Evaluation of Microstructure and Properties for Multi-Materials Laser Densification of Dental Restorations

Xiaoxuan Li*, James E. Crocker*, Erik Geiss*
Leon L. Shaw*, Harris L.Marcus*, and Thomas B. Cameron**

*Institute of Materials Science
University of Connecticut
Storrs, CT

**Degussa-Ney Dental Inc.
Bloomfield, CT

ABSTRACT

Traditional dental restorations are produced by the porcelain-fused-to-metal (PFM) process, in which a dental restoration is cast from a metallic alloy and then covered with dental porcelains by several firing processes, which is both labor intensive and expensive. In this paper, the feasibility of dental restorations is investigated using a multi-materials laser densification (MMLD) process. To evaluate the effectiveness of the MMLD process, nickel powders and commercial dental porcelain powders are laser densified using YAG and CO_2 lasers respectively. Effects of processing parameters, e.g. laser scanning rate and target temperature, are evaluated and the microstructure of processed nickel and porcelain materials are characterized for the optimization of laser densification. Results indicate that densities of laser processed nickel and dental porcelain are strongly dependent of processing parameters. Fully dense layers are achievable with proper processing conditions.

KEY WORDS: Multi-materials laser densification, dental restorations, solid freeform fabrication.

INTRODUCTION

There are between 10,000 and 15,000 dental laboratories in the US and a majority of these laboratories use porcelain-fused-to-metal (PFM) restoration for permanent fixed prosthodontics. There are several steps in PFM restoration as shown in Fig. 1, which shows that the traditional PFM process is both time consuming and labor intensive work. To address this problem, we have investigated the feasibility of using solid freeform fabrication (SFF) to construct dental restorations. The approach we have proposed is point-by-point and layer-by-layer freeform dental restoration through laser densification of multiple dental alloys and porcelains, which will be termed hereafter as multi-materials laser densification (MMLD). MMLD shares many attributes with selective laser sintering (SLS) [1] and direct metal deposition (DMD) [2]. In SLS, a layer of powder is delivered on top of the substrate (or a previously processed layer) before a laser beam scans the powder layer selectively [1]. In DMD, a laser generates a melt pool on a substrate (or a previously processed layer) while the feedstock material is introduced into the laser-created pool either as powder or as wirefeed that melts and forms a metallurgical bond with the substrate [2]. The MMLD process to be developed will deal with multiple materials, and further, dental powders will be delieved and laser densified point-by-point because of the multiple materials involved in almost every single layer.

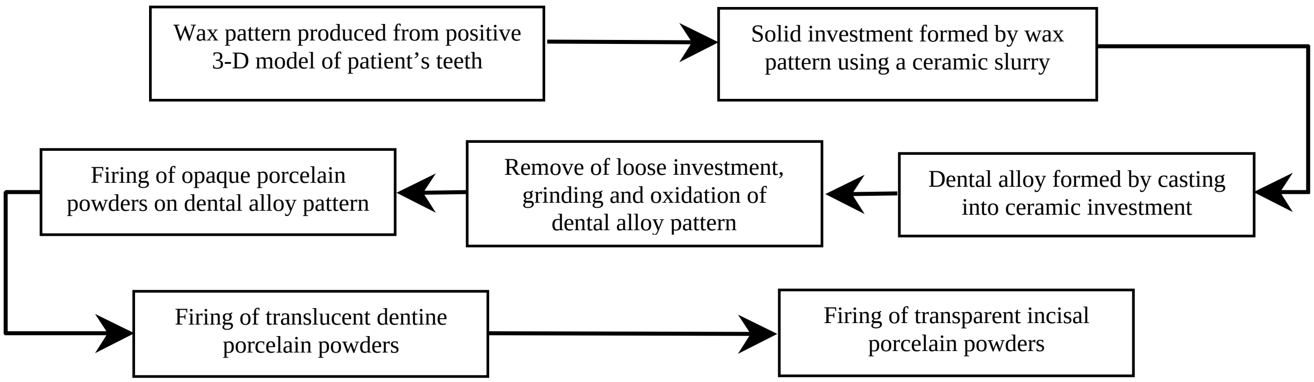


Fig. 1 Traditional PFM restoration process

As the first step in developing the MMLD process, the current study has focused on the laser densification condition for achieving fully dense bodies from dental alloy and porcelain powders.

