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Milling properties of low temperature sintered zirconia blocks for dental use

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ABSTRACT

To investigate the milling properties of different yttria-tetragonal zirconia polycrystalline (Y-TZP) block materials by applying a dental computer numerical control (CNC) milling center. Low temperature sintering zirconia block denoted by KMUZ (experimental) with two commercial zirconia blocks for T block made in Taiwan and a G block made in Germany were compared for the milling properties. Seventy-two specimens were milled using the same CNC milling center, and properties were evaluated by measuring the weight loss (g), milling time (s), margin integrity (%) and broken diameter (μm). The crystalline phases contents were identified by X-ray diffraction and the microstructures of the sintering specimens were observed by scanning electron microscopy and transmission electron microscopy. The mean milling time of G and KMUZ were significantly shorter than T ($P < 0.05$). The KMUZ samples exhibited the least weight loss among the three kinds of blocks ($P < 0.05$). The percentages of marginal integrity after milling were high in G and KMUZ but low in T ($P < 0.05$). The mean broken diameters were from 90 μm to 120 μm. The phase transformation of t-ZrO_2 (KMUZ: 7.4%, G: 5.9%, T: 3.2%) to m-ZrO_2 when facing the milling pressure in ZrO_2 blocks was observed by XRD. The result of TEM microstructure of KMUZ revealed that Y and Si were soluble in grain boundaries. The results show that the milling properties of KMUZ were better than one commercial T and near the G. The hindered grain growth, as a result of the Y^{3+} content in the grain boundaries, also plays a role in promoting the abnormal grain growth of 3Y-TZP.

1. Introduction

Zirconia (ZrO_2) is dioxide of zirconium (Zr), which possesses three possible crystal structures: monoclinic (m), tetragonal (t) and cubic (c). At room temperature and pressure, ZrO_2 is naturally in the monoclinic phase, and when the temperature rises to approximately 1170 °C, phase transformation from m → t occurs; moreover, phase transformation from t → c takes place at 2370 °C [1]. Furthermore, when critical stress is applied to tetragonal zirconia, stress-induced phase transformation from metastable tetragonal into the monoclinic phase causes the crack tip to take place creating crack propagation resistance. This phenomenon is accompanied by about 3–5% volume expansion, which induces compressive stress that hinders crack propagation on the existing stress field [1–4]. This result of controlling the phase transformation from t → m occurring at room temperature leads to efficient milling by avoiding crack propagation and creating high fracture toughness [1].

Yttria-stabilized tetragonal zirconia polycrystals (Y-TZP), magnesium cation-doped partially stabilized zirconia (Mg-PSZ), and zirconia-toughened alumina (ZTA) are usually used in dentistry [5]. Owing to possessing high strength and fracture toughness in comparison with other ceramics, TZP has been regarded as an important biomedical ceramic. Therefore, many different processes for synthesizing the TZP powders and detailed discussions of its phase transitions and properties have been reported [6–17]. ZrO_2 used as a biomaterial was first reported by Helmer and Driskel [18]. In addition, the Y-TZP ceramics have become increasingly popular as an alternative high-toughness core materials in dental restorations as a result of their attractive mechanical properties, biocompatibility and superior natural appearance compared with metal dental restorations as reported by Piconi and Maccauro [1]. Therefore, full-ceramic implant supra-structures, root dental posts, crowns and fixed partial dentures have been used to replace traditional metals and alloys [5,19,20].

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The use of conventional milling machines for comparing the milling properties of different blocks have been studied by many researchers [21–25]. Sieged et al. [21] have pointed out that the milling efficiency of diamond dental burs depended on both the grit of the bur and the load applied to the handpiece. Alternatively, Watanabe et al. [23] reported that the milling efficiency of air-turbine burs on cast titanium and dental casting alloys are significantly ($P < 0.05$) influenced by air pressures. Blue et al. [24] noted that the roughest surfaces were obtained when using the super coarse with a bur size of 150 μm, and the prepared surface of alumina was rougher than zirconia ($P < 0.001$). On the other hand, Choi et al. [25] studied the milling efficiencies between electric and air-turbine handpieces have reported that the electric handpieces were more efficient than the air-turbine handpieces for milling various dentistry materials, especially machinable glass-ceramics, silver amalgam and high noble alloys.

As discussed above, milling efficiency can be affected by the grit of the bur, air pressures, the type of handpiece and the materials. Machine-operated human error still cannot be avoided. Due to the advances in software and hardware for open CNC system development [26], the computer-aided design and milling CNC systems could increase the interoperability and portability between the motion controller and the driver. Therefore, the aim of this study was to compare the milling properties and phase transformation of KMUZ (Kaohsiung Medical University zirconia) zirconia blocks with two kinds of commercial zirconia blocks.

2. Materials and methods

2.1. KMUZ zirconia blocks processing

High purity commercial 3Y-TZP powders (supplied by Tosoh Inc., Japan), SiO_2 (high purity, supplied by Longyan Shenghe Trading Co., Ltd.), Al_2O_3 (purity ≥ 99.0%, supplied by Henan Hengxin Industrial & Mineral Products Co., Ltd.) and $MgCO_3$ (purity ≥ 45%, supplied by Nanning Yihua Trading Co., Ltd.) were used as raw materials. The 3Y-TZP powders with 0.70 wt% SiO_2, 0.10 wt%, Al_2O_3 and 0.20 wt% $MgCO_3$ as additive were blended for 4 h in a laboratory ball mill containing high purity ZrO_2 sintered balls. The resulting powders mixtures were sifted through a 200-mesh sieve, then compacted at room temperature by uniaxial pressing at 176 MPa in a 37.5 mm diameter and 17.5 mm thickness stainless steel die. These compacted pellets were sintered at 950 °C for 3 h in a SiC furnace with a heating rate of 10 °C/min and then cooled in the furnaces. These blocks were denoted as KMUZ blocks. The blocks were sintered at 1350 and 1520 °C for 2 h, respectively, after the milling test.

