Microstructure Evaluation for Laser Densification of Dental Porcelains

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ABSTRACT

The feasibility of dental restorations has been investigated using a multi-materials laser densification (MMLD) process, where dental alloy and porcelain powders are laser densified layer by layer to form solid bodies. The present study focuses on the densification behavior of dental porcelain powders in response to a moving laser source. Effects of the laser processing temperature and the green density of the powder bed on microstructure, distortion, macro-cracks, porosities and phase contents of the laser densified porcelain have been investigated. The condition to generate continuous porcelain bodies from powder compacts have also be studied. It is found that the geometry, composition and density of densified porcelain bodies are strongly affected by the laser processing temperature and the green density of the powder compact.

INTRODUCTION

Dental porcelain-fused-to-metal (PFM) restorations are fabricated by fusing the porcelain to the metal at high temperatures. PFM restoration is a very time consuming and labor intensive work because PFM restoration requires a multi-stage process using multiple materials (both ceramics and metals) and each stage involves multiple processing steps [1]. Typically, 4 – 8 labor hours are needed to make the dental restoration of a three-unit bridge. As such, labor costs account for about 90% of the final cost to the patient while dental materials occupy only less than 5%. The current project is to develop a novel multi-materials laser densification (MMLD) process for dental restorations. This process utilizes laser-assisted solid freeform fabrication (SFF) to fabricate artificial dental units layer-by-layer directly from a computer model without part-specific tooling and human intervention. As such, the labor cost will be substantially reduced, and better and faster dental restorations will be achieved.

To develop a robust MMLD process for dental restoration, the densification behavior of dental alloys and porcelains in response to a moving laser source needs to be investigated. Some investigation results on dental nickel alloys have been reported in a previous paper [2]. The present study focuses on laser sintering of the dental porcelain delivered in both loose powder and paste form. Effects of the laser processing temperature and the green density of the powder bed on microstructure, distortion, macro-cracks, porosities and phase contents of the laser densified porcelain have been investigated. The condition to generate continuous porcelain bodies from powder compacts have also been studied. The results are presented below.

EXPERIMENTAL

Starting Materials – The dental porcelain powder was provided by Degussa-Ney Dental Inc., Bloomfield, CT. The chemical composition of the porcelain is confidential; however, it is within 5% of the nominal composition of the Weinstein patent [3], which has the following composition (wt%): 63.40% SiO_2, 16.70% Al_2O_3, 1.50% CaO, 0.80% MgO, 3.41% Na_2O, and 14.19% K_2O. The typical morphology and microstructure of the porcelain powder are shown in Figure 1 and its main properties are summarized in Table 1. It can be seen that the as-received dental porcelain material is composed of two phases, one being the feldspar-based glass matrix and the other the crystalline leucite ($K_2O{\cdot}Al_2O_3{\cdot}4SiO_2$). Leucite has a high coefficient of thermal expansion (CTE) and raises the inherently low CTE of the feldspathic glass matrix to match that of the PFM dental alloys, typically 13.8 ~ 16.9 x 10^{-6} K^{-1}. The CTE is one of the most critical issues regarding dental restorations because the potential for micro-cracking due to mismatch in CTE between the dental alloy and porcelain cannot be overlooked. From Table 1 it can be seen that the CTE of the dental porcelain is close to that of the dental alloy (13.8 ~ 16.9 x 10^{-6} K^{-1}).

Table 1. Properties of Dental Porcelain Powder

Particle Size	Particle Shape	Glass Transition Temperature	Softening Point	Coefficient of Thermal Expansion
1 – 50 microns	Angular	~ 580^0C	~ 650^0C	14.4~14.9 x 10^{-6} K^{-1}

