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β-TCP coatings on zirconia bioceramics: the importance of heating temperature on the bond strength and the substrate/coating interface

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Abstract
β-tricalcium phosphate (β-TCP) coatings were synthesized on tetragonal zirconia (Y-TZP) discs by heating the apatite coating between 800 oC and 1200 oC. The study results suggest that heating temperature has a strong influence on the coating bond strength and microstructure of the substrate/coating interface. The β-TCP coatings fired at 800 oC and 900 oC exhibited excellent tensile bond strength (~50 MPa) while heating at 1100 oC and 1200 oC led to decreased bond strength (~30 MPa) as the result of substantial structural and microstructural changes: diffusion of Y3+ from the zirconia substrate in the coating resulting in partial crystal transformation (t-m) of zirconia, formation of surface uplifts and nanoporosity in zirconia, as well as generation of large residual thermal stresses leading to microcracking of the β-TCP coatings. However, these structural changes did not have any measurable effect on the flexural strength of the bulk zirconia substrates.

Keywords: tricalcium phosphate zirconia interface bioactive coating bond strength

1. Introduction

Zirconia ceramics, particularly yttria partially stabilized tetragonal polycrystals (Y-TZP), are promising materials for the fabrication of dental bone implants due to their outstanding mechanical properties, biocompatibility and corrosion resistance. However, much work is still needed for them to reach their full potential as biomaterials. For example, zirconia is known to be bioinert meaning that after implantation in the bone tissue, fibrous layer may form around the implant thereby inducing aseptic loosening [1][2]. A well-established strategy for improving the osseointegration of implants is to coat them with calcium phosphates (CaP), which are bioactive and exhibit good bone-bonding ability. Among the different CaP phases, the β-tricalcium phosphate (β-TCP) phase is especially attractive for the fabrication of coatings on zirconia due to its osteoinductivity and well-documented clinical success as a bone filler [3][4]. Nevertheless, despite these promising characteristics, there are several concerns on using β-TCP or any other CaP phase as the bioactive coating that are mainly related to their poor mechanical properties and weak bonding strength to the substrate [5][6][7][8][9]. Therefore, an important challenge for the future is to develop strongly bonded β-TCP coatings on zirconia implants which would combine bioactive characteristics with excellent mechanical properties that wouldn't affect the bulk properties of the implant, so that long-term stability can be achieved.

As β-TCP is a high-temperature CaP phase, synthesis of β-TCP coatings inevitably includes heat treatment at elevated temperatures above 700 ^{o}C [10]. Heating is also expected to influence on the coating adhesion. However, until now there haven't been any systematic studies done to assess the influence of firing temperature on the β-TCP coating mechanical properties and its potential influence on the strength of the bulk zirconia substrate. Therefore, to shed more light on this topic, the aim of the current work was to study the influence of the heating temperature in the range between 800 °C and 1200 °C on the β-TCP coating bond strength to zirconia (3Y-TZP) and on the structural changes at the substrate/coating interface.

2. Materials and Methods

2.1. Preparation of zirconia substrates

In our experiments we used sintered zirconia (3Y-TZP) discs (diameter =15 mm, thickness=1.5 mm). The discs were prepared by the dry pressing of commercially available yttria-stabilized tetragonal zirconia powder containing 3 mol.% yttria and 0.25 wt.% alumina (TZ-3Y-BE, Tosoh, Japan). After sintering at 1400 °C for 4h one side of the discs was polished to a mirror finish. Prior to the synthesis of coatings, the discs were cleaned with soap and then sonicated for 10 min in the following sequence: acetone, ethanol and deionized water.

2.2. Synthesis and characterization of β-TCP coatings on zirconia substrates

The β-TCP coatings were synthesized on zirconia substrates in two steps encompassing: (i) wet-chemical deposition of a biomimetic apatite (calcium-deficient hydroxyapatite; CDHA, Ca/P~1.5) coating, followed by (ii) post-deposition thermo-mechanical processing, which included thermal conversion of CDHA into β-TCP by a) heat treatment between 800 °C and