2.2. Milling properties evaluation

Twenty-four experimental zirconia blocks of KMUZ blocks with 30 mm diameter and 14 mm thickness were selected in the present study. In addition, 48 samples of two commercial yttria-stabilized tetragonal zirconia blocks, T (made in Taiwan) and G (made in Germany), with a standard diameter of 98 mm and a thickness of 14 mm, were used in this study. Each specimen was milled from the center of the zirconia block. The experimental block was fixed in place using a resin block with a diameter of 98 mm to fit the milling machine. Altogether, 72 samples for the three kinds of zirconia blocks were milled to two kinds of milling forms, coping and monolithic crown. The dimensions of the different milling forms are shown in Fig. 1.

All samples were milled by the same open CNC system milling center (Ardenta CNC mill, DT100-4A, Tainan, Taiwan) with new tungsten carbide burs for each sample during the milling procedure. The milling efficiency was calculated from measuring the weight loss (g) by an electronic scale (Prema, India) and milling time (s) recorded by milling machine (DT100-4A, Tainan, Taiwan). The marginal surface integrity (%) of each sample was observed by light microscopy (Olympus BX51, Japan) and the diameter of mean breakage area (μm) was measured.

All statistical analyses were completed using SPSS software (Version 19.0 for windows, SPSS, Chicago, IL, USA). The level of significance was 5% ($P < 0.05$).

2.3. Samples characterization

The crystalline phase for the various kinds of zirconia block milling powders were identified by X-ray diffraction (XRD, XRD-6000, Shimadzu) with monochromatic CuK_α radiation ($\lambda = 0.15405$ nm) and a Ni filter operation with a voltage and current of 40 kV and 30 mA, respectively, at a scanning rate (2θ) of 1°/min. The phases content of tetragonal and monoclinic were determined by the Eqs. (1) and (2) [27],

$$f_M = \frac{I_M\left(\bar{1}11\right) + I_M(111)}{I_M\left(\bar{1}11\right) + I_M(111) + I_r(111)} \tag{1}$$

$$f_T = \frac{I_T(111)}{I_M\left(\bar{1}11\right) + I_M(111) + I_r(111)} \tag{2}$$

where f_T and f_M denote the fractions of the tetragonal and monoclinic ZrO_2 phases, respectively. $I_T(111)$ is the intensities of the tetragonal ZrO_2 (111) reflection, $I_M(\bar{1}\ 1\ 1)$ and $I_M(1\ 1\ 1)$ are the intensities of the monoclinic ZrO_2 ($\bar{1}$ 1 1) and (111) reflections, respectively.

The crystallite size of the tetragonal ZrO_2 zirconia was calculated using the Scherrer's equation [28]:

$$d = 0.89\lambda/\beta_{hkl}\cos\theta \tag{3}$$

where d is the crystallite size of tetragonal ZrO_2, βhkl is the calibrated width of the diffraction peak measured at the half maximum intensity of tetragonal ZrO_2, and λ is the wavelength of the X-ray radiation and θ is the Bragg angle.

The microstructure of the samples after sintering at 1520 °C was observed by SEM (JSM-6360, JEOL, Japan). The SEM samples were prepared using a thermal etching method, TEM (JEM 2100F, JEOL, Japan) was used to observe the morphology and selected area election diffraction (SAED) was used to determine the crystalline structure of the zirconia samples after sintering. TEM samples were prepared using a dual focus ion beam system (FIB, Seiko, SMI3050 SE).

3. Results

3.1. Milling efficiency and quality of various zirconia blocks

Milling efficiency and quality of various zirconia blocks are listed in Table 1. It is seen that the milling time of KMUZ samples were 963.8 ± 3.5 and 1136 ± 15.1 s ($P < 0.05$) for coping and crown forms respectively. In comparison with other ones, the milling time of coping form of KMUZ was lower than T and the same as G. For milling time of crown form, the KMUZ sample was lower than T but longer than G.

The weight loss of KMUZ blocks were 10.1 ± 0.8 g and 10.0 ± 1.0 g ($P > 0.05$) for coping and crown forms respectively. These values were different with the T and G ($P < 0.05$). For the mean diameter of breakage area after milling among 12 samples, the coping and crown forms of KMUZ were 92.4 ± 5.9 μm and 104.4 ± 4.7 μm ($P < 0.05$) respectively, which was close to 93.9 ± 7 μm and smaller than 116.9 μm for corresponding forms of G blocks. However, the 92.4 ± 5.9 μm was smaller than the 100.2 ± 81.9 μm of T blocks for coping form, but the 104.4 ± 4.7 μm was greater than the 89.8 ± 60.5 μm of T blocks for crowns.

The mean breakage diameter among three blocks was from 90 μm to 120 μm. The marginal integrity percentage of KMUZ and G blocks were high but low in T blocks ($P < 0.05$).