The Flexural Strength of Y-TZP Coated with Carbonate Apatite for Dental Implant Material

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Abstract. In order to gain acceleration of the osseointegration process after implant placement, micro retention using inorganic elements such as Hydroxyapatite (HA) were commonly used as a coating material in dental implant. Meanwhile, another inorganic material such as Carbonate Apatite (CO₃Ap) has been known as bone substitute for decades. The purpose of this study is to investigate the flexural strength of Yttria-Stabilized Zirconia (Y-TZP) as dental implant material after being coated with CO₃Ap. Ten specimens of Y-TZP were divided into two groups. The first group was coated with CO₃Ap while other groups without coatings were used as the control. Biaxial flexural strength was determined using piston on three balls-technique and data were evaluated by statistical analysis. The specimens surface were analyzed through images taken by Scanning Electron Microscope (SEM). As the result, this study showed that there was no statistically significant found between the group with coating and the control group (p>0.05). The biaxial flexural strength’s mean of the group with coating and control were 212.80 MPa and 209.35 MPa; while micro Vickers hardness’ means were 229.56 HV and 245.40 HV. It can be concluded that there was no difference in the mean flexural strength between Y-TZP before and after coating.

Introduction

The use of ceramic material in dentistry has been increased worldwide due to higher esthetic demand and biocompatibilities that needed to achieve better result for dental restoration. The benefit in using zirconia as a ceramic biomaterial was its chemical and dimensional stability, also mechanical strength. Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) was developed as an alternative to metal frameworks for fixed dental prostheses because of its high strength and toughness [1,2].

Surface coatings of zirconia and titanium were one of the best approaches to improve the adhesion to other materials in restorations and increase the implant fixation to bone tissue. Bioactive materials such as hydroxyapatite and bioactive glass were used for coating zirconia and titanium implants to improve the biological interactions within tissue cells [3]. Meanwhile, Carbonate apatite (CO₃Ap) is an inorganic element of bone which contains 4–8 weight% of carbonate on its’ apatitic structure. [4-8]. According to Hirota, et al., carbonate-containing hydroxyapatite apatite coating has good biological activity on partially stabilized zirconia, rather than the non-coating [9]. CO₃Ap prepared from the present method has higher possibility to be used...
as an ideal bone replacement because the set CO$_3$Ap had low crystallinity similar to bone apatite since it was synthesized at body temperature [4,10]. After surface coatings, there is a chance of alteration on physical or chemical properties of the surface. The surface roughness could enhance micro-mechanical interlocking and also change the surface chemistry. This goes to activation of the surface towards the adhesion to other materials and enhancement of the biological activity to implant fixation [3]. Good biological properties must be accompanied with good physical properties, so the abutment could withstand the conditions in the oral cavity. The potency of any dental ceramic including clinical behavior and the limitation can be predicted by testing their strength [11]. The strength of dental ceramics can be assessed through flexural strength testing since these brittle materials are stronger in compression than tension [12]. Other factors such as temperature peaks are able to transform the metastable tetragonal crystalline phase of zirconia ceramic, resulting in a decrease in their strength [13]. This research aims to evaluate the mechanical properties of a CO$_3$Ap coated on Y-TZP using the dip coating method.

Materials and Method

Specimen Preparation. Commercially high purity 3 mol% Yttrium stabilized zirconia (Y-TZP) powders (Zirai Guangzhou Hongwu Material Technology Co., Guangzhou, China) were used to fabricate the specimens. All the specimens were compacted into the mold and uniaxially pressed at 15 MPa and subsequently sintered at 1500°C for 7 hours (Thermolyne 59300 High-Temperature Tube Furnace, Iowa, USA). The specimens were prepared in partially sintered state and in an enlarged size to compensate sintering shrinkage. The final dimensions were 11 mm x 1 mm (+/- 0.05 mm). The CO$_3$Ap was prepared based on the previous study [4,10]. All the specimens divided into two groups, CO$_3$Ap coated and non-coated as a control group. Five specimens were prepared to surface coating with CO$_3$Ap with a dip coating method. The CO$_3$Ap suspension was stirred for 10 minutes. Each of the specimens was etched with 9% hydrofluoric acid (HF) for 1 min at room temperature and immersed vertically in a CO$_3$Ap suspension for 1 minute and then removed, rinsed with distilled water, and dried in an incubator at 37°C for 24 hours. The coated specimens then heated until 500°C in oven furnace. The CO$_3$Ap will decompose at range of 700-1400°C from XRD and Thermogravimetry (TGA) analyses [14,15]. In addition, the TGA analysis of CO$_3$Ap decomposition at 500°C did not evidence a substantial change of the C content, the weight loss is 0.1 wt% in terms of carbonate, so the CO$_3$Ap coating layer still existed in this study [15].

