Laser Densification of Extruded Dental Porcelain Bodies in Multi-Material Laser Densification (MMLD) Process

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ABSTRACT

In this study commercial dental porcelain powder was deposited via slurry extrusion and laser densified to fabricate dental restorations in a Multi-Material Laser Densification (MMLD) process. The processing conditions for laser densification of single lines and closed rings were investigated in order to avoid warping and cracking. Multi-layer rings were also investigated to study the dependence of bonding between layers on the laser densification conditions. The laser densified rings showed no warping, and good bonding between layers could be achieved when the laser densification condition was selected properly. The mechanism to achieve porcelain rings without warping and cracking is discussed. The understanding developed will pave the way for fabricating a physical dental restoration unit.

KEY WORDS: Solid freeform fabrication, multi-material laser densification, dental restoration

INTRODUCTION

Porcelain-fused-to-metal (PFM) is a dominant dental restoration method currently being used for permanent fixed prosthodontics. In PFM the dental restoration is cast from a dental alloy and then the metal substrate is coated with several dental porcelain layers by firing processes in a furnace. PFM restoration requires a multi-stage process using multiple materials (metals and porcelains) and each stage involves multiple processing steps. As such, PFM is time-consuming and costly. Labor costs account for about 90% of the final cost, while only 5% of the cost is due to dental materials.

Solid Freeform Fabrication (SFF) has provided an extensive change in design and manufacturing of 3-D parts. In SFF, physical objects are fabricated additively layer-by-layer directly from a computer model of the desired part without part-specific tooling and human intervention [1]. The fabrication time and cost can be greatly reduced, and the complex shapes can be fabricated in one operation that would otherwise require multiple operations in traditional manufacturing procedures. More than 24 SFF techniques have been developed by research organizations and commercial companies in the recent past [2]. Furthermore, some of the SFF techniques have been directly applied or modified to make dental restorations recently. The variants of these techniques include (i) milling of composite blocks into 3D crown models by a CAM machine, (ii) direct ceramic machining (DCM) of a zirconia dental bridge, (iii) dental models fabricated via Stereolithography Apparatus (SLA), and (iv) fabrication of 3D crown models by a ModelMaker (MM) inkjet system [3-6]. These techniques can only fabricate either a dental model which needs traditional investment casting procedure to make an artificial tooth, or a restoration unit made of non-commercial dental materials. Therefore, a new dental restoration process, called Multi-Material Laser Densification (MMLD), is currently under development in order to make dental restorations made of multiple materials (e.g., dental alloys and porcelains) similar to PFM restorations. MMLD entails the extrusion of dental powder pastes in a layer-by-layer fashion and densification of the extruded powder layer using a laser beam before the next layer is extruded [7-10]. MMLD has the potential to fabricate dental restoration units in one operation and thus provides an alternative for the labor-intensive and costly PFM restoration process.

EXPERIMENTAL

Starting Materials

The dental porcelain powder Ceramco© II Silver Body used in this study was provided by Dentsply Ceramco, Burlington, NJ. The chemical composition of this porcelain is within 5% of the composition of the No. 1 Component in Weinstein patent, shown in Table 1. The size of the as-received powder particles ranges from 10 to 50 μm and particles have irregular shape. The as-received powder was ball milled in order to decrease the particle size down to an average size of 1 μm, Figure 1. The ball-milled porcelain powder was mixed with de-ionized water at a ratio of 50% - 50% (vol. %) to make a porcelain paste for extrusion.