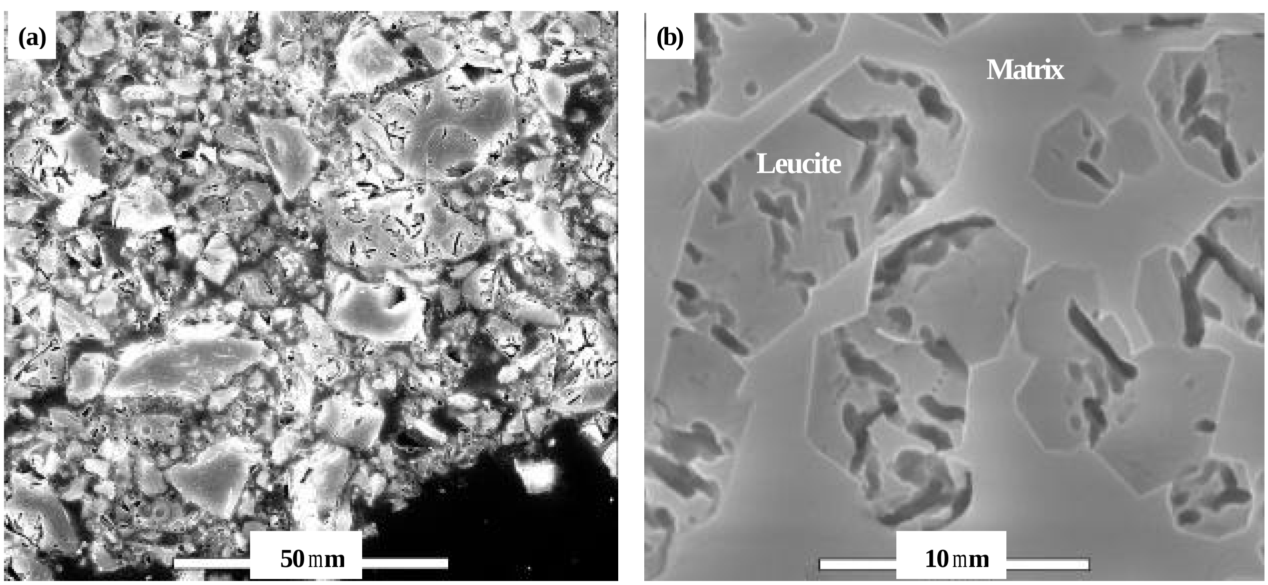


Figure 1. The as-received dental porcelain powder contains crystalline leucite before PFM and MMLD process: (a) an overview of several porcelain particles, and (b) the microstructure of the porcelain powder, showing leucite particles dispersed in the feldspathic glass matrix.

Laser Densification Process - An integrated SFF system was used for the laser densification process during which a continuous wave laser beam was emitted from a 50W CO_2 laser, and steered into a atmosphere-controlling chamber by a series of motion-controlled mirrors, hitting and sintering the desired areas of the powder bed surface. The whole sintering process was closed-loop-temperature controlled through a pyrometer by comparing the pyrometer reading with a user-defined target surface temperature. The effect of the target surface temperature on the quality of laser densified porcelain bodies was evaluated. The dependence of the quality of laser densified porcelain bodies on green density of powder compacts was also investigated by comparing the results obtained from a dry powder bed and a dried powder paste. The dry powder bed was a loose powder compact. However, the paste was prepared by dispersing the porcelain powder in de-ionized water, then delivered on a steel substrate, and finally dried on an electric plate before laser densification.

Microstructure Characterization – Densified porcelain bodies were mounted with epoxy and finish-polished with one-micron diamond suspension. The polished coupons were subsequently etched in 1% hydrofluoric (HF) acid for 20 seconds and then gold sputter-coated before characterized using an environmental scanning electron microscope (PHILIPS ESEM 2020), or examined using a digital optical microscopy. The x-ray energy-dispersive spectrometry (EDS) was used to evaluate the chemical composition.

RESULTS AND DISCUSSION

Effects of the Green Density of Porcelain Powder Compacts – Typical microstructures of laser densified porcelain bodies using a dry powder bed and a dried powder paste are shown in Figure 2. It is obvious that the densified porcelain body derived from the dried powder paste has a better quality than that derived from the dry powder bed. Specifically, the dried powder paste has led to smooth surfaces at both the top and bottom of the densified body, whereas the dry powder bed has resulted in the densified part that contains substantial amounts of porosities and voids. The marked difference is believed to be due to the high green density achieved via the dried powder paste. Thus, it can be concluded that the high powder packing density in the dried paste is beneficial for obtaining laser-densified bodies with good surface finish and low porosities.