EXPERIMENTAL

Starting Materials - Since nickel has been used as the major constituent (79%) in a commercial dental alloy, produced by Degussa-Ney Dental Inc. and widely used in the PFM restoration, a commercial nickel powder was selected as the starting metal for laser densification investigation. The physical properties of the nickel powder used are shown in Table 1. A commercial dental porcelain powder provided by Degussa-Ney Dental Inc. was chosen as the starting porcelain for investigation. Table 2 shows some properties of the selected dental porcelain.

Laser Densification Process - The laser densification was carried out in an integrated SFF system composed of a laser, a process chamber, powder delivery system, 3-D laser beam scanning control stage, vacuum and atmosphere control system, observation and recording system, and closed-loop temperature control system. Detailed description of the SFF apparatus can be found in Ref.[3]. The nickel layers were fabricated using a Nd: YAG laser in a continuous wave mode with a maximum output power of 150W and a wavelength of 1.064 microns. The dental porcelain powder layers were manufactured by a continuous wave, maximum power 50W CO_2 laser with a wavelength of 10.6 microns. The laser beam was steered into the vacuum chamber with the pressure of up to 60 millitorrs by a series of motion-controlled mirrors. The densification was performed through scanning a thick powder bed (about 10mm thick) in a linear pattern. During densification the average surface temperature of the powder under the laser beam was monitored by an emissivity-measuring infrared pyrometer and maintained by a closed-loop control system to match a user-defined target temperature.

Table 1. Nickel powder physical properties

Size	Shape	Apparent density	Melting Temperature	Purity	Coefficient of Thermal Expansion
-300 mesh	Spherical	3.6 g/cm^3	1453^{0}C	99.9%	13.3 x 10^{-6} K^{-1}

Table 2. Properties of dental porcelain powder

Size	Shape	Glass Transition Temperature	Coefficient of Thermal Expansion
1~50 microns	irregular	650^0C	$14.4 \sim 14.9 \times 10^{-6} K^{-1}$

Two laser processing parameters, i.e. the scanning rate and the target temperature, were controlled to evaluate the effect of processing conditions on the density of processed nickel and dental porcelain bodies. The combination of the various processing parameters used to form the primitive samples is summarized in Tables 3 and 4.

Characterization of Microstructure - The primitive samples obtained from these experiments were mounted using an epoxy resin, cut along the direction perpendicular to the scanning direction using a diamond saw, ground and finish polished using an 1-micron diamond suspension. For densified nickel bodies, the samples were examined using an optical microscope directly. For dental porcelain bodies, the samples were etched in 1% HF for 20 seconds, rinsed with water, and then carbon sputter-coated before examination using an environmental scanning electron microscope (PHILIPS ESEM 2020).

Table 3. Nickel powder laser densification (150W YAG) parameters

No.	Target Surface Temperature (C)	Scanning Rate (micron/second)	Beam Diameter (mm)	Number of Scanning Pass
A-1	1200	2.0	2	one
A-2	1200	3.8	2	one
A-3	1200	5.7	2	one
A-4	1200	7.6	2	one
A-5	1200	10.0	2	one
B-1	1300	2.0	2	one
B-2	1300	3.8	2	one
B-3	1300	5.7	2	one
B-4	1300	7.6	2	one
B-5	1300	10.0	2	one
C-1	1400	2.0	2	one
C-2	1400	3.8	2	one
C-3	1400	5.7	2	one
C-4	1400	7.6	2	one
C-5	1400	10.0	2	one

Table 4. Dental porcelain powder laser densification (50W CO_2) parameters

No.	Target Temperature (^0C)	Scanning Rate (micron/second)	Beam Diameter (mm)	Number of Scanning Pass
D-1	800	8	1	one
D-2	800	16	1	one
D-3	800	32	1	one
E-1	900	8	1	one
E-2	900	16	1	one
E-3	900	32	1	one

RESULTS

Microstructure of Laser Processed Nickel Bodies - The microstructure of processed nickel in all the trials is similar and can be divided into two zones, as shown in Fig. 2(a). Zone I is almost fully dense with little or no porosity (see Fig. 2b). This zone is formed via melting and solidification. Zone II is a transition region between the dense body and the initial loose powder bed, and has a microstructure with a density gradient. Presumably, this zone is formed partly through incomplete infiltration of the liquid nickel from Zone I and partly through localized powder sintering. The temperature gradient present during laser scanning leads to a gradient in the density of the powder sintered region in Zone II. Viewing from the laser scan direction, Zone I has a V-shape profile, as shown in Fig. 2(a). The investigation to identify the mechanism(s) responsible for the formation of V-shape profile is currently under way.

