Development of a 3D Printing Method for Production of Dental Application

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Abstract

Traditionally, the manufacturing of dental restorations, including crowns, veneers and other structures made by ceramics, is a labor-intensive and time consuming process. Additive manufacturing has the potential to significantly decrease the time and cost associated with this process. This work performed preliminary investigation for the feasibility of dental restoration parts printing using the ExOne M-Lab system with a commercialized dental porcelain powder. The porcelain powders were characterized, and two measurements, including pre-sintering and addition of flow agent, were taken in the attempt to improve the processability of the original powder feedstock. The results showed that while the addition of flow agent has more significant effects in improving the flowability of the powder used, the post sintered parts exhibit considerable shrinkage and residual porosity that necessitates further investigation.

Introduction

Over the past decades, there has been a trend of using metal-free restorations in the dental field (Denry and Holloway, 2010, Guess et al., 2011). Ceramic dental restorations have been increasingly used due to their outstanding aesthetic features and resistivity to chemicals (Conrad et al., 2007, Kelly, 1999). Traditionally, the dental ceramics were produced by hot-pressing, sintering or slip casting processes (Denry and Holloway, 2010), which in general lack sufficient flexibility in the accurate customization of the parts. Machining was used as well, but often suffers from the high hardness and low toughness of the ceramic feedstock. In recent years, a CAD/CAM based method was developed to produce dental restorations with geometries accurately matching that of the patients' (Filser et al., 2003, Guess et al., 2011). The basic procedures of this process involves the pre-sintering of ceramic blocks followed by a CNC machining process that produces accurate shapes, then the green machined parts are subject to a densification sintering process to generate the final densities as well as mechanical performance. The use of the two-stage sintering helps reduce the issues associated with machining of hard ceramics, therefore increasing the efficiency of the process with lower cost. However, due to the shrinkage in the second sintering stage, the accuracy of the machining could be significantly reduced, which compromises the benefit intended to be provided via machining. In addition, the relatively complicated procedures also result in longer manufacturing time and higher cost.

With the ability of manufacturing parts directly from a CAD model with adequate accuracy and minimal waste, additive manufacturing holds great potential for the future production of custom dental restoration parts. Several laser sintering based studies have been presented in the attempt to fabricate dental restorations with various materials (Li et al., 2000, Hagedorn et al., 2011). The use of a laser for the direct

sintering of ceramics enables one-step processing for the manufacturing of these parts, and provides the potential for direct manufacturing of ceramic-over-metal dental parts. On the flip side, thermal cracking is a common issue for these processes (Wilkes, 2010), and the feature resolution is also often limited due to the partial sintering of the surrounding ceramic powders (Hagedorn et al., 2011). Recently, a direct write based printing process was investigated for the fabrication of zirconia dental prostheses (Ebert et al., 2009). The process selectively deposits a zirconia based suspension on graphite substrates, followed by a post sintering process to achieve the final density. The resulting parts exhibit high geometrical accuracy and density, as well as good mechanical strength. In this work it was also reported the potential issue with the nozzle clogging (Ebert et al., 2009).

In this study, the ExOne M-Lab was used in an attempt to fabricate ceramic parts from off-the-shelf commercial ceramic powders used for dental applications. While the system is relatively new, there have been studies that utilize binder jetting to make ceramic parts (Cima et al., 1995, Uhland et al., 1999). This process offers some potential advantages in ceramic printing, such as the flexibility with different ceramic materials, the relatively high feature resolution, and easy process control; therefore binder jetting was adopted for this study with future developments in mind. The goal of this study is to identify the feasibility of the system for quick and accurate fabrication of complex-shaped, 3D dental restoration parts. Based on the preliminary study, future work can be determined in the effort to produce high quality parts with minimal defects.

