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Brushite ceramic coatings for dental brace brackets fabricated via aerosol deposition

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ABSTRACT

Biocompatible ceramic coatings were fabricated at room temperature by via aerosol deposition (AD), in order to improve the bonding strength between the dental sapphire bracket and the resin. First, the deposition properties of the brushite films coated on the flat sapphire substrate were investigated, confirming their good deposition rate and appropriate adhesive strength. Then, the mesh and check patterns of the coatings were formed on the sapphire brackets in order to obtain a sufficient shear bond strength. The mesh-pattern grown by converging nozzles exhibited a low film thickness (< 10 μm) and shear bond strength (< 7 MPa) due to the sharp triangular-shaped cross sections. In contrast, the check-pattern showed a high film thickness of ~70 μm at the same deposition time, as well as a high shear bond strength of ~14 MPa, implying that this check-pattern brushite coating formed via the AD process is a promising candidate for application in dental brace brackets.

1. Introduction

Ceramic orthodontic brackets were first introduced in the middle 1980 s to satisfy the aesthetic needs of patients, and they were designed to combine the aesthetics of plastic with the reliability of metallic brackets [1–3]. Moreover, ceramic brackets were more transparent than plastic brackets and had better color stability. Of these, single crystal sapphire brackets, from which aluminum oxide is made, are widely used due to their aesthetics, strength, and resistance to chemical degradation. However, despite these advantages, fabricated sapphire brackets cannot chemically adhere to any of the currently-available bonding resins. For these reasons, numerous conditioning methods, including chemical, mechanical, or a combination of chemical and mechanical methods of retention, have been suggested to pretreat ceramic surfaces [4–6].

Chemical retention is provided by an intermediate layer of glass and using a silane coupling agent to obtain a chemical bond between the bracket and the adhesive. Although the shiny surfaces of the ceramic brackets that are chemically bonded allow a much greater distribution of stress over the whole adhesive interface without the presence of any localized stress areas, the debonding stress can migrate from the bracket-adhesive interface to the adhesive-enamel interface, consequently damaging the enamel. Thus, the major problem associated with chemically-bonded ceramic brackets is the occurrence of an enamel fracture after debonding [7–9]. To solve this problem, new ceramic brackets were developed with mechanical retention.

Ceramic brackets that offer a mechanical bond with the adhesive have retentive grooves with edge angles of 90°. There are also crosscuts to prevent the brackets from sliding along the undercut grooves that have sharp edge angles, thus leading to high localized stress concentrations around the sharp edges and resulting in brittle failure of the adhesive [3]. Moreover, mechanical retention can be provided by various indentations or recessions in the bracket base. These indentations primarily provide mechanical interlocking with the intermediate resin adhesive. The bracket bases were formed with various base designs, such as beads, grooves, or round pits, for the purpose of making and improving the mechanical interlocking between the brackets and the teeth [10,11]. To produce mechanically-retentive ceramic brackets, ceramic powders and beads were commonly formed on ceramic brackets with a proper binder, and were fired according to the manufacturer's instructions. However, the process to make ceramic powders or beads is very complex. Post processing at a high temperature is also required to fasten the ceramic powders and this result in high production costs. Thus, a new alternative process technology is needed to reduce production costs and process temperature.

Recently, aerosol deposition (AD) has received a significant amount of attention for its potential use in the application of biocompatible coatings [12–18]. It is a powder spray coating technology that creates dense ceramic coatings with a high deposition rate, high hardness, and

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high film density [19,20]. This process uses fine ceramic particles as the starting materials, and deposition is simple because the coating layer is obtained by the collision of the particles onto a substrate [21,22]. In addition, the AD process can form dense ceramic coatings at room temperature without any post heat treatment. Accordingly, it is favorable for the application of ceramic brackets with mechanical retention.

The aim of this work is to fabricate a ceramic bracket with high shear bond strength through the deposition of ceramic coating layers via AD without post processing. We used well-known biocompatible brushite ceramic powders [23,24], and we then verified their deposition properties in terms of crystallinity, microstructure, film thickness, surface roughness, and adhesive strength of the brushite films deposited on the flat sapphire substrates. Then, mesh and check patterns of the brushite coatings were formed on the sapphire brackets and their shear bond strength was measured.

2. Experimental procedures

2.1. Ceramic coating methods

The AD system consists of an aerosol chamber, a deposition chamber, nozzles, vacuum pumps, and mass flow controllers. The aerosol flow generated in the aerosol chamber is delivered to the deposition chamber by a pressure difference between the two chambers, and it is accelerated by a nozzle. The AD process is based on accelerated particles being solidified through impact on the substrate. The specific details of the AD apparatus and coating process have been described elsewhere [12–20,25]. Biocompatible brushite powder (Dio, Co. Ltd., Busan, Korea) was prepared as a ceramic starting powder. The brushite ceramic particles were sprayed on flat sapphire substrates and sapphire brackets via the AD using a conventional nozzle with an orifice size of 1.0×0.4 mm^2. In particular, three types of designed converging nozzle with orifice sizes of 0.5×0.5 mm^2, 0.8×0.8 mm^2, and 1.0×1.0 mm^2, having different widths, were also used to produce various mesh-patterned brushite coating on the sapphire brackets. The process parameters are summarized in Table 1.