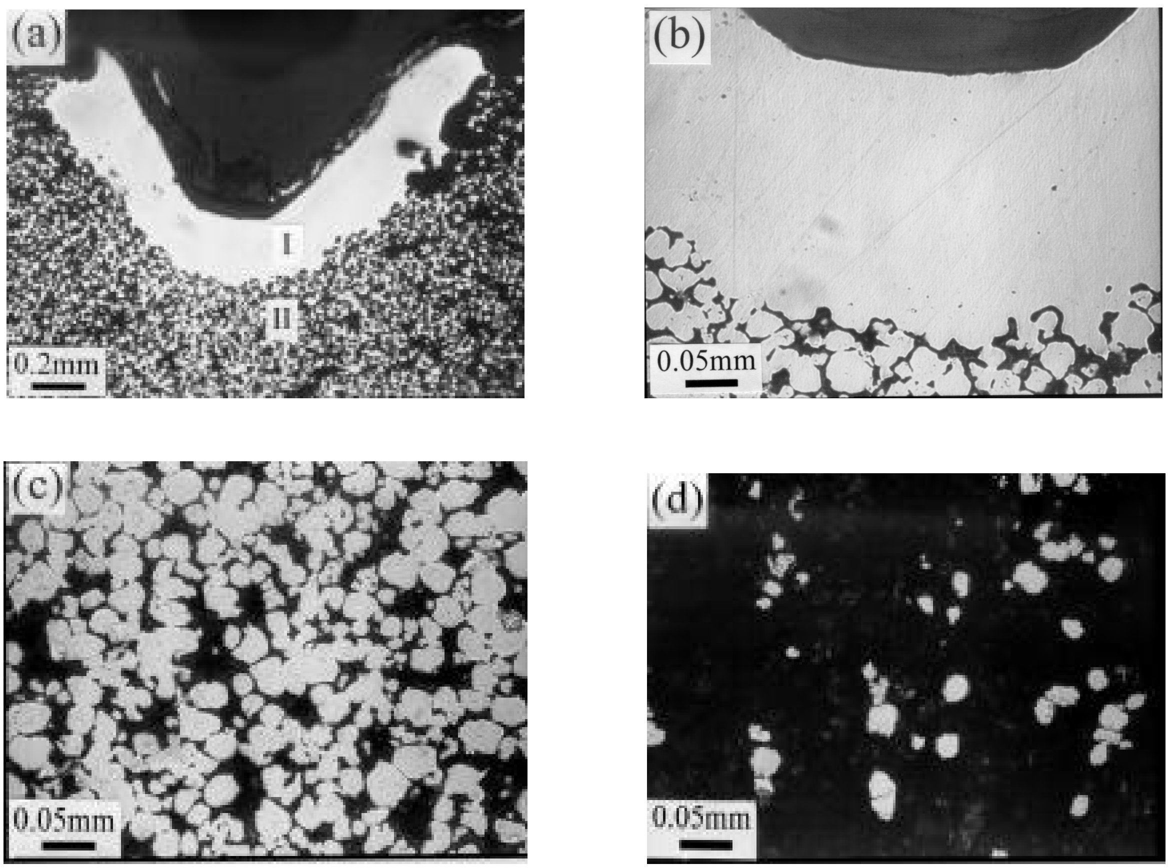


Fig. 2 The typical microstructure of a laser densified nickel layer: (a) the overall shape of the densified nickel with Zone I and II, (b) a high magnification of Zone I, (c) a high magnification of Zone II, and (d) the powder area far away from Zone I. Many powder particles in (d) have been pulled out during polishing, resulting in a very low apparent compact density. The scanning conditions are: target temperature 1400^0C, scanning rate 10 μm/s, and beam diameter 2 mm.