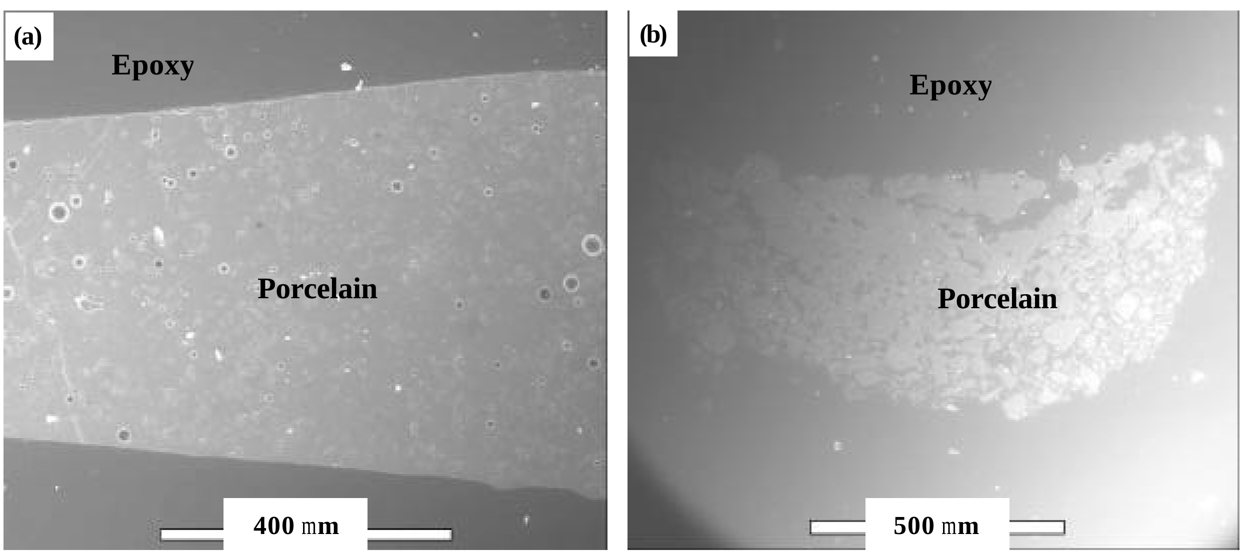


Figure 2. Typical morphology of laser processed porcelain bodies from (a) a dried powder paste and (b) a dry powder bed.

Temperature Dependence of the Quality of Densified Bodies – The effect of the laser sintering temperature was evaluated by comparing the parts laser processed with single line scanning under different target surface temperatures. Shown in Figure 3 are the overall morphology and microstructures of the laser densified bodies using different target surface temperatures. The top micrographs in Figures 3(a), (b) and (c) are the microstructures before etching and the bottom micrographs after etching. The micrographs before etching can reveal the presence of porosities and microcracks with relative ease, whereas the micrographs after etching can show the morphology and size of the crystalline leucite in the densified bodies. The dark spots emerged after etching in the bottom micrographs of Figures 3(a), (b) and (c) are the images of leucite particles. The morphology of these leucite particles can be revealed clearly under high magnifications, as shown in Figure 3(d). Note that the morphology of leucite particles is similar to that in the as-received porcelain powder (Figure 1b).

