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Dissertation Abstract

博士の専攻分野の名称：博士（歯学）

氏名：范 斯佳/ Fan Sijia

学 位 論 文 題 名

Establishment of fundamental techniques for fabrication of monolithic zirconia crown based on LCD technology

-Debinding effects of green bodies on the sintering behavior of zirconia-

(Additive Manufacturing技術の応用によるモノリシックジルコニアクラウン製作に向けた基盤技術の構築 —TOSOH-ZIRCONIA TZ-3YS-Eを用いたグリーン体の脱脂工程に関する基礎的研究—)

Keywords (5) zirconia ceramic, LCD, additive manufacturing, debinding process, heating rate

The aims of this study were to (1) investigate the feasibility of fabricating zirconia ceramic components using LCD printing; (2) evaluate whether increased heating rates during debinding compromise the final product qualities.

The cure depth for ELEGOO photopolymer resin and slurry containing 50wt%, 70wt%, 75wt% solid loadings of 3 mol% yttria-stabilized zirconia particles through UV light by ELEGOO MARS LCD 3D printer were first evaluated with different exposure time ranged from 8 to 256 seconds. Rectangular cubes (10.0 mm × 10.0 mm) with 1.0 mm thickness were designed, sliced, and printed with only one layer. Each specimen was measured for thickness at different 8 orientations varied 45°. Secondly, discs (diameter: 8.0 mm, height: 2.0 mm) were cured with 50 µm per layer under 50 seconds of exposing by ELEGOO MARS with four commercial resins, NOVA 3D, RESIONE C01, TrinDy DT-CM-220, and CREALITY. Debinding process was then performed at 100°C, 200°C, 300°C, 400°C, and 500°C, with 30, 60, 90, 120 minutes, respectively. The diameter and thickness of each disc were measured before debinding for calculating the trueness, precision, and after debinding for obtaining the dimensional shrinkage. Heatmaps were applied to visualize the thermal dynamics of resins during debinding. Finally, discs (Ø: 8.0 mm, h: 1.0 mm) were sliced with 20 µm per layer and printed with 50 wt% of zirconia particles under 40 seconds of exposing for each layer. Zirconia products were debound at 200°C for 10 minutes and heated to 700°C and kept for 10 minutes with assigned heating rates varied from 20 to 120°C/min. Then 6.5 hours of sintering followed identically. Thickness, diameter, and weight were measured before and after the post-processing, volume value was calculated from the cylindrical volume relation. And dimensional shrinkage as well as weight loss were subsequently obtained. The surface of each disc was examined by SEM to evaluate the final

product qualities.

The extended exposure markedly increased the curing depth of resin with a strong monotonic increasing trend within the range from 8 to 128 seconds. However, the elongated exposure time had no effect on the thickness of zirconia products against different solid loadings, 50wt%, 70wt%, 75wt%. In comparison of four resins, NOVA 3D rendered high producing repeatability with relatively low CV (thickness: 25.8%, diameter: 30.8%), albeit with the largest dimensional deviation from CAD model. Although the heatmaps suggested a shared thermally driven mechanism regarding dimensional change, NOVA 3D exhibited the consistency across debinding temperatures from 100 to 500°C, indicating its superior thermal stability among four resins. Printed zirconia products displayed a dimensional discrepancy from CAD with smaller diameter (7.78 ± 0.06 mm) and excessive thickness (1.67 ± 0.17 mm). After post-processing, the thickness shrinkage significantly dropped at highest heating rates (110-120°C/min) compared to that at lower heating rates (20-40°C/min, $p < .05$; 50-70°C/min, $p = 0.063$), and volume shrunk substantially at lowest heating rates (20-40°C/min) compared to the higher heating rates (80-100°C/min, $p < .05$; 110-120°C/min, $p < .01$). In contrast, diameter shrunk steadily across heating rates. Additionally, weight loss remained statistically similar despite a broad overlap of distributions across rates. The surface of the sintered zirconia products in contact with the Fluorinated ethylene propylene (FEP) membrane appeared smoother than that adjacent to the support structures. Notwithstanding, FE-SEM micrographs showed that all zirconia specimens displayed similar fracture morphologies, characterized by numerous cracks, crevices, and chipping after post-processing. Furthermore, a yellowish coloration was observed after sintering.

This study offers a new perspective on manufacturing zirconia ceramic products by the LCD method. Resin plays a crucial role in both photopolymerizing process and post thermal treatment depending on its individual optical characteristic and thermal stability. Although elongated exposure time ensures thickness expansion by overcuring, layer-by-layer printing generates weak interlayer bonding, which emerges as an important factor associated with the anisotropic dimensional change throughout the VPP and post-processing. This effect is exacerbated by rapid heating in the debinding stage. On the other hand, the intrinsic characteristics of VPP contribute to distinct microstructural differences at the interfaces between the zirconia products, the FEP membrane, and the supports. Defects are extensively generated at heating rates between 20 and 120°C/min owing to the substantial internal pressure caused by gas evolution. In addition, an inadequate debinding scheme leaves carbonaceous residuals, resulting in discoloration during sintering.