Materials and Methods

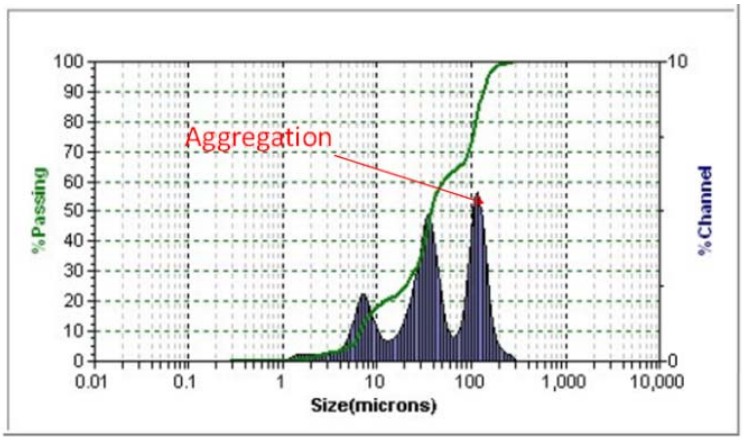
The primary material used for this study is VITA VM13 Base (Vita Zahnfabrik, Germany), which is a leucite-reinforced glass ceramic widely used for dental practice. The chemical composition and basic properties of the powder provided by the manufacturer is shown in Table 1 (Vita VM-13, 2009).

Powder	Chemical composition (wt%)	Particle size	Linear coefficient of thermal expansion	Flexural strength
VM-13 Base	SiO_2: 59-63%, Al_2O_3: 13-16%, K_2O: 9-11%, Na_2O: 4-6%	~18µm	13.6-14x10^{-6} K^{-1}	~120MPa

Table 1 General information of VM-13 Base

The original powders exhibit significant aggregation which made it unsuitable for the process. Preliminary particle size analysis (Microtrac S3000) indicated that the characteristic size of the aggregation was around 100-105µm, as shown in Fig.1. Two factors were considered to contribute to the aggregation. From Fig.2, it could be seen that the powders have a very irregular morphology and a rather wide size distribution, which could significantly reduce its flowability. In addition, the large surface activity of

the silica powders also makes them prone to clumping (Bagwe et al., 2006). Therefore, Pre-sintering and flow agent addition were implemented in an attempt to improve the issue.



Summary

Data Item	Value
MV(um):	60.19
MN(um):	2.153
MA(um):	17.1800
CS:	3.49E-01
SD:	56.0500

Fig.1 Particle size analysis results for the VM-13 Base powder

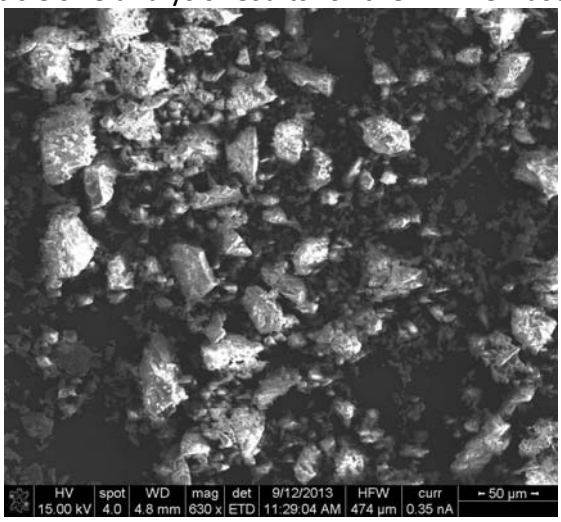


Fig.2 Morphology of the original VM-13 powder

Through a preliminary trial it was found that at about 600°C of sintering temperature, significant densification does not occur for the powder. Therefore, 600°C was chosen to be the pre-sintering temperature in order to achieve fast effects. The holding time for pre-sintering was kept at 30 minutes, followed by furnace cooling.

Surface-modified R972 SiO_2 powder (COSMOS Plastic & Chemicals) was used as the flow agent. The powder is composed of >99.8% fumed silica treated with dimethyldichlorosilane (DDS), with an average particle size of 16nm. The flow agent was added to the original VM-13 powder and mixed by hand until satisfactory improvement of flowability was observed.

The flowability was evaluated by the angle of repose for the original and treated powders. In addition, micrographs were taken for the pre-sintered powder to evaluate the evolution of powders. After the treatment, the processable powders were used to

print sample crown parts using the M-Lab. The binder used for the process was the ExOne PM-B-SR1-04, an ether solvent based binder. After printing, the green parts were dried in the oven at 70°C overnight. The schematic of the post sintering process is shown in Fig.3. The green parts were held at 500°C for 1 hour to burn out the binders, followed by a two-step sintering sequence at 700°C and 850°C, respectively. The dimensions of the final parts were measured with caliper before and after sintering, and results were compared to evaluate the shrinkage. The cross sectional microstructure of the sintered samples were also prepared and observed using optical microscopy.