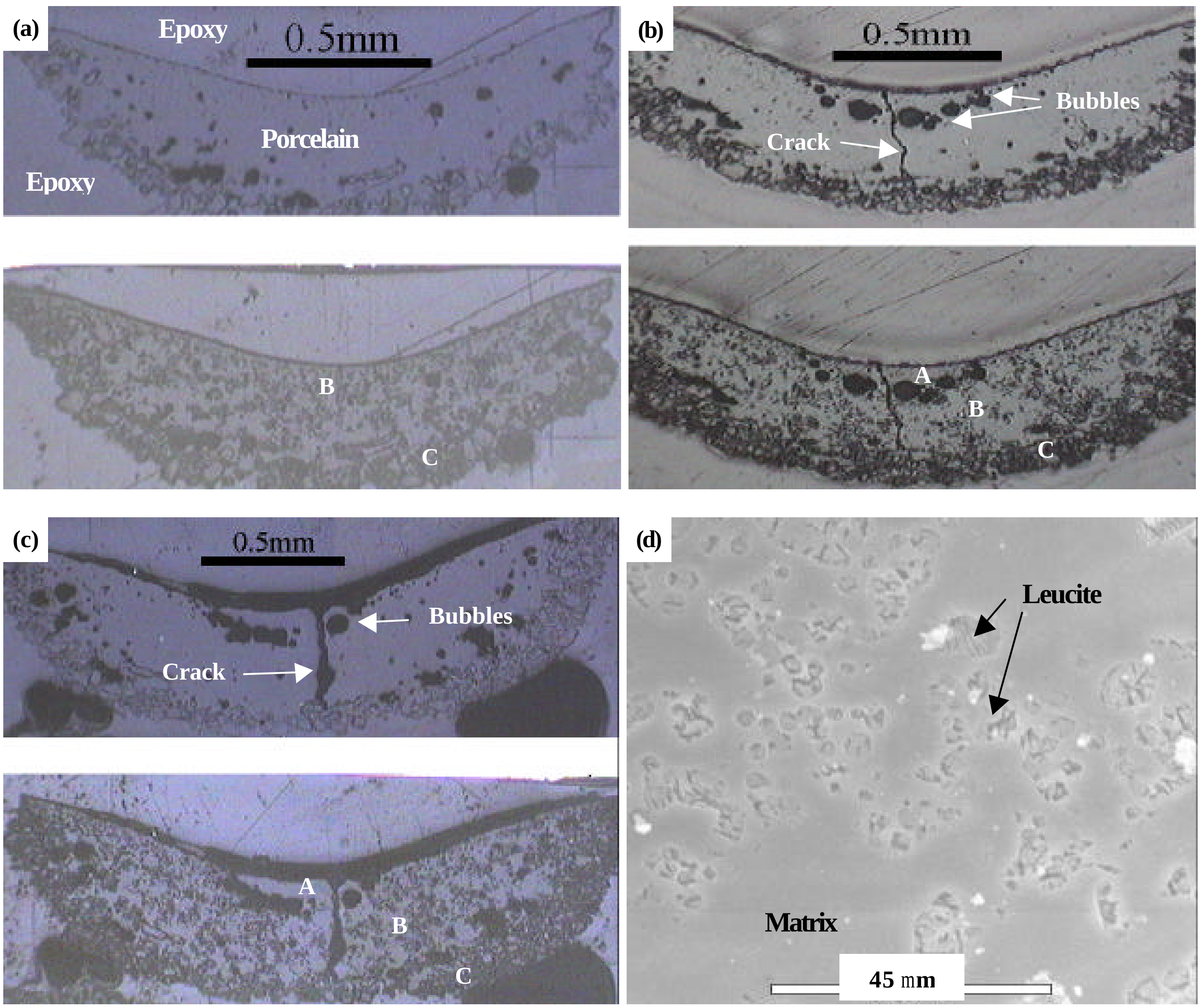


Figure 3. Morphology of laser densified porcelain bodies with scanning speed 0.24 mm/s and the target surface temperature of (a) 900°C, (b) 1000°C, and (c) 1050°C. The top micrographs of (a), (b) and (c) are obtained before etching and the bottom micrographs after etching. (d) is a high magnification micrograph of Zone B shown in (a).

A close examination of Figure 3 reveals that a laser processed body can normally be divided into three distinct zones, A, B and C. Furthermore, the processed body may contain macro-cracks and bubbles as marked in Figures 3(b) and (c). Zone A is the region that appears featureless even after etching (Figure 4), suggesting that this zone does not contain leucite particles. Zone B is the region where leucite particles are finely dispersed in the feldspathic glass matrix (Figure 3d), and it has a similar microstructure as the as-received porcelain powder (Figure 1b). Zone C is the area with little laser sintering.

