

Solid Freeform Fabrication of Artificial Human Teeth

Jiwen Wang¹, Leon L. Shaw¹, Anping Xu², Thomas B. Cameron³

¹ Department of Materials Science and Engineering, University of Connecticut
Storrs, CT 06269, USA

² School of Mechanical Engineering, Hebei University of Technology, Tianjin 300130,
P. R. China

³ Dentsply Ceramco, Burlington, NJ 08016, USA

Abstract

In this paper, we describe a solid freeform fabrication procedure for human dental restoration via porcelain slurry micro-extrusion. Based on submicron-sized dental porcelain powder obtained via ball milling process, a porcelain slurry formulation has been developed. The formulation developed allows the porcelain slurry to show a pseudoplastic behavior and moderate viscosity, which permits the slurry to re-shape to form a near rectangular cross section. A well-controlled cross-section geometry of the extrudate is important for micro-extrusion to obtain uniform 2-D planes and for the addition of the sequential layers to form a 3-D object. Human teeth are restored by this method directly from CAD digital models. After sintering, shrinkage of the artificial teeth is uniform in all directions. Microstructure of the sintered teeth is identical to that made via traditional dental restoration processes.

Keywords: Solid freeform fabrication; Micro-extrusion; Dental restoration; Artificial Teeth

Introduction

Tooth loss has many causes: decay, injuries and gum disease are the most common ones. Missing teeth may make people feel self-conscious and make eating and speaking more difficult. Sometimes the strain on the remaining teeth invites more tooth loss. In order to prevent these problems, dental restoration is needed. Dental restoration includes bridges, dentures, crowns, and implants. There are currently more than 10,000 dental laboratories in the US and a majority of these laboratories use porcelain-fused-to-metal (PFM) restoration for permanent fixed prosthodontics. However, PFM restoration is a very time consuming and labor intensive work. For example, it normally takes 2-4 weeks to make a three-unit bridge and labor costs account for about 90% of the final cost.

Solid freeform fabrication (SFF) is an automated manufacturing process that builds three-dimensional complex-shaped structures layer-by-layer directly from CAD data without part-specific tooling and human intervention [1-3]. Human dental parts fabricated via solid freeform fabrication could have potential to largely reduce the processing time and the final cost. In this paper, artificial teeth fabricated via a micro-extrusion-based SFF process is described. The result presented here demonstrates that SFF has the potential to reduce the processing time as well as the cost for dental restoration.

Experimental Procedure

The dental porcelain powder was provided by Degussa-Ney Dental Inc., Bloomfield, CT. The chemical composition of the porcelain is within 5% of the nominal composition of the Weinstein patent [4], which has the following composition (wt%): 63.40% SiO₂, 16.70% Al₂O₃, 1.50% CaO, 0.80% MgO, 3.41% Na₂O, and 14.19% K₂O. The as-received powder has angular shapes and their equivalent particle sizes range from 1 to 50 micrometers with the particle distribution as shown in Table 1. Large particles are not suitable for preparing stable suspensions and slurries, and are especially undesirable for micro-extrusion where the width (or diameter) of the extrudate is smaller than 1 mm. Thus, to reduce the particle size of the porcelain powder to sub-micrometers, ball milling process was conducted at NORTON low speed mill machine in a ceramic jar with Al₂O₃ balls as grinding media. After milling, the powder was dispersed in de-ionized water and the submicron-sized particles were separated from coarse particles (> 1 μm) through sedimentation. Only the submicron-sized particles were used to prepare the slurries. Measured by Dynamic Light Scattering (DSL), sub-micron particles had an average particle size of 0.48 μm with the particle size distribution also shown in Table 1.

