neither heating treatment nor material, therefore. Further studies still need to be done in order to apply stronger materials and heating treatments in this concept.

Additionally, Figure 5 indicates a comparison diagram of geometrical concept feasibility as a function of holding temperature and holding time. In this figure, it is possible to see a feasibility line that separates the results which were considered feasible and unfeasible from the geometrical point of view.

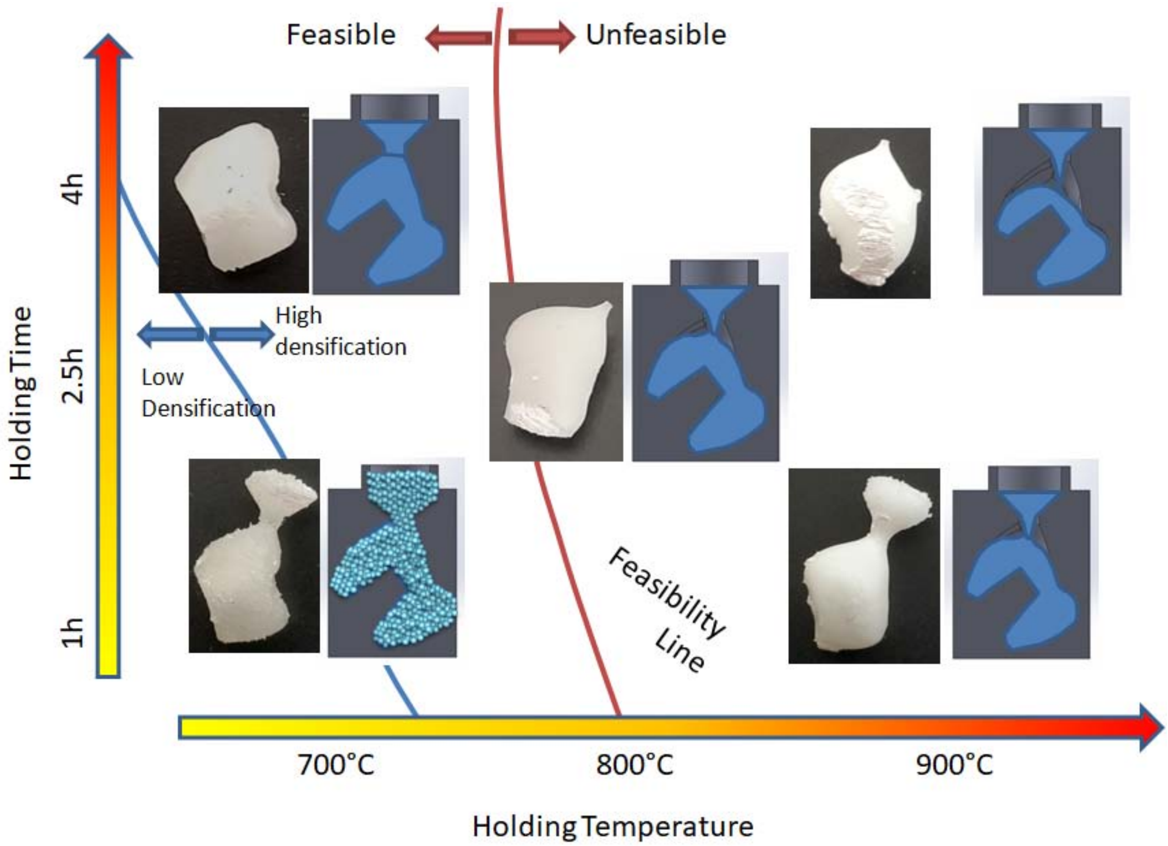

Figure 5: Comparison diagram of concept feasibility as a function of Holding temperature and holding time

We also separated the feasibility in 2 areas, whereas high grain growth is obtained in low temperature (700°C) during long holding time (4h). In these areas, it is possible to see a specimen with high densification. On the other hand, low grain growth is obtained in low temperatures (700°C) during short periods of time (1h), where the specimen presented low densification. Noteworthy, the centre point of the study indicated a limit of feasibility so that the sintering process can be directed correlated to the absorbed energy as a function of time and Temperature.

The unfeasible area is highlighted by high densification, and excess of deformation because of viscosity decrease. In addition, this reduction implied on mould infiltration and subsequent incrustation of collapsed mould on object surface.

It was seen that the material obtained in high temperature and long holding time implied on high densification and grain size which are bigger than grain in material from low temperatures. This situation can be seen in Figure 6, where the comparison of material densification and grain size is presented.