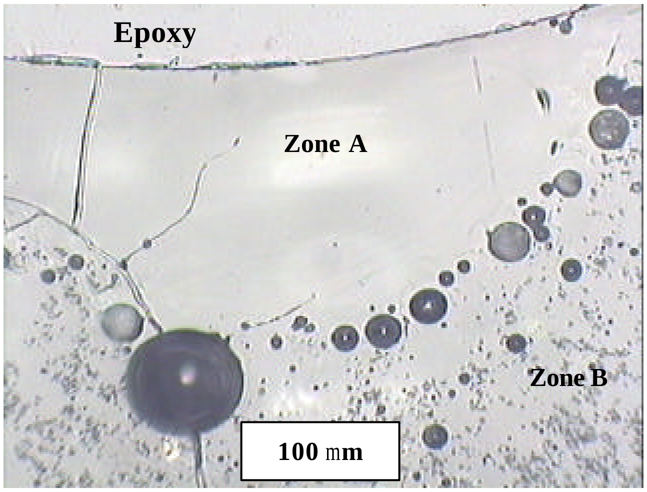
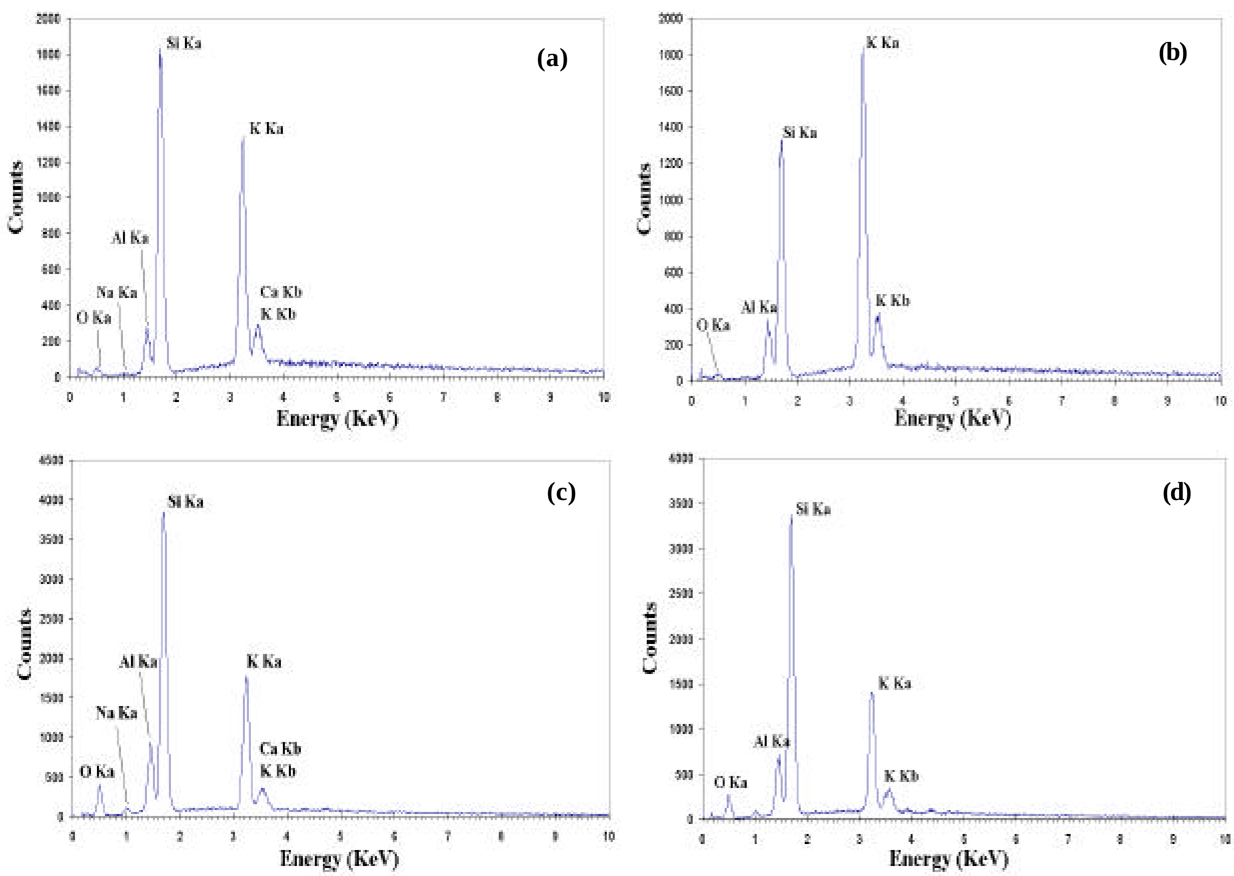


Figure 4. The typical appearance of Zone A.