Table 1. Experimentally determined powder size distributions and surface area

Powder	Distribution percentiles			Surface area (m ² /g)
	≤ 10%	≤ 50%	≤ 90%	
As-received	2 μm	5 μm	15 μm	2.82
After milling	0.3 μm	0.5 μm	0.7 μm	16.33

The porcelain slurries were prepared using de-ionized water as the solvent with a solid loading of 40 – 45 vol %. Uniform and stable slurries were obtained through high speed milling using a SPEX machine for 5 – 10 min to break down agglomeration of the submicron-sized dental powder particles. To obtain optimal extrusion properties, the pH value of the slurry was adjusted to be close to 7.5. This resulted in a slurry with pseudoplastic behavior and moderate viscosity, which was critical for obtaining a near-rectangular cross section of the extrudate. The rheological analysis was conducted using a Brookfield DV II digital viscometer. The zeta potential of the slurries was measured at the National Science Foundation Engineering Research Center for Particle Science and Technology in the University of Florida using a Zeta-Reader instrument. The pH adjustment was made using 1.0N HNO₃ (Fisher Scientific), and the pH value of the slurry was measured using a Denver basic pH meter. Particle size distribution was obtained using dynamic light scattering (BI-200SM goniometer from Brookhaven Instruments Corporation, 2.5 W Ar laser light source, Model Innova 70-3, from Coherent). Mean specific surface area was determined by means of BET analysis (NOVA 1000, QUANTACHROME Corporation).

The porcelain slurry was extruded using a SFF micro-extrusion machine designed and constructed at the University of Connecticut (Figure 1). The machine consists of four major components: (1) an electric cylinder as the micro-extruder; (2) a precision force sensor in front of micro-extruder for monitoring the extrusion pressure; (3) a positioning system containing a X-Y table and Z stage; and (4) a syringe for housing the porcelain slurry with changeable nozzles from 100 to 800 μm in diameter; the syringe is pressed by the micro-extruder with pressure transferring through a solid pipe containing water. The slurry was extruded onto a Cu substrate.

The motion of X, Y, and Z axes and the extrusion of the micro-extruder were controlled by a custom-designed computer program (teeth 1.0) which allowed the reading and slicing of STL-format files, and sent the position and extrusion control signals to the machine through a Galil DMC-1800 multi-axis motion control card.

After extrusion, green parts were dried in air for 24 hrs at ambient temperature, followed by sintering in an EPD high temperature furnace (The Kanthal Corporation) at 950°C for 5 min. Macro-photos were recorded using a Sony DSC-P5 digital camera and a Panasonic industrial CCD camera, whereas microstructures of the sintered artificial teeth were characterized using an optical microscope (Nikon METAPHOT) and an environmental scanning electron microscope (Philips ESEM 2020).