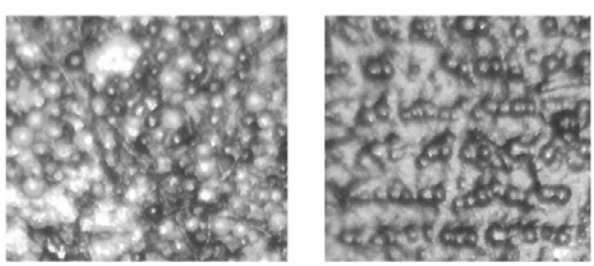

Figure 6: Comparison between low (a) and high (b) densification

In order to better understand the behaviour of material properties as a function of holding temperature and holding time, Figure 7 exposes contour diagrams of feasibility, shrinkage and flexural strength. Additionally, this figure also presents an overlap diagram which indicates the process window where high values can be obtained.

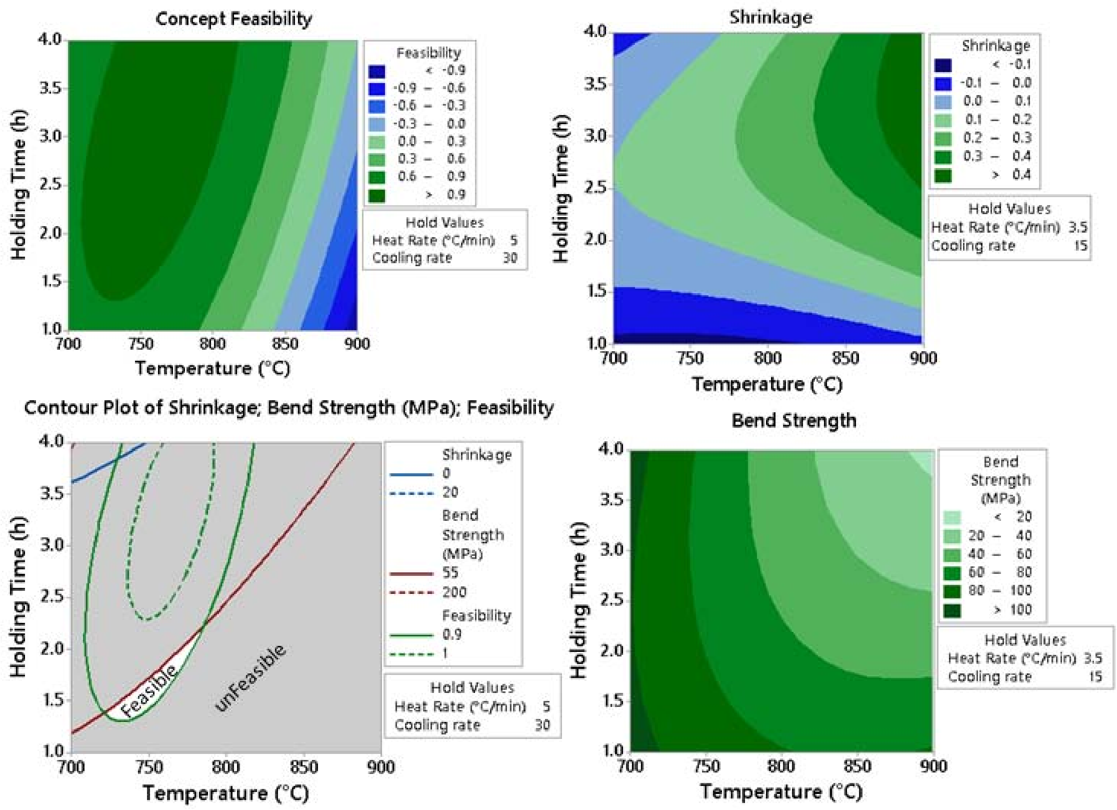

Figure 7: Contour diagrams of Flexural strength, feasibility, shrinkage and the overlap diagram with combination of high values

According to this figure, high values of feasibility flexural strength and low values of shrinkage are obtained by holding time around 750°C and holding temperature around 1.5h. In this case, values of flexural strength fluctuate around 65MPa, while shrinkage values do around 15%. In this case, feasibility ratio was considered higher than 0.9.

This concept has been shown to work and open a new possibility to fabricate glass-ceramic materials by AM technologies. However, further studies are still needed to improve mechanical strength, diversify the glass-ceramic materials and applications.

IV. Conclusions

To sum up, this work evidenced the feasibility of the glass-ceramic fabrication based on collapsible additive manufactured mould of ZrSio4. The working proof of this concept generates new perspectives to AM in dentistry, ceramics and medical applications, whereas this collapsible AM mould supports up than 2300°C.

This study identified that the holding temperature was the factor that mostly influences the feasibility, strength and shrinkage, being followed by holding time. In this case, holding time directly affects the material densification and grain growth. As consequence, long time and high temperatures increases the densification and soften the material so that the geometry is distorted and the process become unfeasible.

On the other hand, flexural strength fluctuated between 25 and 82MPa, and has been highly affected by heating rate and cooling rate. However, this study did not analyse heating treatment. Therefore, further studies are still needed to be done in order to apply new materials, heating treatments and increase mechanical and geometrical properties.

Acknowledgement

The authors would like to thank Concep3D R&D and FAE Centro Universitário for the support and infrastructure.

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