Microstructure of Laser Processed Porcelain Bodies - Dental PFM feldspathic porcelains contain crystalline leucite, a potassium aluminum silicate mineral ($K_2O.Al_2O_3.4SiO_2$)

with a high coefficient of thermal expansion (CTE) in order to elevate the bulk CTE of the dental porcelain to a level compatible with PFM dental alloys [4]. However, the large mismatch between the CTE of leucite and surrounding glass matrix of the porcelain creates thermal mismatch stresses that can cause microcracking around the leucite particles during the PFM firing. Fig. 3 shows a typical microstructure of a fully dense dental porcelain body after traditional firing. From Fig. 3(a) it can be seen that the overall microstructure is composed of two phases: feldspathic glassy matrix (i.e. the featureless region except microcracks) and colonies of leucite crystals. Fig. 3(b) and (c) show the detail of a cluster of leucite particles with different magnifications, and on the top and bottom of Fig. 3(b) there are microcracks. Fig. 3(d) reveals the morphology of the glassy matrix with several microcracks. When feldspathic dental procelain is cooled, the leucite crystals contract more than the surrounding glassy matrix, leading to the development of radial tensile stresses around the leucite particles and thus microcracks within and around the crystals, as shown in Fig. 3(b).

The microstructure of a laser densified dental porcelain body is shown in Fig. 4. It can be seen that the top region is denser than the bottom region because of the temperature gradient. The fully dense area has almost the same microstructure as that shown in Fig. 3(b) and (c). Finely dispersed small crystalline leucite particles lead to reduced radial tensile stresses around the leucite particles; thus less microcracks are found in Fig. 4(b) and (c) than in Fig. 3.

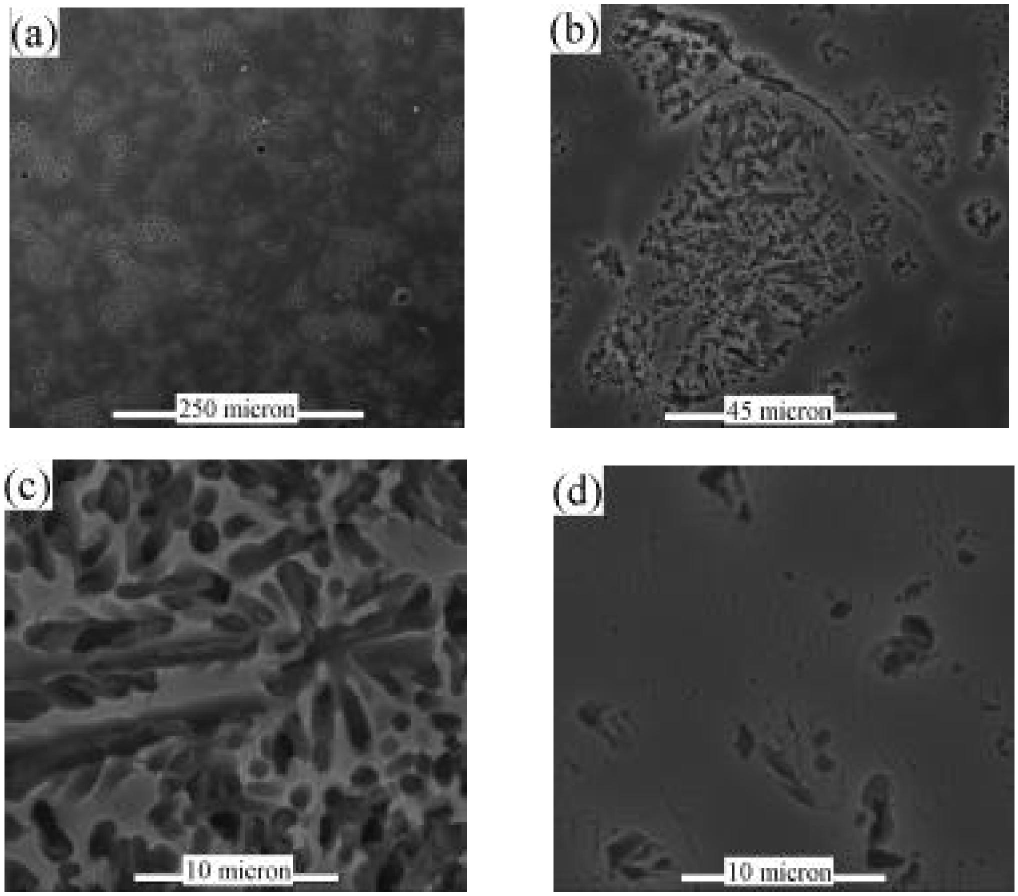
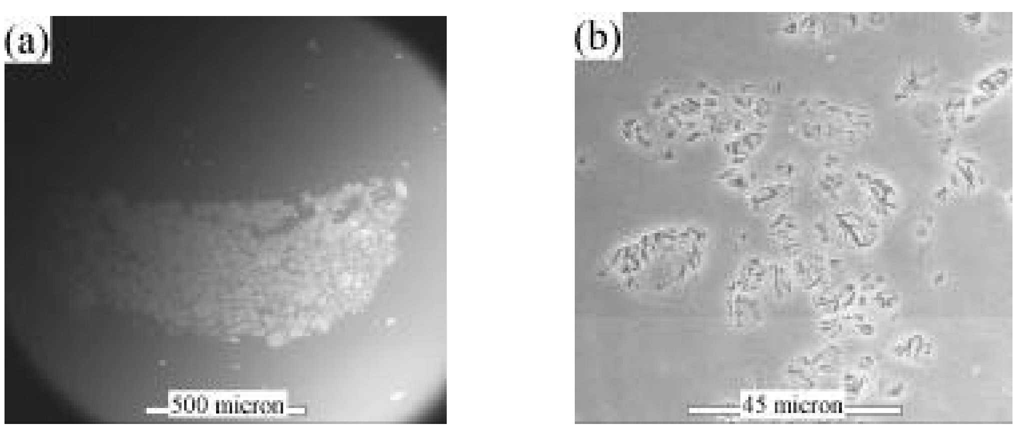