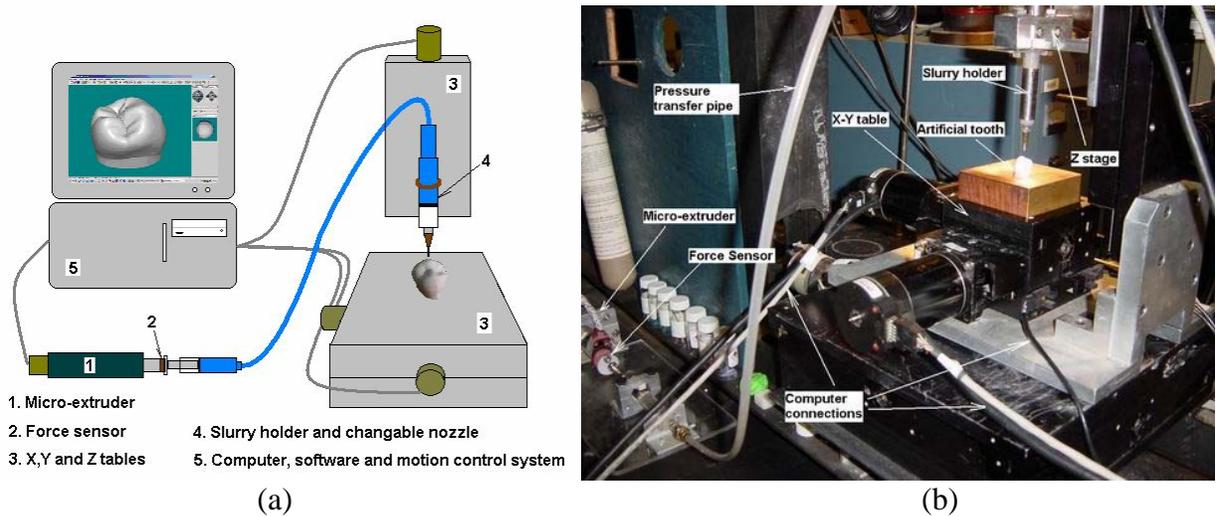


Figure 1. (a) Schematic of the SFF micro-extrusion machine and (b) the photo of the machine.

Results and Discussion

Rheological/Extrusion Properties

Figure 2a shows the zeta potential of the dental porcelain powder as a function of the pH value [7]. It indicates that the iso-electric point (i.e.p.) of the dental porcelain powder is at about 5.0, which is between the i.e.p. of Al_2O_3 (8.7) and SiO_2 (2.0). It can be inferred from Figure 2a that slurries with the pH value far away from i.e.p ($\text{pH} < 2.0$ or $\text{pH} > 9.0$) should be relatively stable. This is because the surfaces of particles with these pH values are mainly positively charged (for $\text{pH} < 2.0$) or negatively charged (for $\text{pH} > 9.0$). When the pH value of the dental porcelain slurry is close to $\text{pH} = 5.0$, the electrostatic repulsion will become weak and the particle interactions are dominated by long-rang van der Waals forces, which drives the particles into a highly flocculated state with a significant increase in the slurry viscosity (Figure 2b).

To prepare suitable slurries for extrusion, we first mixed 40 vol% ~ 45 vol% porcelain micro-sized powder with de-ionized water. The original pH value of the slurry after mixing was 11.5, which indicated the slurry was well dispersed and had the dilatant property. The dilatant

property is not suitable for dental restoration because we need the slurry to “freeze” immediately after it flows out of the nozzle. In contrast, pseudoplastic shear thinning behavior is preferred because this allows the slurry to “freeze” when it flows out of the nozzle [5]. To achieve this, we adjusted the pH value of the slurry towards the iso-electric point (pH = 5.0). The porcelain slurry began to show pseudoplastic behavior when the pH value was lowered to 9.5, but at this point the shear thinning behavior was not strong enough to “freeze” the extrudate immediately. Ball extrudates were easily formed at this point [Fig. 3(a)]. When the pH value was continuously adjusted down to 7.5, the viscosity of the slurry increased. The shear thinning property at this pH value became much stronger as the extrudate “froze” immediately to form a rod [Fig. 3(b)]. Such a condition is ideal for building 2-D slurry planes.