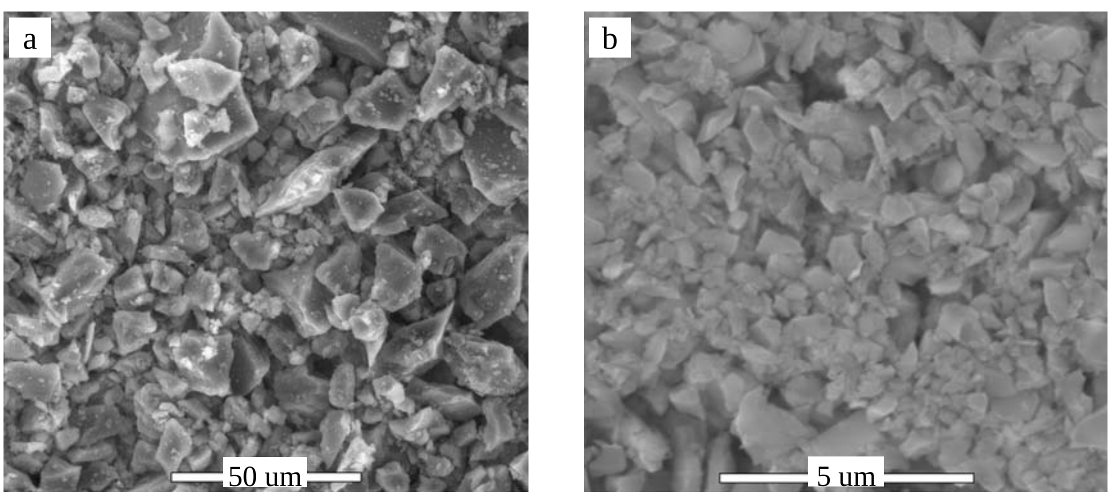

Figure 1. The scanning electron micrograph of (a) as-received dental porcelain powders and (b) ball-milled powders. Different scales are used in (a) and (b).

Previous studies [7-10] have shown that the as-received dental porcelain powder is composed of two phases, the crystalline leucite ($K_2O{\cdot}Al_2O_3{\cdot}4SiO_2$) particles imbedded in a feldspar-based glass matrix. Leucite has a high coefficient of thermal expansion (CTE) in the range of 20-25 × 10^{-6}/°C and raises the inherently low CTE of the feldspathic glass matrix (7-8 × 10^{-6}/°C) to a level, which matches the CTE of the dental alloy substrate in the PFM procedure, typically 14-17 × 10^{-6}/°C. The CTE match is one of the most critical issues in PFM restorations because the probability for micro-cracking due to mismatch in CTE between the dental alloy and porcelain coats cannot be overlooked.

Table 1. Composition of No. 1 Component in Weinstein patent

Oxides	SiO_2	Al_2O_3	K_2O	Na_2O	CaO	MgO
Wt. %	63.40%	16.70%	14.19%	3.41%	1.50%	0.80%

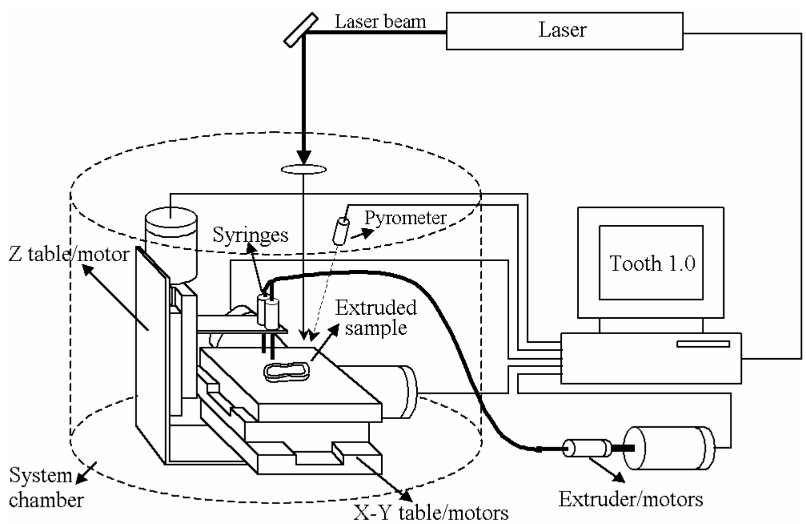

Figure 2. Schematic of the MMLD system used for delivering and laser densification of dental porcelains.