Flexural Strength Preparation. Biaxial flexural strength values were determined in accordance with the ISO Standard 6872 [16]. A universal testing machine (LRX/LRX5K, Lloyd Instruments, Fareham, England) was used to test all the specimens at room temperature. The sample holder for the biaxial flexural strength test comprised three tempered steel balls with a diameter of 3.2 mm. The steel balls formed an equilateral triangle with a radius of 4 mm and the ball support circle was 120°. Each specimen rested centrally on three symmetrically based balls and the load was applied (1.0 mm/min) to the center of the top surface by a flat piston (diameter 1.0 mm) until fracture occurred. The biaxial flexural strength is calculated as (Eq.1):

$$S=0.2387P(X-Y)/d^2$$  \(1\)

where S is the biaxial flexural strength (MPa), P is the fracture load (N), and d is the specimen disk thickness at fracture origin (mm). V is the Poisson's coefficient (ceramic = 0.25, ISO 6872), A is the radius of support circle (mm), B is the radius of a loaded area (mm) and C is the radius of specimen disk (mm) (Eq.2) [13].
Micro Vickers hardness test was performed in order to examine whether the coating process will affect the hardness of the surface of the zirconia disk. Five pieces of specimens with coating and non-coating from the biaxial flexural testing were randomly selected to measure micro Vickers hardness using an indentation tester. Five indentations were placed on both coating and non-coating sides within the same specimen using a loading mass of 1.5 kg.

After the fracture strength test was complete, Scanning Electron Microscope (SEM, Hitachi, Tokyo, Japan) images of 3 fractured disks were obtained from each group. Cross-sections of the specimens were evaluated to observe the coating layer and substrate [17].

**Results and Discussion**

Figure 1 showed the mean biaxial flexural strength of Y-TZP coated with CO3Ap and non-coated were 212.80 MPa and 209.35 MPa. The result revealed no significant effect of surface treatment on flexural strength. The t-test statistical analysis indicated that there was no statistically significant from two groups of Y-TZP.

This biaxial flexural strength data was supported by Young’s modulus as the followed results of 8.76 GPa for coating and 8.78 GPa for non-coating as shown in Figure 2.
The effect of the dip coating on the biaxial flexural strength of zirconia specimens was evaluated. The result showed no changes affected regardless of the coating type or method. Based on the results of this study it was found that there were no statistically significant in flexural strength between Y-TZP before and after coating. The difference happened compared with previous studies may be due to the synthesis process of ZrO$_2$ powder. This might be due to the precursors of HCl and ZrCO$_3$ that left behind during the synthesis processed, its allowed changes in mechanical properties.

Figure 3 showed the mean Vickers hardness of the specimen non-coating and coating which were 229.56 HV and 245.40 HV. In addition, the non-coating sides had mean hardness values lower than coating sides; however, there was no statistically significant difference between both sides.

Zirconia is a polymorphic material existing in three stages; monoclinic (m), tetragonal (t), and cubic (c), which are stable at different temperatures. The tetragonal phase exists at high temperatures; however, it can be stabilized at room temperature by the addition of oxides of yttria or ceria to the zirconia structure (Partially Stabilized Zirconia; PSZ) [12]. This strength lies in the transformations happening in the metastable tetragonal crystalline structure of zirconia at room temperature. When under stress, the tetragonal grains transform into monoclinic with a 3–4% volume expansion. The consequent volume expansion creates compressive stresses that oppose the
tensile stresses leading the induced crack, hence preventing further crack propagation [18,19]. However, progressive transformation of the metastable tetragonal phase into monoclinic, and consequently an increase in the monoclinic phase, leads to a reduction in strength and toughness of zirconia [18]. The tetragonal to monoclinic transformation can be enhanced by high peaks in temperature. In addition, temperature peaks in the range 900–1000°C may also induce a reverse monoclinic to tetragonal transformation, which again negatively affects the mean flexural strength [20,21].

![Fig. 4 SEM images of the cross-sectional observation of Y-TZP non-coated (a) and coated (b)](image)

![Fig. 5 EDX Spectrum Non-coated Y-TZP (a), Coated Y-TZP (b)](image)
The surface appearances of Y-TZP disks were observed using a scanning electron microscope. Specimens were sputter coated with gold before being examined. Cross-sectional views of specimens were performed. Figure 4 shows SEM images of the cross-sectional views of CO3Ap films on Y-TZP disks. The CO3Ap films were detached from the substrate at any part and non-homogenous. To confirm the presence of CO3Ap layer, detection of the calcium and phosphorous, which are components of CO3Ap coating layer, was performed by EDX spectrum. The presence of calcium and phosphorous in EDX spectrum Y-TZP coated disk showed that there is coating layer in Y-TZP disk which was not present at Y-TZP non-coated (Figure 5).

It can be concluded that the CO3Ap coating has no effect on the mechanical properties of Y-TZP. Therefore, by adding a CO3Ap coating will accelerate the osseointegration process without changing its mechanical properties. Lower Y-TZP flexural strength at this study may be caused by a less uniform compacting process, which causes the thickness of the specimen to be less homogeneous. Generally, the zirconia compacting process use cold isostatic pressing (CIP) pressure to improve the densification of slip-casted zirconia blocks. CIP should be conducted under a compaction pressure of 250 MPa to produce dense and homogeneous zirconia blocks [22]. And also there is a possibility that grain size of Y-TZP might vary from the manufacturer. A correlation between grain size as well as flexural strength was reported for the first dental zirconias [23]. The increased grain size may result in enhanced crack formation [24]. The grain size significantly influences the mechanical properties of Y-TZP, whereas high temperature and longer sintering periods produce larger grain sizes and will subsequently diminish the mechanical properties due to large pore sizes. Therefore, higher sintering temperatures lead to larger grain sizes. Consequently, the sintering process becomes the determining factor and thus the process control needs to be emphasized [25].

Summary

Based on the findings of this in vitro study, there was no statistically significant of flexural strength between Y-TZP non-coated and Y-TZP coated with CO3Ap. The etched processes and CO3Ap coating did not change the mechanical properties of Y-TZP itself.

References