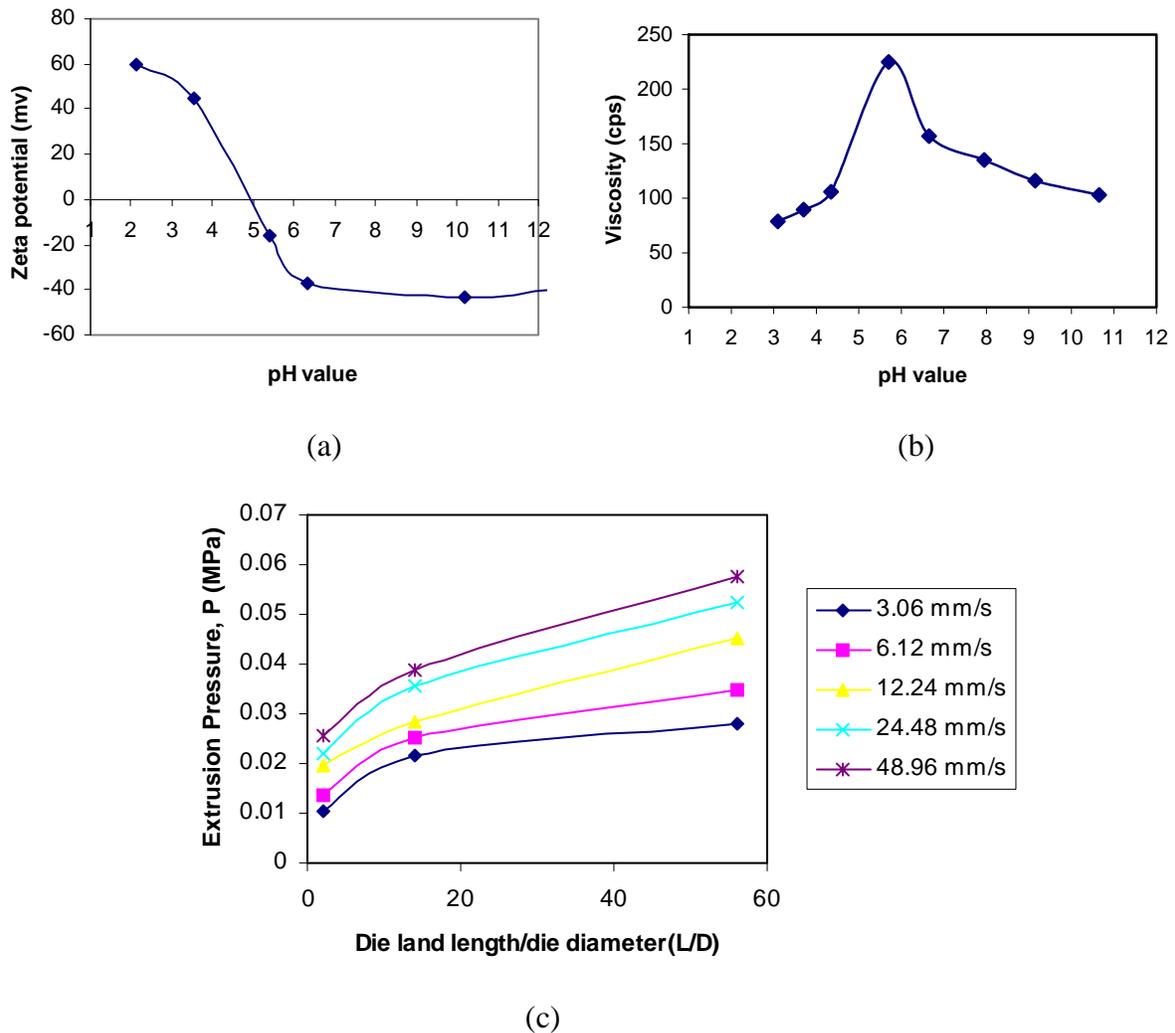


Figure 2. (a) Zeta potential of the dental porcelain powder as a function of the pH value, (b) viscosity change of the porcelain slurry (20 vol% solid) with the pH value at a shear rate of 20 s^{-1} , and (c) the extrusion pressure versus the ratio of the die land length to the die diameter, L/D, at different extrusion speeds as indicated.

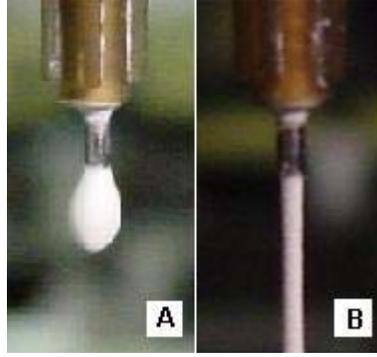


Figure 3. (a) The ball extrudate at pH = 9.5 and (b) the rod extrudate at pH = 7.5. Both are extruded with a nozzle 650 μm in diameter.

