Particle Size Effects and Mechanical Properties of Alumina Dental Crown Fabricated by Stereolithography.

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Abstract
Ceramic dental crowns have a low risk of metallic allergies and are esthetically pleasing and so are actively investigated and developed in worldwide medical industries. In this study, three-dimensional dental-crown models composed of alumina and glass composite were fabricated successfully by using stereolithography. These precursors were dewaxed and sintered in air. Alumina bending test specimens of 1.2×4×20 mm in dimensions were fabricated by using the similar materials and processes. The highest bending strength of alumina ceramics with La$_2$O$_3$-B$_2$O$_3$-Al$_2$O$_3$-SiO$_2$ glasses was about 400 MPa. These glass components were coated on the alumina specimens to close micro cracks on the surface.

KEY WORDS: (Ceramic Dental Crown), (Alumina), (Stereolithography), (Mechanical Strength)

1. Introduction
In recent years, all ceramic dental crowns have been focused in the area of dental restoration. They have superiorities of the desirable aesthetics and biocompatibility compared with traditional metallic ones. A variety of methods have been contrived to manufacture the ceramic structure [1-3]. In particular, cutting work is currently a predominant method of forming the ceramic crowns. However, it is difficult to cut high hardness ceramics such as alumina and zirconia [4-5]. In addition, only a single crown can be fabricated during one operation by this method. In this study, a highly accurate and high mechanical strength ceramic dental crown was made by laser-scanning stereolithography. Using this technique, it is possible to make ceramics that are porous, dense, and those having complex structures. Alumina ceramics dental crown models were fabricated using laser-scanning stereolithography that employed computer-aided design and manufacturing (CAD/CAM). According to graphic data of the dental crowns obtained using computed tomography scanning, dense objects made of alumina ceramics and used as biomedical components were fabricated successfully by powder sintering processes. Moreover, the formed crowns were coated by dental glass to improve esthetic and mechanical properties.

2. Experimental
The computer graphic models of the dental-crown and plate samples for flexural strength were designed CAD software (Magics, Materialize NV). The created computer graphic models of the dental-crown and plate specimens were automatically converted into a numerical data format and sliced into file sets of cross-sectional planes with uniform thickness. These operational data sets were transferred to the stereolithography apparatus of the CAM equipment (SCS-300P, D-MEC Ltd.). Figure 1 shows a schematic illustration of the fabrication system. Photosensitive acrylic resin (KC-1159, JSR Corp.) with 40vol. % alumina powder (d017, grain size ave.: 0.17 μm, TM-DAR, Taimei Chemicals Co., Ltd.) or 70vol. % alumina powder (d180, grain size ave.: 1.8 μm, AL-170, Showa denko K. K.) were used for the materials of slurry in this investigation. This slurry was spread on a flat stage and smoothed. An ultraviolet laser beam (λ = 355 nm) was scanned over the deposited layer to create cross-sectional planes. Through layer-by-layer processes, solid components were fabricated. The fabricated precursors were dewaxed at 600°C for 2 h at a heating rate of 10°C/min. After dewaxing, the precursors were sintered in a reducing atmosphere (N$_2$-H$_2$) at 1500°C for 2 h. The sintered dental crowns were polished by a polishing wheel and coated with dental glass to improve esthetic and mechanical properties.
0.5°C/min and sintered at 1500°C for 2 h at a heating rate of 8.0°C/min in air. The sintered ceramic components were coated with La₂O₃-B₂O₃-Al₂O₃-SiO₂ dental glass (VITA In-Ceram ALUMINA, VITA Zahnfabrik). A paste of the dental glass powder was made with pure water. The paste was applied to the sintering sample surface and subjected to heat treatment at 1100 °C for 2 h in air. The glass infiltration process was repeated two times. The flexural strengths of plate specimens were measured using a three-point bending test machine (EZ-Test, Shimadzu Corp.). The sintered densities were measured by the Archimedes’ method.

3. Results

**Figure 2**-(a), (b) and (c) show the different direction views of the dental-crown precursor including with the Al₂O₃ particles of 0.17 μm in diameter at 40 vol. % fabricated by the stereolithography. The average dimension tolerance was within approximately 100 μm. The grooves at the top surface of green body have been precisely shaped, and the peripheral edge on the bottom formed sharply. Large stacking faults of layers were not observed on the surface. Figure 2-(d), (e) and (f) show the sintered body shapes of the dental-crown model along the different directions. The measured relative density reached approximately 98 %. X-ray diffraction peaks of carbides were not observed. Nano-size Al₂O₃ powders were considered to be sintered effectively at 1500 °C. The linear shrinkage ratios of the horizontal and vertical axes were in approximately 24 and 28 %, respectively. The large cracks or pores were not observed on the top and side smooth surfaces. However, some cracks were formed on the inside surface. The distortion in the shrinkage during the heat treatment is considered to cause the cracking.

**Figure 3**-(a), (b) and (c) show the green body shapes of the dental-crown model composed of photosensitive resin including with the Al₂O₃ particles of 1.8μm in diameter along different directions. The average dimension tolerance was within approximately 100 μm. Figure 3-(d), (e) and (f) show the sintered body shapes. The relative density reached approximately 97 %. X-ray diffraction peak of carbon was not identified. The linear shrinkage ratios of horizontal and vertical axes were approximately 7 and 9 %, respectively. The larger shrinkage for the vertical axis is considered to be decreased compared with nanoparticles sintering as shown in figure 2 because of the alumina particles dispersion with higher volume percent. The ceramics particle dispersion with the high volume fraction realized restraints of the shape deformations during the dewaxing and sintering.

**Figure 4** shows the flexural strength of alumina sintering body with and without dental glass. The flexural strengths without glass coating were 64±5.5 and 197±12 MPa as d017 and d180, respectively, while those with coating were 105±20 and 415±34 MPa, respectively. The d180 has a large powder diameter and sintering temperature is higher than the d017, so the alumina sample could not complete sintering process. Thus, the high flexural strength alumina was obtained with dental glass infiltration. The value of over 400MPa
is an acceptable level for the single ceramic dental crown.

4. Conclusion
Alumina dental crowns were accurately fabricated by laser-scanning stereolithography. Subsequently, dense alumina bodies were successfully obtained after heat treatment. The La₂O₃-B₂O₃-Al₂O₃-SiO₂ dental glass was infiltrated into open pores and cracks of the sintered alumina specimens. By the infusing process, the glass materials into sintered alumina bodies, maximum flexural strengths were obtained over 400 MPa (d180 with glass), which is considered to be a required level for use of single ceramic dental crowns.

5. Reference