Results and discussion

Pre-sintering did not seem to have significant effect on the flowability of the powder. Fig.3 shows the morphology of the powders after the pre-sintering. From Fig.3 it could be clearly observed that significant sintering occurred which resulted in the generation of large powder aggregations. On the other hand, the flow agent had a more significant effect on the flowability of the powder at the ratio of approximately 7% in volume. Fig. 4 shows the angles of repose for each sample, and Table 2 shows the results of the measurement. Pre-sintering was used for ceramic powders with fine particle sizes in previous studies using selective laser sintering (Harlan et al., 1999). However, from this study it seems apparent that with highly irregular powders, even if the pre-sintering could reduce the total surface area, the increase of particle irregularity will cancel out all the benefits and potentially result in the reduction of flowability. It was expected that the addition of flow agent could improve the overall powder flowability by serving as a lubrication interface between the large VM-13 powders, which was verified by the study.

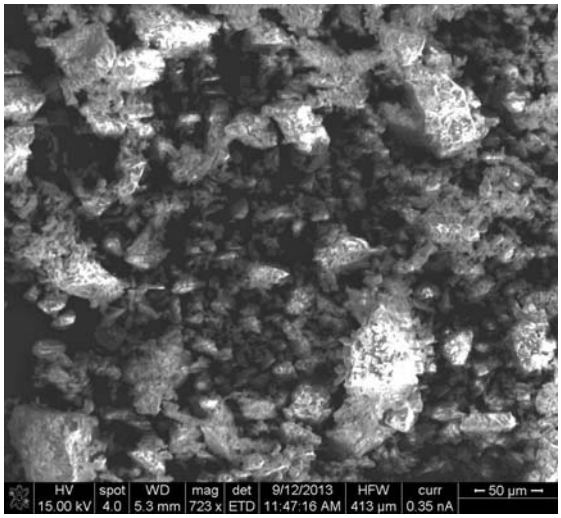


Fig.3 Morphology of VM-13 powder after pre-sintering

(a) Original (b) Pre-sintered (c) Flow agent addition

Fig.4 Angles of repose for each type of powder

Powder	Angle of repose (degree)
Original	64
Pre-sintering	66
7%v addition of flow agent	56

Table 2 Angles of repose for each type of powder

The powder with flow agent addition was successfully used for the printing of crown parts in the M-Lab and sintered subsequently. Fig.5 shows the original part as well as the post sintered parts. Significant shrinkage could be observed. The dimensions as shown in Fig.6 were measured for three samples, and the results are shown in Table 3. The results are quite consistent for each dimension, and an average of 25%-32% shrinkage was observed in each dimension compared to the green parts. The shrinkage values are considerable but reasonable considering that the powder is highly irregular. A slight difference of shrinkage between the build direction (aligned with the dimension H) and the other two directions were observed, which could be caused by the burnout of the binder. There is also a slight difference of shrinkage between the two planar directions perpendicular to the build direction, as could be seen from Table 3. This could be associated with the specific geometry of these samples such as wall thickness. However, without further investigation, it is difficult to determine the likely cause of this phenomenon