Calculation of extrusion parameters from the experimental relationships between the extrusion pressure and the extrusion speed was carried out. Data from one set of experiments are shown in Figure 2(c) which shows the relationship between extrusion pressure and the length-to-diameter ratio, L/D , at different extrusion speeds for the slurry prepared with pH = 7.5. The curves show some nonlinear characteristics at 3 different L/D ratios. Nevertheless, to simplify the calculation, the four-parameter description [6] was applied to describe the mathematical relationship between the extrusion pressure, P , and the L/D ratio:

$$P = 2(\sigma_0 + \alpha V)\ln(D_0/D) + 4(\tau_0 + \beta V) (L/D) \quad (1)$$

where σ_0 stands for the yield stress extrapolated to zero velocity, α a factor characterizing the effect of the extrusion speed on σ_0 , τ_0 the wall shear stress extrapolated to zero velocity (i.e., the initial wall stress), and β the wall velocity factor. Using eq. (1) the following extrusion parameters are obtained.

$$\begin{aligned} \sigma_0 &= 0.00455 \text{ MPa,} \\ \alpha &= 0.003885 \text{ MPa s m}^{-1} \\ \tau_0 &= 0.0001043 \text{ MPa} \\ \beta &= 0.000596 \text{ MPa s m}^{-1} \end{aligned}$$

Compared with the traditional macro-extrusion, σ_0 and τ_0 of the dental porcelain slurry in our micro-extrusion is much smaller (by an order of 1 to 2). The small yield stress and initial wall stress ensure the high sensitivity of the slurry and low friction on the wall, both of which greatly reduce delay and phase separation during extrusion.

Fabrication of Single Wall Crowns and Solid Artificial Teeth

Shown in Figure 4 is the 3-D STL digital model used for dental restoration. The 3-D file was sliced in the Z-direction at the desired thickness. On each slice the outlines and hatching lines were generated along which the extrudate would be deposited from the micro-extrusion nozzle. Stacking of these 2-D sections resulted in the formation of the desired dental unit.

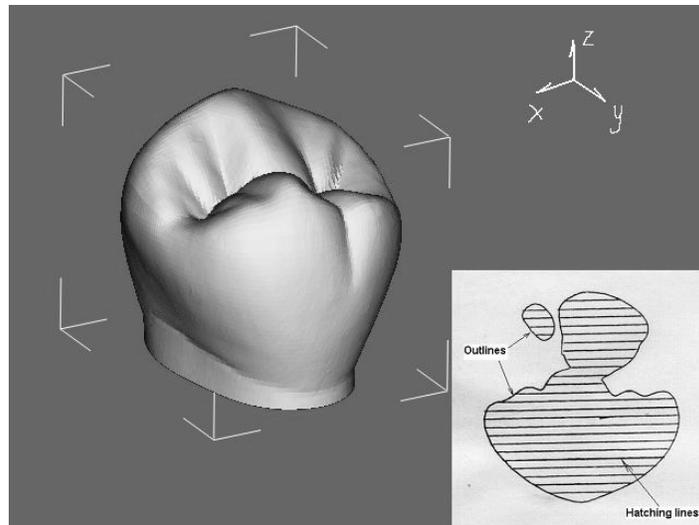


Figure 4. The STL digital tooth model with the insert showing the outlines and hatching lines on one of the slices obtained from the 3D model.

Fabrication of two types of dental units has been investigated. The first is single-wall crowns and the second is solid artificial teeth. These two types of dental units have been selected because they present different fabrication issues for dental restoration. The key issues related to single-wall crowns include slumping, overhang angles, and bridging capability, while the major issues pertinent to solid artificial teeth are material overfilling, generation of porosity, and dimensional accuracy. Of course, these issues could all be present in one type of the dental unit; however, some of them are more prominent in single-wall crowns, while some more noticeable in solid teeth.