Moreover, two coating methods were used to make the mesh and check patterns of brushite ceramic coatings, as shown in Fig. 1. One method for the mesh patterns involves direct writing via nozzle movement, and the other for check patterns is shadow mask pattering. To obtain mesh-pattern coating layers with different widths of the patterns on sapphire brackets, the brushite ceramic powders were sprayed using the three types of nozzles, and line scanning based on direct writing was conducted on the brackets, as shown in Fig. 1(a). This patterning is achieved by moving the substrate holder or the nozzle. Next, to fabricate the sapphire brackets with check-patterned coating layers, a metal shadow mask of cross stripes was designed and immobilized on the brackets, as shown in Fig. 1(b). Brushite ceramic powders were sprayed on the mask using a conventional nozzle with an orifice size of 1.0×0.4 mm^2 in order to obtain a wide scanning area, and the brushite bumps of the check pattern were then obtained after removing the mask from the brackets.

Table 1
Experimental parameters of an aerosol deposition process.

Powder	Brushite
Substrate types	Flat sapphire, sapphire bracket
Size of nozzle orifice	10×0.4 mm^2 0.5×0.5 mm^2, 0.8×0.8 mm^2, 1.0×1.0 mm^2
Consumption of carrier gas	8 L/min
Carrier gas	He
Scanning rate	1–2 mm/s
Working pressure	1–50 Torr
Distance between substrate and nozzle	10–20 mm
Deposition temperature	Room temperature
Deposition time	5–10 min
Vibration speed	300–500 rpm

2.2. Characterizations

The crystallinities of the brushite starting powder and coatings were examined using an X-ray diffractometer (XRD, X′Pert PRO, Philips Ltd., USA) with monochromatized CuK$_\alpha$ radiation at 40 kV and 30 mA. The microstructures of the powder and coatings were observed via field-emission scanning electron microscopy (FE-SEM, S-4700, HITACHI Ltd., Japan). The surface roughness (Rq) and thickness of the ceramic coating layers were measured with an atomic force microscope (AFM, XE-100, PSIA Ltd., Suwon, Korea) and a surface profiler, respectively. The adhesion strength was evaluated through tensile adhesion testing to provide a measure of the resistance to detachment of a coating from the substrate. The adhesive strength of the coating layers was measured using a universal testing machine (UTM, DUT-300CM, Daekyung Engineering Corp., Seoul, Korea). Ceramic coating layers with a size of 10 mm×10 mm and 3 µm in thickness were prepared to evaluate the adhesive strength. A cylindrical rod (5 mm diameter and 50 mm length) was bonded to the surface of the ceramic coatings using epoxy adhesive (4-META/MMA-TBB). The rod was then pulled using a UTM at a loading speed of 5 mm/min until the ceramic coatings peeled off. A force-displacement curve was derived from the tensile test, and the peak load f of the breaking force was estimated. The adhesive strength F was obtained by dividing an area that was swept off the coatings by the dividing peak load f, and the shear bond strength measurements were also performed with a UTM. Fig. 2 shows a schematic drawing of the measurement method for the shear bond strength between the ceramic bracket with ceramic coating and enamel using 4-META/MMA-TBB resin. The shear force was applied to the enamel-adhesive interface with a crosshead speed of 1 mm/minute until debonding [26]. The bond strength values were calculated by dividing the maximum load (in Newtons) by the base area of the bracket to convert the results to megapascals (1,000,000 N/m^2).

3. Results and discussion

3.1. Deposition properties of Brushite film fabricated via AD

Before fabricating a brushite ceramic coated sapphire bracket for a dental brace, a brushite thick film was first deposited on a flat sapphire substrate via AD with a conventional nozzle. Despite of the condition at room temperature, a dense brushite ceramic layer successfully formed on the sapphire substrate. In general, it is difficult to deposit thick ceramic films on a hard substrate like sapphire, but a high consumption of the carrier gas, implying a high kinetic energy of the particles, can form the anchoring layer on the hard substrate [19,20,27]. Therefore, we applied a high carrier gas of 8 L/min in this study. Fig. 3 shows the XRD patterns of the starting brushite powder and deposited film. No secondary phase was observed in the film, but the XRD patterns of the film showed peak broadening, indicating a decrease in crystallite size. As one of the features in the AD process, aerosol-deposited ceramic films were reported to show a small crystallite size due to crushing the particles when they collide with the substrate and with each other [19,20]. Moreover, the 020 and 040 peaks showed a relative decrease in intensity after the film growth. This phenomenon has been reported for various coating films, indicating the preferred orientation as the origin [28–30], and the same reason might be applied to our XRD results.