Fig. 3 The typical microstructure of dental porcelain body after traditional firing: (a) the general view; (b) and (c) details of a cluster of crystalline leucite; and (d) the microstructure of feldspathic glassy matrix.



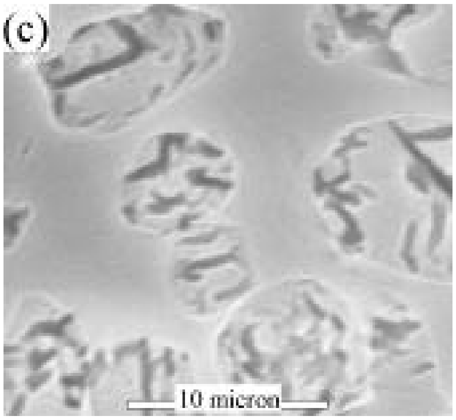


Fig. 4 Microstructure of laser densified dental porcelain bodies: (a) overall profile of a laser processed layer, (b) and (c) colonies of leucite particles at different magnifications. The processing parameters: target temperature 900^0C, scanning speed 32 micron/sec.

Effects of Processing Parameters on Densified Nickel Bodies - The size of Zone I is found to be dependent of the scanning rate and target temperature. For a given target temperature, the width and depth of the densified nickel layer decrease with an increase in the scanning rate. The relationship is shown in Fig. 5. For a given scanning rate the dimension of the densified body increases with increasing the target temperature, as shown in Fig. 6. These experimental results are consistent with the expectation on the basis of changes in the heating time and energy input with variation of the scanning rate and target temperature.

