

## Dental zirconia can be etched by hydrofluoric acid

Tool SRIAMPORN<sup>1</sup>, Niyom THAMRONGANANSKUL<sup>2</sup>, Chumphol BUSABOK<sup>3</sup>, Sushit POOLTHONG<sup>4</sup>, Motohiro UO<sup>5</sup> and Junji TAGAMI<sup>6,7</sup>

<sup>1</sup> Faculty of Dentistry, Chulalongkorn University, 34 Henri-Dunant Rd, Palumwan, Bangkok 10330, Thailand

<sup>2</sup> Department of Prosthodontics, Faculty of Dentistry, Chulalongkorn University, 34 Henri-Dunant Rd, Patumwan, Bangkok 10330, Thailand

<sup>3</sup> Department of Material Innovation, Thailand Institute of Scientific and Technological Research (TISTR), Pathumthani, 12120, Thailand

<sup>4</sup> Department of Operative Dentistry, Faculty of Dentistry, Chulalongkorn University, 34 Henri-Dunant Rd, Patumwan, Bangkok 10330, Thailand

<sup>5</sup> Advanced Biomaterials Section, Graduate School of Medical and Dental Sciences, Tokyo Medical and Dental University, 1-5-45 Yushima, Bunkyo-ku, Tokyo 113-8549, Japan

<sup>6</sup> Cariology and Operative Dentistry, Department of Oral Health Sciences, Graduate School of Medical and Dental Sciences, Tokyo Medical and Dental University, 1-5-45 Yushima, Bunkyo-ku, Tokyo 113-8549, Japan

<sup>7</sup> Global Center of Excellence Program; International Research Center for Molecular Science in Tooth and Bone Diseases, Graduate School, Tokyo Medical and Dental University, 1-5-45 Yushima, Bunkyo-ku, Tokyo 113-8549, Japan

Corresponding author, Niyom THAMRONGANANSKUL; E-mail: niyom.t@chula.ac.th

The surface morphology and crystal structure change of dental zirconia after hydrofluoric acid (HF) etching were evaluated. Four groups of sintered zirconia specimens were 1) control group, 2) immersion in 9.5%HF at 25°C for 1, 2, 3, or 24 h, 3) immersion in 9.5%HF at 80°C for 1, 3, 5, or 30 min and 4) immersion in 48%HF at 25°C for 30 or 60 min. The specimens were evaluated under SEM and XRD. The SEM analysis revealed changes in surface topography for all the HF-etched zirconia specimens. The irregularities surface increased with increasingly longer immersion times and higher etching solution temperatures. The XRD analysis of the HF-etched zirconia specimens revealed the presence of a crystalline monoclinic phase along with a tetragonal form. It was concluded HF can etch dental zirconia ceramic, creating micro-morphological changes. Tetragonal-to-monoclinic phase transformation was induced on the etched zirconia surface.

**Keywords:** Acid etching, Hydrofluoric acid, Nano-porosity, Surface treatment, Zirconia

### INTRODUCTION

The use of high-strength zirconia ceramics has become increasingly popular in dentistry because of their excellent mechanical properties such as high flexural strength and toughness<sup>1</sup>, esthetic appearance<sup>2</sup>, and biocompatibility<sup>3-5</sup>. These ceramics are suitable for a variety of clinical applications, including: posts and cores<sup>6,7</sup>, dental implants<sup>8,9</sup>, orthodontic brackets<sup>10,11</sup>, and fixed-partial dentures<sup>12-15</sup>. Moreover, zirconia ceramic has a polycrystalline structure, which is acid resistant<sup>5,16</sup>. For cementation, silica-based ceramics are etched to achieve good bonding through the use of hydrofluoric acid (HF) followed by silanization<sup>9</sup>. In contrast, zirconia is a silica-free ceramic, and is resistant to conventional etching techniques<sup>5,16</sup>. Surface treatments such as air abrasion with aluminum oxide particles roughen the zirconia surface and increase the bond strength<sup>17</sup>. Other surface treatments of zirconia including laser treatment<sup>18,19</sup>, selective infiltration technique (SIE)<sup>20</sup>, hot etching solution<sup>21,22</sup>, nano-structured alumina coating<sup>23</sup>, and slurry-coated ceramic<sup>24</sup> have been developed to improve and enhance the surface roughened area to facilitate the mechanical interlocking of the ceramic and bonding resin.

Chemical bonding to the zirconia surface can be achieved using a variety of chemical substances. Silane coupling is recommended for use with silica-based material, but not for zirconia-based materials even if

the zirconia surface has been coated with silica<sup>25-28</sup>. Zirconate coupling agents and primer agents containing phosphate ester monomer such as 10-methacryloyloxydecyl dihydrogen phosphate (MDP) have been shown to increase the bond strength to zirconia<sup>29-31</sup>.

Hydrofluoric acid (HF) treatment is commonly used on silica-based ceramics to react with, and remove, the glassy matrix that contains silica. This leaves the crystalline phase exposed, generating surface roughness<sup>32-34</sup>. This process also results in enhanced wettability and surface energy on the ceramic surface, which allows greater penetration of the resin cement tags, increasing the bond strength between the ceramic and cement<sup>16,35</sup>.

Recently, several studies have reported that high strength alumina- and zirconia-based dental ceramics cannot be etched with hydrofluoric acid because of their high crystalline phase content<sup>5,17,20,23,24,26,31,36-40</sup>. Further, most studies have shown hydrofluoric acid etched zirconia in terms of mechanical property, bond strength between the zirconia surface and resin cement, but did not vary the etching times and the concentrations of hydrofluoric acid<sup>25,36-38</sup>. They also did not investigate the surface morphology changes. As a consequence, the aim of this study was to investigate the effect of hydrofluoric acid treatment on the surface topography and crystal structure of three commercially available partially-stabilized zirconia ceramics. The