Figure 5 shows several snapshots in fabrication of a single-wall crown and the cross section image of the crown fabricated. The dimensions of the crown are 14.97, 17.78 and 16.76 mm in the X, Y and Z direction, respectively. It can be seen that the stacking angle at the bottom of the crown is near zero (Fig. 5A and B). However, it changes to more than 45° on the top of the crown (Fig. 5D and E). Figure 5F clearly shows the variation of the stacking angle with the Z-position. Resistance to slumping is required for the bottom portion, whereas at the middle section the extrudate must have additional resistance to shear stresses due to the overhang structure with a stacking angle of about 20° . Finally, the extrudate at the top should have capability to bridge gaps as wide as 15 mm. The solution to all of these challenges is to control the rheological property and drying rate of the slurry. As discussed previously, pseudoplasticity is a required feature of the slurry in order for the slurry to flow smoothly during extrusion but then solidify in place once shear stresses are removed. However, the volume fraction of the solid loading in the slurry is also important. Relatively high solid loading (> 40 vol.%) allows the extrudate to dry right after extrusion and thus offers the extrudate the rigidity and bridging capability. The rigidity can prevent slumping. As shown in Fig. 5F, there is no slumping in single-wall crowns because the wall thickness at both the bottom and middle section of the crown is uniform. At the bridging section (i.e., the top of the crown) the wall thickness, however, is thinner due to the fact that the crown is fabricated under the condition of the nozzle height being only half of the critical

nozzle height (to be discussed more in the next section). Thus, a proper combination of the pseudoplasticity achieved by adjusting the pH value and the high solid loading (> 40 vol.%) is the key to successfully fabricating single-wall dental crowns with overhang structures and no slumping.

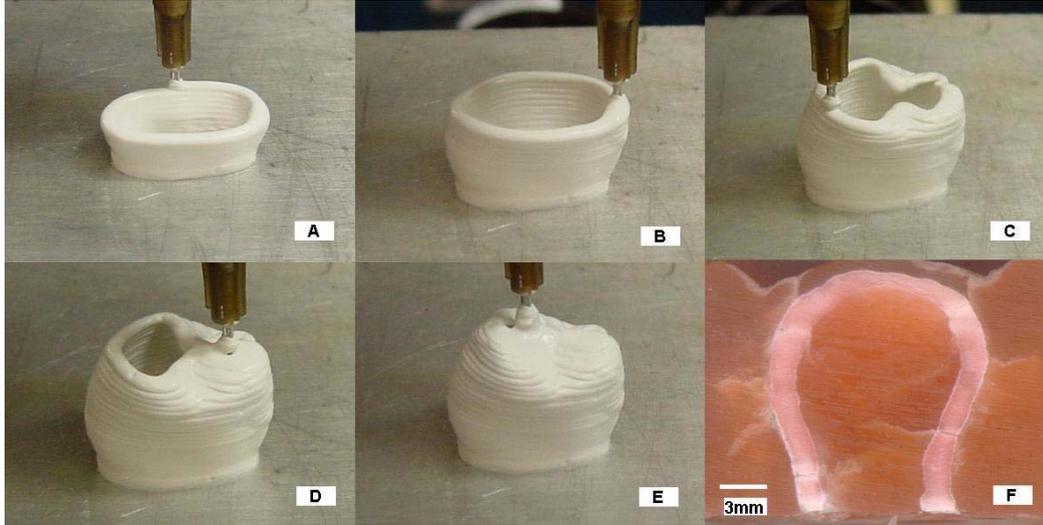


Figure 5. Snapshots in fabrication of a single-wall dental crown (A to E) and a cross section image of the crown fabricated.