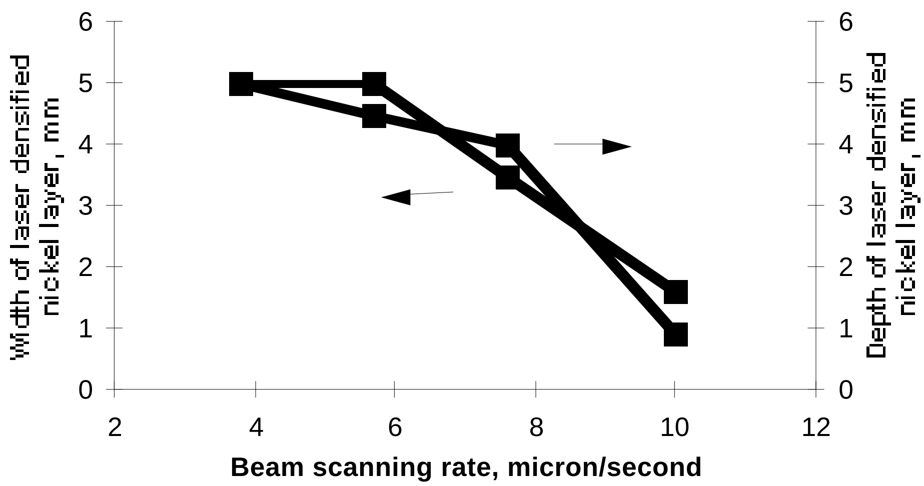


Fig. 5 The depth and width of laser densified nickel bodies as a function of the scanning rate with a target temperature 1400^0C.

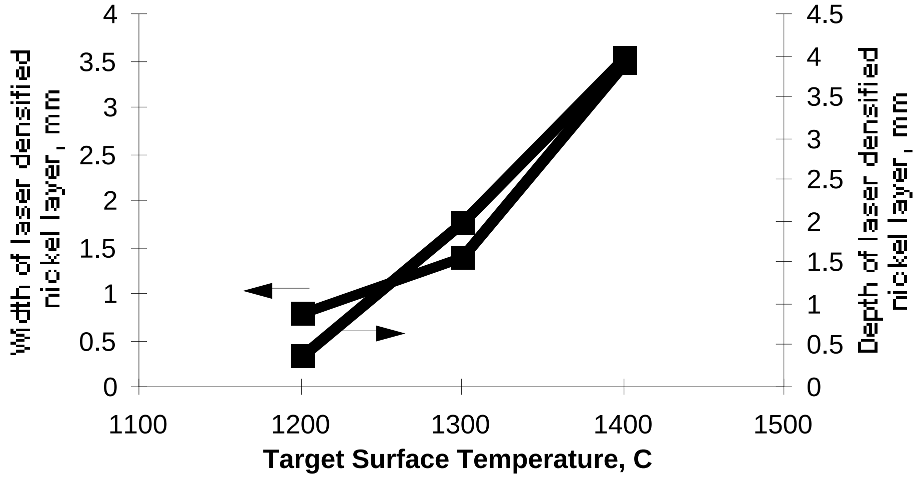


Fig. 6 The depth and width of laser densified nickel bodies as a function of the target temperature at a scanning rate of 7.6 micron/second.

Effects of Processing Parameters on Densified Dental Porcelain Bodies - The relationship between the dimension of laser densified dental porcelain bodies and the processing parameters are similar to that of nickel bodies, as shown in Fig. 7 and Fig. 8.