Paste Extrusion and Laser Densification

The laser densification of dental porcelains was carried out in an integrated MMLD system composed of a continuous wave CO_2 laser, a process chamber, a powder paste extrusion system, an X-Y-Z positioning system, and a closed-loop temperature control system, Figure 2. The porcelain paste was loaded into a micro-extruder and then extruded onto a SiC substrate on the X-Y table. The extruded paste was heated by a laser beam with a large diameter of about 5-8 mm at very low power to make it dry, and then at high power to densify it. After the first layer was densified, the second layer would be delivered onto the first layer and densified by the laser beam. Such extrusion-drying-densification sequence was repeated to fabricate single-line and multi-layer shapes. Two control modes could be used in the laser densification process: (i) temperature control mode, where the user-defined surface temperature (also termed as the target surface temperature) was maintained by a closed-loop control system and the laser output power was adjusted according to the feedback of the pyrometer; (ii) laser power control mode, where the laser output power was defined by the user and maintained constant in the entire densification process. The second mode was used in this study. The laser densification conditions included: laser beam size 1-7 mm, laser output power 1-3 W for 1-2 minutes to dry the green paste, and 20-30 W for 10-30 seconds to densify it. The laser beam was moving either along a straight line or a circle, or stationary.

Microstructure Evaluation

Densified porcelain bodies were mounted using an epoxy and finish-polished with 1 μm diamond suspension. The samples were examined first at the as-polished condition using an optical microscope to evaluate the general microstructure and porosity of the laser densified bodies. Subsequently, the polished samples were etched in 1% hydrofluoric (HF) acid for 15 seconds to reveal the leucite phase from the glass matrix. The microstructure observation was then accomplished using an optical microscope or an environmental scanning electron microscope (PHILIPS ESEM 2020) after carbon sputter-coated.

RESULTS AND DISCUSSION

Cross Sectional Geometry Control of Laser Densified Porcelain Lines

The cross sectional shape of a laser densified porcelain line is a very important issue in laser densification processes because the densified line works as a “building block” to produce the final physical artificial tooth. The ideal laser densified line should possess a near rectangular cross section that has relatively vertical walls and flattened top, so that a densified line will be accommodated perfectly by the previously densified lines with a similar near rectangular shape, avoiding large voids between them. This is especially important for dental restoration where each single line can be less than 0.5 mm in width and the surface tension effect can be a major factor in determining the cross section geometry of the laser densified line.

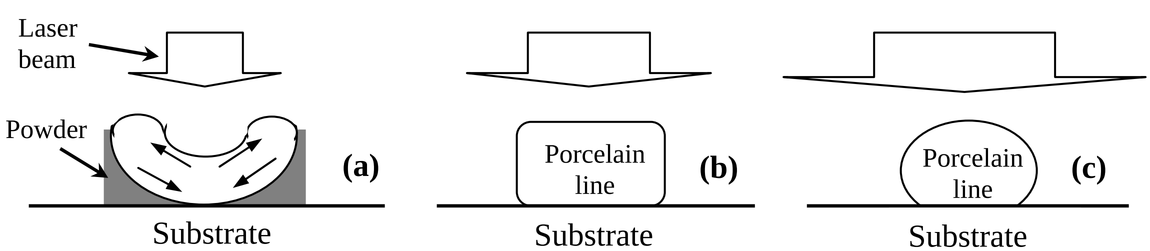

Figure 3. Schematic of (a) Marangoni effects, (c) balling due to surface area minimization, and (b) laser densification conditions between (a) and (c) and the possible cross-section geometry of a single line.

It is proposed schematically in Figure 3 that production of a laser-densified line with near rectangular cross section is achievable through controlling laser densification conditions. The most important parameter has been found to be the ratio of the laser beam size to the line width, *K*, defined by

$$K = \frac{2R}{w} \tag{1}$$

where w is the width of the powder line and $2R$ is the laser beam diameter. When K is very small, it is the case where the laser beam scans a powder bed or the laser beam is much smaller than the line width, Figure 3(a). On the one hand, due to the Gaussian power intensity profile of the laser beam, the scanning path center has the highest temperature, leading to the greatest shrinkage in the center; on the other hand, after the porcelain powder is melt, "Marangoni effects" will occur due to surface tension gradient, so the melt will flow in a way shown in Figure 3(a), leading to a concave downward cross section. Previous studies [8-10] have showed that this undesired cross section is un-avoidable in the case of powder bed densification. When K is very big, it is similar to the condition of furnace densification because of the relatively uniform laser power intensity over the thin line, so balling of the porcelain line will occur due to minimizing of surface energy, Figure 3(c). Thus, there might exist such a condition that when K is intermediate between the conditions of Figure 3(a) and (c), a near rectangular cross section could be formed.