Shown in Figure 6 are snapshots in fabrication of a solid artificial tooth. The outer dimension of the solid artificial tooth is identical to that of the single-wall crown. To obtain solid artificial teeth with good dimensional accuracy, the first requirement is again the pseudoplastic behavior of the slurry which, when combined with relatively high solid loading, will eliminate slumping and allow a high build rate. However, consideration should also be given to extrusion conditions so that the space to be filled by the extrudate will not be over-filled or under-filled. The former will give rise to a swollen tooth, while the latter will lead to porosity in the tooth. A previous study [12] has indicated that to prevent these problems from happening, the nozzle height (i.e., the distance between the nozzle tip of the micro-extruder and the previously extruded layer) should be equal to the critical nozzle height, h_c , as defined below.

$$h_c = \frac{V_d}{v_n D_n} \quad (2)$$

where V_d is the volume of the slurry extruded per unit time (called the extrusion rate hereafter), v_n the nozzle moving speed with respect to the substrate, and D_n the nozzle diameter. The physical meaning of eq. (2) is that the volume of the slurry extruded per unit time is equal to the volume available per unit time between the nozzle and the substrate (or the previously deposited layer) [12]. When the nozzle height is lower than h_c , there will be a squeezing effect, i.e., the slurry will be forced to take up the space beyond the volume defined by the product of the nozzle height, the nozzle diameter, and the distance traveled by the nozzle per unit time. As a result of this squeezing effect, the extrudate will be forced to flow sideward and the width of the extrudate

will be larger than the nozzle diameter. Thus, to avoid the over-filling, the spacing between the adjacent extrusion paths should be larger than the nozzle diameter to take this forced flow into account, or the nozzle height used in the extrusion should be equal to the critical nozzle height. The latter approach is adopted in this study so that neither over-filling nor under-filling is present. Finally, it should be noted that V_d , v_n and D_n are all experimental variables and can be varied independently. Thus, a particular h_c can be obtained by a combination of different V_d , v_n and D_n .

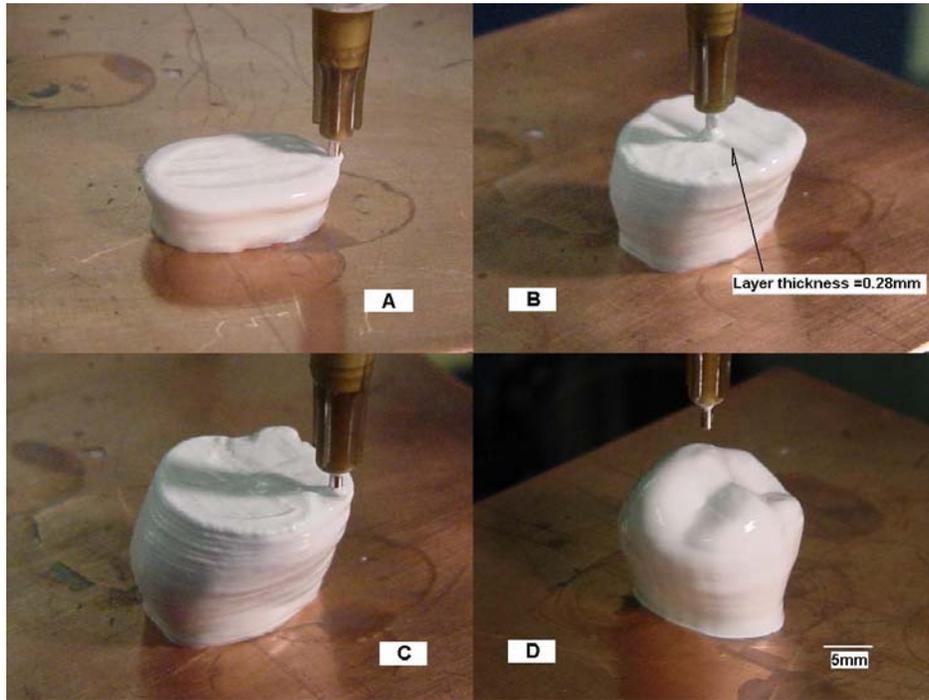


Figure 6. Snapshots in fabrication of a solid artificial tooth at the position of (a) the bottom, (b) middle, and (c) top. (d) shows the fabricated tooth in the green state.