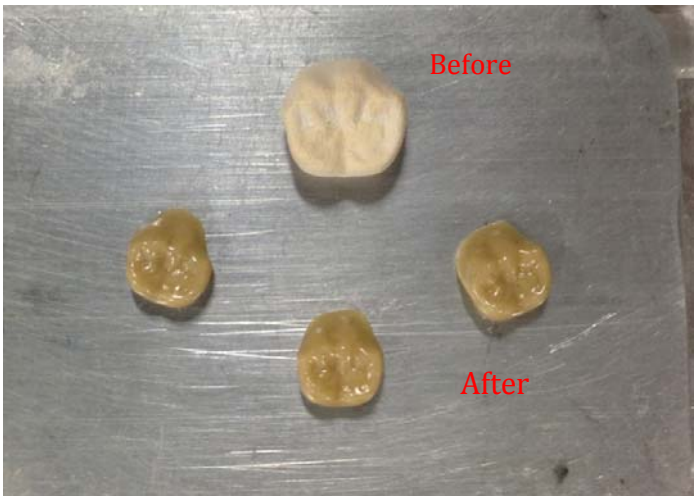


Fig. 5 Green and post sintered crown parts

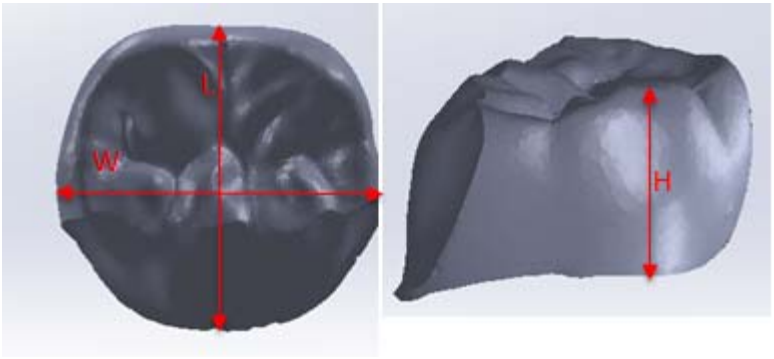


Fig.6 Measurement of dimensions

Sample	L (mm)	W (mm)	H (mm)
Before sintering			
1	11.50	11.55	6.00
2	11.45	11.55	6.07
3	11.43	11.58	6.03
Post sintering			
1	8.80	8.16	4.12
2	8.61	8.11	4.08
3	8.60	8.09	4.13
Linear shrinkage			
1	23.48%	29.35%	31.33%
2	24.80%	29.78%	32.78%
3	24.76%	30.14%	31.51%
Average	24.35%	29.76%	31.88%

Table 3 Shrinkage of the samples

Fig.7 shows the microstructure of the ceramic samples after sintering. In general the sintering seemed to have achieved a relatively homogeneous densification. Large voids as well as some light particles could be clearly observed. The size of the voids varied but

were at the magnitude of 50-100µm. This seemed to have a correlation with the size of the aggregation of the original powder. One possible cause could be that during the powder spreading process, the large aggregation was displaced by the roller due to the temporary adhesion to the roller surface, which resulted in surface voids on the newly spread layer. Another potential source that could contribute to the void formation is the addition of the flow agent. The mechanism of the surface modification in the flow agent was to create a hydrophobic surface on the silica particles. Since sufficient wetting is required to form a continuous bonding between the binder and the powder, the existence of the flow agent could potentially affect the continuity of the green part, leaving voids that become defects after the subsequent processes. Further studies are needed to identify the potential impact of the flow agent in the final densities of the parts. In addition, the use of 850°C as the densification sintering temperature was largely due to the limitation of the furnace, while in the reference instruction for the VM-13 powder (Vita VM-13, 2009), it was recommend that the green part be sintered at 920°C. The lower temperature could also contributed to the porosities observed in the sample.