The laser densification results corresponding to the conditions shown in Figure 3(a), (b) and (c) are present in Figure 4. It can be seen that the top of the cross section in Figure 4(a) is relatively flat, not concave downward as expected to be similar to the cross section of powder bed densified bodies, reported in references [8-10]. This is because the green line before laser densification in Figure 4(a) had a hemi-circle cross section, and during laser densification the line top shrank first, leading to the relatively flat top in Figure 4(a). If the top of the green line were flat (same case in powder bed densification) before laser scanning, the resultant cross section would be the same as that of powder bed condition, i.e. concave downward. The green porcelain lines leading to cross section in Figure 4(b) and (c) had relatively square cross section. Balling did occur as shown in Figure 4(c). The laser densified single line with a near rectangular cross section is shown in Figure 4(b). It is desired that the near rectangular cross section be maintained after the green line is laser densified, which is the case in Figure 4(b). The mechanism for producing such kind of the cross section is as follows: the line center on the top surface of the green line shrinks first when the laser beam scans the line due to the Gaussian nature of the laser beam. Then the edges of the line become rounded, resulting in the final cross section near rectangular. Of course, the laser densification conditions have to be controlled well in order to avoid balling.

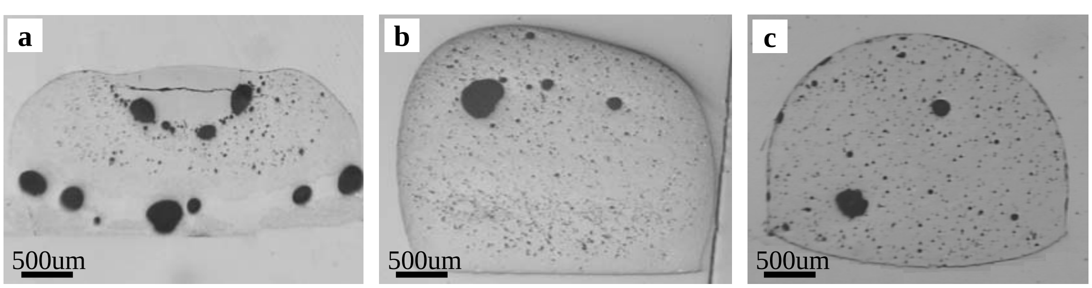

Figure 4. The cross section evolution after laser densification with different K value where (a) K=0.5, Marangoni effects occurred, (b) K=2.0, near rectangular cross section resulted, and (c) K=3.0, balling was dominant. Note that each of them corresponds to its schematic counterpart (a), (b) and (c) in Figure 3.

Macroscopic Continuity in Closed Lines after Laser Densification

Previous studies [8-11] have shown that when a scanning laser beam densifies a powder bed in a line mode or a green line is extruded and then laser densified, the warping occurs along the line length direction at both ends of a densified line body. In the case of densification of closed rings either from a powder bed or an extruded closed ring using a moving laser beam, a gap or groove at the connection point where the laser beam starts and ends the scanning can occur. The appearance of the discontinuity is related to the volume shrinkage changing from loose powder to a dense body at the starting point of scanning.

A new laser densification method was used in this study, i.e. using a stationary large laser beam to heat the green porcelain ring. A typical sample laser densified using this method is shown in Figure 5. It can be seen that the whole ring is continuous macroscopically without any gaps or grooves. This is because the whole porcelain ring was heated at the same time. In the case that a small beam size is used to scan the extruded ring or powder bed, there is always a scan starting and ending point on the ring. Because of this starting point the volume-shrinkage-induced discontinuity is unavoidable. However, when a stationary laser beam with a large size is used to scan the whole ring at the same time, the whole ring will be heated and shrink simultaneously. As a result, no discontinuity will be present.