It is noted that Zone A appears at the location where the laser beam center has traveled through. Thus, Zone A has been exposed to a much higher temperature than the surrounding area because of the Gaussian power distribution of the laser beam. In order to understand the appearance of Zone A, the various zones in Figure 3 have been examined using the energy-dispersive spectrometry. The results are shown in Figure 5. For comparison, the EDS results of the traditional PFM dental body feldspathic matrix and leucite precipitates are also included in the figure.

As expected, the EDS of the traditional PFM body shows that the glass matrix contains lower potassium concentration than the leucite particles [3,4]. Furthermore, the matrix contains sodium and calcium both of which are not found in the leucite particles, again consistent with the porcelain and leucite compositions [3,4]. A comparison between the traditional PFM porcelain body (Fig. 5(a) and (b)) and the laser densified body (Fig. 5(c) and (d)) indicates that both the matrix and leucite in the laser processed body have lower potassium concentrations than the counterparts in the traditional PFM porcelain body. This suggests that some of the potassium, a volatile element, has evaporated in the laser densification process. The loss of the potassium is especially severe at the locus of the laser beam center where the temperature is the highest. It is also noted that Zone A always appears at the locus of the laser beam center (Figure 3) and has the lowest potassium concentration (Figure 5(e)) among all the regions measured in this study. It has been well established that the feldspathic porcelain needs a minimum amount of potassium to form leucite precipitates [5,6]. Thus, the disappearance of leucite particles in Zone A is due to the substantial loss of the potassium element in that particular area. Since the appearance of Zone A is associated with the loss of potassium, Zone A is then expected to increase with increasing the target surface temperature. This is exactly what we observed in the experiment. As shown in Figure 3, there is no Zone A when the target surface temperature is low (e.g., 900^0C). Zone A appears when the target surface temperature is set at 1000^0C and becomes larger when the target surface temperature is 1050^0C.

Macro-cracks, Bubbles and Distortion – The integrity of the densified porcelain body is one of the important criteria for determining what laser processing conditions should be. Figure 3 reveals three major defects that degrade the integrity of the laser densified porcelain body. These are (i) macro-cracks, (ii) bubbles and (iii) distortion. Macro-cracks appear only when the laser processing temperature is high (e.g., 1000 and 1050^{0}C of the target surface temperature). Further, the higher the target surface temperature is, the larger the macro-crack is, as exhibited by Figures 3(b) and (c). These results suggest that macro-cracks are caused by thermal shock. Higher laser processing temperatures lead to higher temperature gradients between the top and bottom of the densified porcelain body, thereby producing more severe thermal shock and larger macro-cracks. Thus, to avoid macro-cracks the target surface temperature should not be too high. 900^{0}C appears to be appropriate for the porcelain used in this study. Preheating of the substrate may be a useful approach to combat the macro-cracking problem especially when a high target surface temperature is required for densification of the porcelain body.



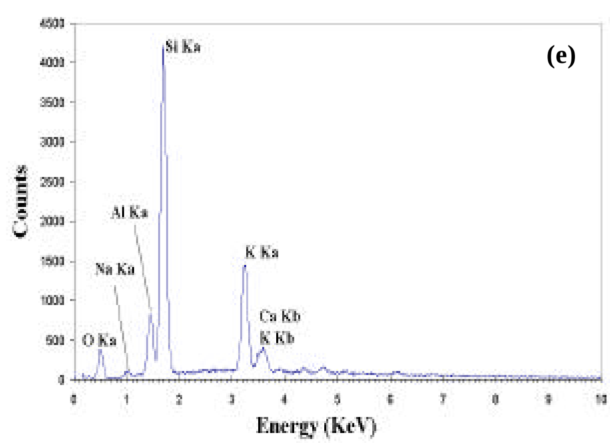


Figure 5. EDS of (a) the feldspathic glass matrix and (b) leucite precipitates in the traditional PFM porcelain body, (c) the feldspathic glass matrix and (d) leucite precipitates in Zone B of the laser densified porcelain body, and (e) Zone A in Figure 4.

The appearance of large bubbles in the densified porcelain body also appears to be associated with high laser processing temperatures. As shown in Figures 3(b) and (c), most of the large bubbles gather around Zone A. The formation of the bubble clusters around Zone A is clearly undesirable and efforts are currently under way to identify the exact mechanism and prevent its formation.