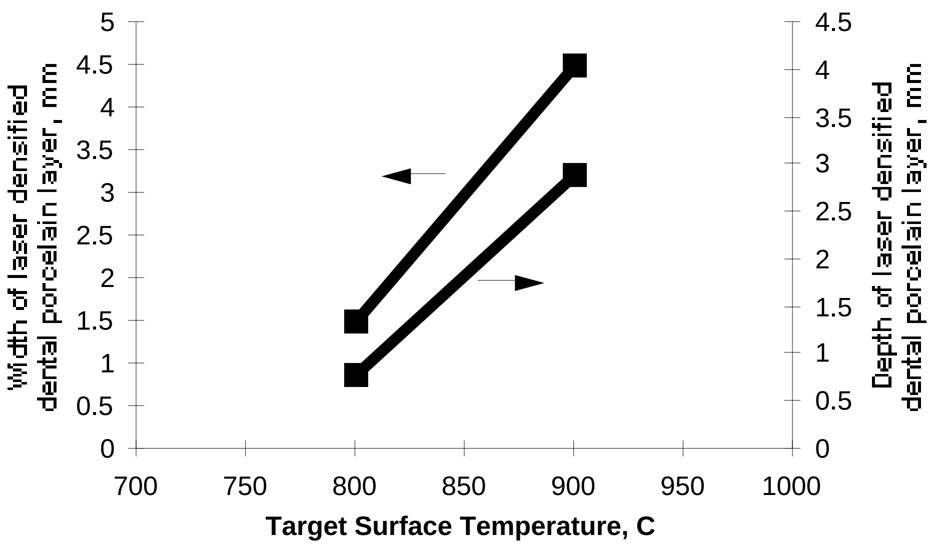


Fig. 7 The dimension of laser densified dental porcelain bodies as a function of target temperature at a scanning rate of 16 micron/second.

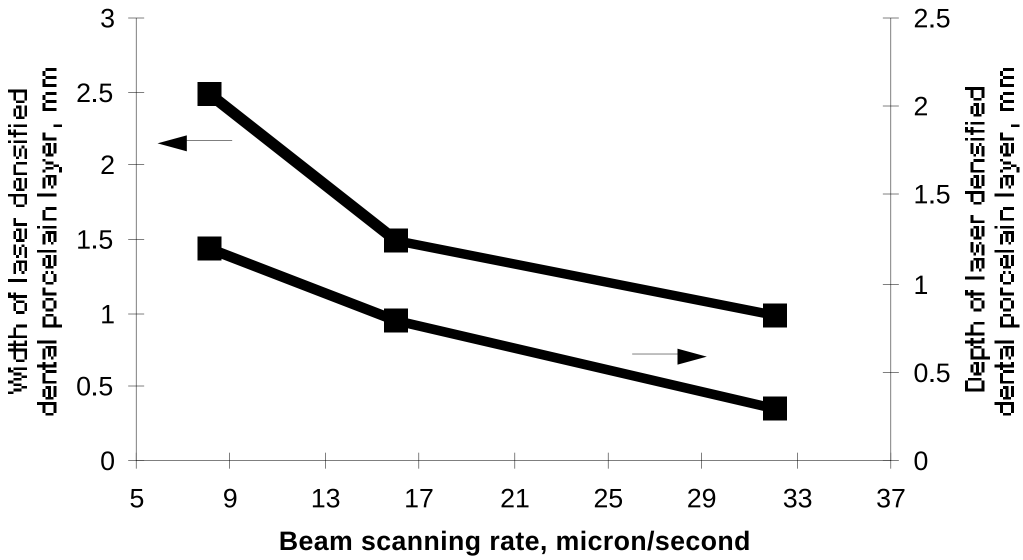


Fig. 8 The depth and width of laser densified dental porcelain bodies as a function of the scanning rate at a target temperature of 800^0C.

CONCLUSIONS

(a) Morphology and size of the laser densified nickel and dental porcelain bodies are dependent on scanning conditions.
(b) The size of crystalline leucite particles is smaller in the laser processed porcelain bodies than that processed through traditional firing. Accordingly, less microcracks are found in the laser processed porcelain bodies.
(c) Fully dense nickel body is achievable under appropriate processing conditions.

Acknowledgement – The authors gratefully acknowledge financial support provided by the National Science Foundation under Grant No: DMI-9908249.

REFERENCES

1) J. J. Beaman, J. W. Barlow, D. L. Bourell, R. H. Crawford, H. L. Marcus and K. P. McAlea, “Solid Freeform Fabrication: A New Direction in Manufacturing”, Kluwer Academic Publishers, MA (1997).
2) J. Mazumder, J. Choi, K. Nagarathnam, J. Koch and D. Hetzner, “The Direct Metal Deposition of H13 Tool Steel for 3-D Components”, JOM, 49 [5] 55-60 (1997).
3) L. C. Sun, K. J. Jakubenas, J. E. Crocker, S. Harrison, L. Shaw and H. Marcus, “In-Situ Thermocouples in Macro-Components Fabricated using SALD and SALDVI Techniques: II. Evaluation of Processing Parameters”, Materials and Manufacturing Processes, 13 [6] 883-907 (1998).
4) J. R. Mackert, Jr. and A. L. Williams, “Microcracks in dental porcelain and their behavior during multiple firing”, J. Dent. Res., 75[7] 1484-1490 (1996).