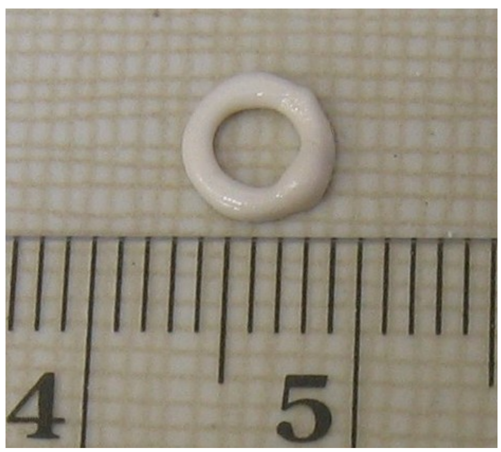

Figure 5. A circular porcelain ring after laser densification. The shrinkage rate is about 14% in terms of the ring diameter. Experimental conditions: beam size 7.0 mm, laser power 30 W, scanning time 15 s, outer diameter of the green ring 7.0 mm, and line width 1.5 mm.

The cross section of a multi-layer porcelain ring after laser densification is shown in Figure 6. From Figure 6(a) it can be seen that the interface between different layers are continuous, suggesting a good bonding strength. Also some bubbles are present at the interface. This was caused by residual air that had been trapped at the interface after extrusion of green porcelain ring onto the previously densified ring. Also note that in Figure 6(b) the third layer has relatively weak bond with the second layer because of the presence of a gap.

The key issue for good bonding between different layers is that there should be good contact between the extruded green layer and the already densified layer below, i.e. large air bubbles and gaps should be avoided at the interface before laser densifying the top layer. If there are gaps between the layers before laser densification, balling will be dominant during laser densification, leading to poor or no bonding at all with the previously densified layer. Prevention of large bubbles or gaps at the interface before densification is achievable through controlling the properties of the porcelain paste. The proper viscosity and pseudoplastic behavior of the paste are the keys to allow the appropriate flow of the extruded paste before it freezes in place [12].

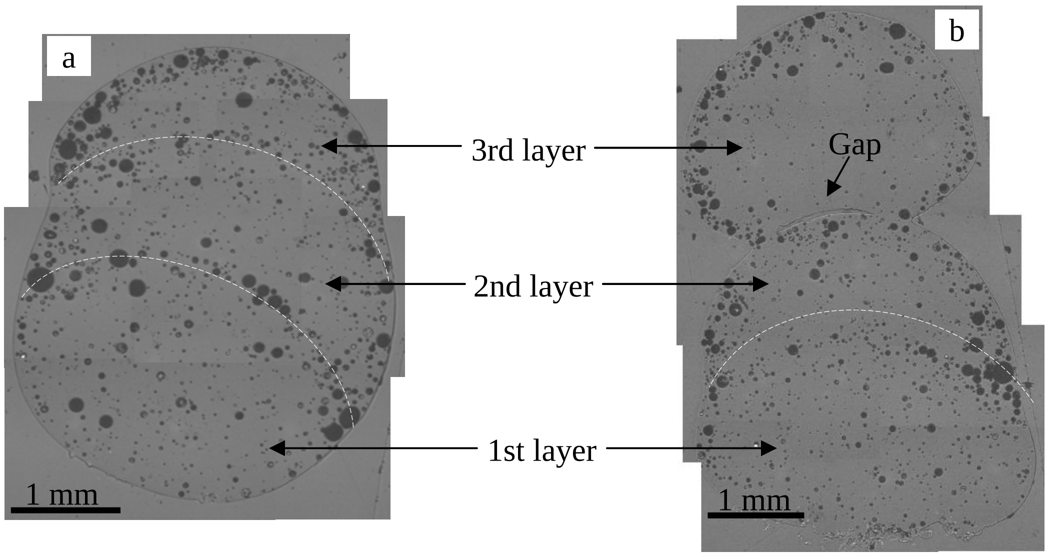

Figure 6. Typical cross sectional morphology of 3-layer porcelain sample after laser densificaiton. It can be seen that there are some bubbles (porosity) at the interface (highlighted by dash curves) of two layers. Note that in (b) the 3rd layer has weak bond to the second layer because of the presence of a gap between the two layers.

SUMMARY

Dental porcelain lines and rings were delivered using a MMLD system and laser densified. The densified single line could have a near rectangular cross section geometry when the laser densification conditions are set properly. A stationary laser beam with a large size was used to densify extruded porcelain rings. The result showed that the ring was smooth and continuous macroscopically. Laser densified multi-layer porcelain rings showed good bonding between different layers after densification. The viscosity and flowability of the extruded paste line should be well controlled in order to achieve good contact between the green layer and previously densified porcelain layer.

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