In fabrication of solid teeth, hatching lines were extruded first, which is followed by extrusion of the outline(s). Such extrusion sequence was found to provide good surface finish and minimize staircase effects. A solid artificial tooth, as shown in Fig. 6, can be fabricated within 30 min. Furthermore, the dimensions and shape of the green tooth match that of the digital model quite well. However, some details on the top of the tooth are lost, which is caused by the delayed response of the extrusion process to the commands as the extrusion nozzle moves between different outline loops at the top. The delayed response results in additional extrusion at the ending point and insufficient extrusion at the starting point, both of which lead to the loss of the shape and dimension accuracy. However, hardware upgrade and software compensation could solve this problem.

Shrinkage of the Green Parts after Sintering and Microstructure Analysis

After the green parts were dried in air for about 12 hrs, they were sintered in an EPD high temperature furnace at 950°C for 5 min. Sintered teeth had 20%, 23% and 21% shrinkage in the

X, Y and Z direction, respectively. Thus, the shrinkage is relatively uniform in all three directions. This makes the matching of the dimensions of the artificial tooth to the desired dimensions easier and allows the inclusion of shrinkage in the design of the green tooth.

Optical microscopy and SEM analyses (Fig. 7) indicate that the microstructure of the artificial tooth after sintering is composed of two constituents; one is the glassy matrix, and the other the crystalline leucite particles. The presence of some bubbles is also observed, as shown in Fig. 7(a). The microstructure obtained is identical to the standard microstructure of the sintered dental porcelain in traditional dental restoration laboratories [11].

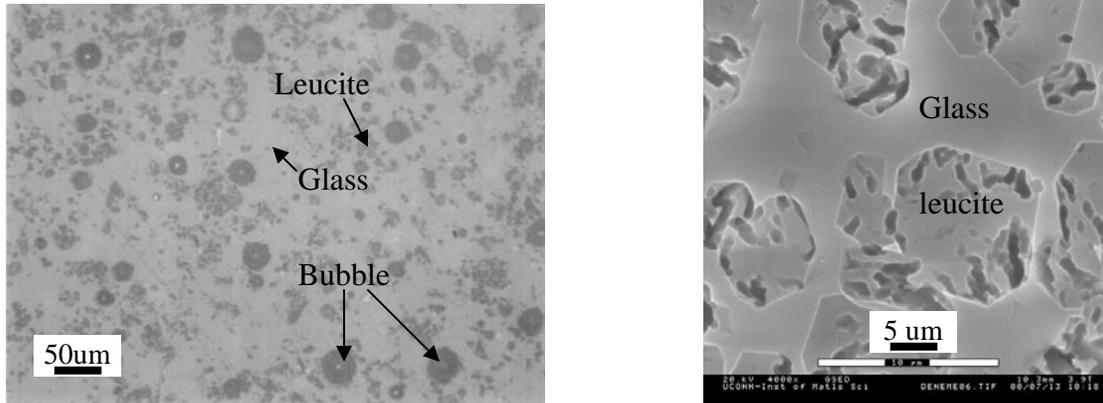


Figure 7. Microstructure of the sintered artificial teeth: (a) an optical micrograph and (b) an SEM micrograph, showing crystalline leucite particles embedded in a glassy matrix and the presence of some bubbles.

Summary

Artificial teeth have been fabricated via a micro-extrusion-based SFF process. The dimensions and shape of artificial teeth fabricated match that of the digital model quite well. Shrinkage of the green teeth during sintering is uniform along the X, Y and Z directions (at about 21%). The microstructure after sintering is identical to that made from traditional fabrication processes. Compared with the current, tedious, labor-intensive restoration procedure in dental laboratories, solid freeform fabrication of artificial teeth via slurry micro-extrusion has great potential to significantly reduce the restoration time and costs..

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