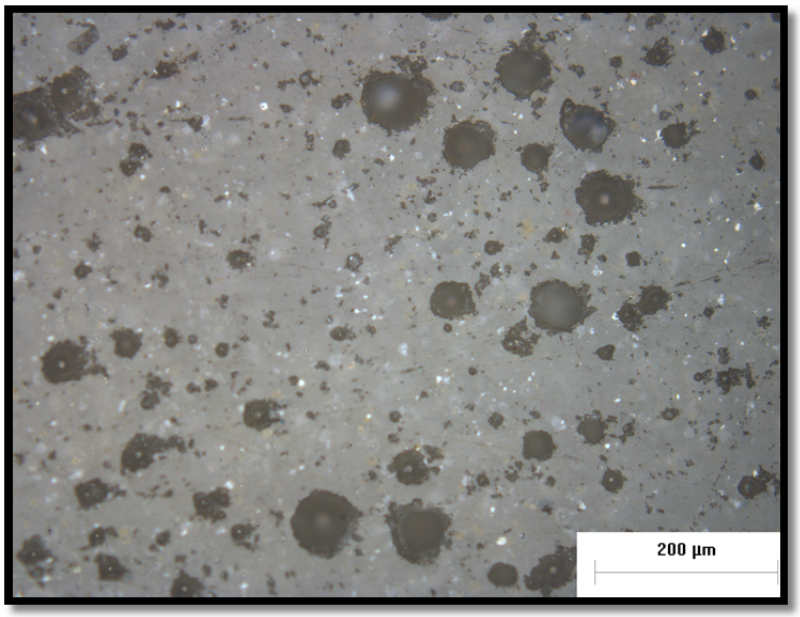


Fig.7 Microstructure of the post sintered part

Conclusion

In this study, an off-the-shelf dental veneer powder was successfully printed using an M-Lab. The original powder had significant aggregation issues, and two measurements were used in the attempt to improve the flowability of the powder. Addition of the flow agent of about 7% volume was shown to be an effective method to improve the original powder flowability. At relatively low sintering temperature, the final parts showed homogeneous densification and continuous microstructures, although large voids were

present in the final parts. The shrinkage of the parts after sintering was around 25-30% in each directions, and anisotropic shrinkage was observed in all three directions. Further investigations are needed to identify the cause of the anisotropic shrinkage as well as the source of the large voids in the final parts, and additional mechanical testing is also required to further characterize the quality of the process.

Reference

R. P. Bagwe, L. R. Hillard, W. Tan. Surface Modification of Silica Nanoparticles to Reduce Aggregation and Nonspecific binding. Langmuir. 22(2006): 4357-4362.

M. J. Cima, J. Yoo, S. Khanuja, M. Rynerson, D. Nammour, B. Giritlioglu, J. Grau, E. M. Sachs. Structural Ceramic Components by 3D Printing. Proceedings of the 6th International Solid Freeform Fabrication Symposium. Austin, TX, USA. 1995.

H. J. Conrad, W.-J. Seong, I. J. Pesun. Current ceramic materials and systems with clinical recommendations: a systematic review. Journal of Prosthetic Dentistry. 98(2007): 389-404.

I. Denry, J. A. Holloway. Ceramics for Dental Applications: A Review. Materials. 3(2010):351-368.

J. Ebert, E. Ozkol, A. Zeichner, K. Uibel, O. Weiss, U. Koops, R. Telle, H. Fischer. Direct Inkjet Printing of Dental Prostheses Made of Zirconia, Journal of Dental Research. 88(2009): 673-676.

F. Filser, P. Kocher, l. J. Gauckler. Net-shaping of ceramic components by direct ceramic machining. Assembly Automation. 23(2003): 382-390.

Y.-C. Hagedorn, N. Balachandran, W. Meiners, K. Wissenbach, R. Poprawe. SLM of net-shaped high strength ceramics: new opportunities for production dental restorations. Proceedings of the 22nd International Solid Freeform Fabrication Symposium. Austin, TX, USA. 2011. N. Harlan, S.-M. Park, D. L. Bourell, J. J. Beaman. Selective Laser Sintering of Zirconia with Micro-Scale Features. Proceedings of the 10th International Solid Freeform Fabrication Symposium. Austin, TX, USA. 1999.

J. R. Kelly. Clinically relevant approach to failure testing of all-ceramic restorations. Journal of Prosthetic Dentistry. 81(1999): 652–661.

X. Li, J. E. Crocker, E. Geiss, L. L. Shaw, H. L. Marcus, T. b. Cameron. Evaluation of Microstructure and Properties for Multi-Materials Laser Densification of Dental Restorations. Proceedings of the 11th International Solid Freeform Fabrication Symposium. Austin, TX, USA. 2000.

S. Uhland, R. Holman, B. DeBear, P. Saxton, M. Cima, E. Sachs. Three-dimensional printing, 3DP, of electronic ceramic components. Proceedings of the 10th International Solid Freeform Fabrication Symposium. Austin, TX, USA. 1999.

VITA VM-13 Work Instructions. VITA Vident webiste. http://vident.com/products/veneering-materials/metal-ceramics/vita-vm13/.

J. Wilkes, Y.-C. Hagedorn, S. Ocylok, W. Meiners, K. Wissenbach. Rapid manufacturing of ceramic parts by selective laser melting. Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials IV. Vol.31. 2010.