Finally, it is noted that regardless the laser processing temperature, the laser densified porcelain body always exhibits substantial distortion, showing a severe concave upward warping (Fig. 3). Our recent thermal and stress analyses of laser processing using finite element modeling [7] have indicated that such distortion is caused by the temperature gradient between the top and bottom surfaces of the densified body. One of the methods to eliminate warping is having the same temperature for the top and bottom surfaces [7]. Although this is hard to achieve, warping can be minimized by preheating of the substrate so that the temperature gradient can be reduced.

Continuity of Laser Densified Porcelain Bodies – Another important issue for laser densification of porcelain bodies is whether or not laser densified bodies are continuous macroscopically. The continuity issue covers a wide range of laser scanning conditions. The simplest case is the continuity issue when a single-track laser scan is performed. The more complicated cases are the continuity issues when multi-track laser scans to form a solid plate or a single-track laser scan to form a ring, a rectangle or a triangle is needed. The present study shows that continuity is not a problem for a single-track laser scan. However, continuity becomes a problem when fabrication of a ring-shaped porcelain body is desired. Figure 6 shows a porcelain ring produced using a single-track laser scan with the end point of the scan overlapping with the starting point. It can be seen that there is a groove near the starting point, i.e., the ring is not smoothly connected at the starting point even though the end point of the scan overlaps with the starting point. The appearance of the groove is related to the volume shrinkage of the porcelain powder changing to a dense body and spheroidizing of the molten porcelain under the surface tension during the laser melting and solidification process. The attempt to eliminate the groove by overlapping the starting point of the scan with the end point by a 45 degree additional scan was not successful. Efforts are currently under way to identify a suitable approach to eliminate the groove.

Concluding Remarks

The present study has investigated the densification behavior of dental porcelain powders in response to a moving laser source. Based on this investigation, the following conclusions can be made.

(a) The high powder packing density is beneficial for obtaining laser densified bodies with good surface finish and low porosities.
(b) The potassium content in the laser densified porcelain body is lower than that of traditional PFM body because of the volatile nature of potassium and the high local temperature at the center of the laser beam.
(c) When the target surface temperature is higher than 1000°C, the leucite content at the locus of the laser beam center is substantially reduced. As a result of this large loss of potassium, a leucite-free zone is formed. The size of the leucite-free zone increases with increasing the target surface temperature.
(d) Thermal shock-induced macro-cracks appear when the laser processing temperature is too high (e.g., the target surface temperature greater than 1000°C). The higher the target surface temperature is, the more severe the cracking phenomena.

(e) Regardless the laser processing temperature, the laser densified porcelain body always exhibits substantial distortion, showing a severe concave upward warping. Such distortion is caused by the temperature gradient between the top and bottom surfaces of the densified body.
(f) The macroscopic continuity of laser densified porcelain bodies may become a problem when fabrication of a porcelain body with a shape having macroscopic connectivity such as rings and triangles are performed.

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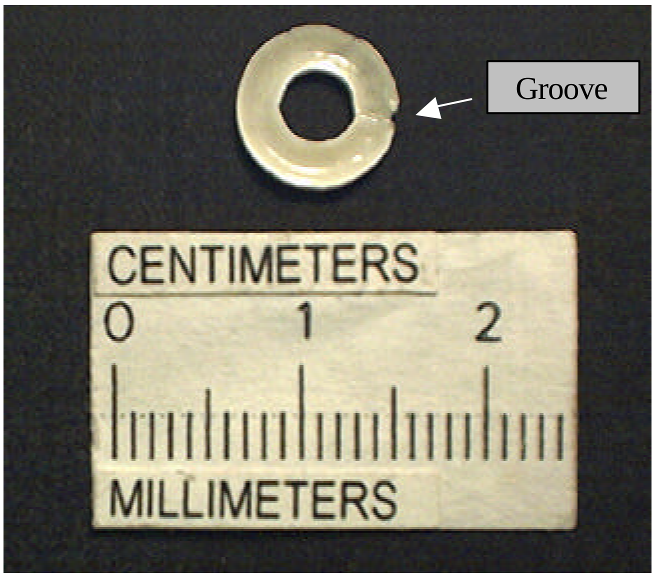

Figure 6. Top view of a ring-shaped porcelain body fabricated using a single-track laser scan with the end point of the scan overlapping with the starting point, showing the presence of a groove near the starting point.

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