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Original article

Reactivity of zirconia with phosphate-bonded investments for hot-pressing



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ABSTRACT

Purpose: The aim of the present study was to investigate the reactivity of zirconia to phosphate-bonded investment on the process of hot-pressing technique.

Methods: Disc-shaped specimens, 16 mm diameter and 0.4 mm thick were prepared by cutting and grinding. These specimens were finally sintered. Half of them were sandblasted. The non-sandblasted specimens were used for controls. Both of zirconia discs with and without sandblasting were invested into the three kinds of phosphate-bonded investment. The investments were fired and broken to take out specimens. The biaxial strengths were measured in the way of the biaxial flexural tests according to ISO-6872.

Results: The biaxial strengths were ranged 762–1200 MPa at the average. The biaxial strengths of sandblasted zirconia discs heated with phosphate-bonded investment were decreased significantly ($p < 0.01$). SEM and EPMA revealed that the various compounds were observed on the sandblasted specimens. XRD revealed that surfaces of specimens heated with the phosphate-bonded investment showed the formation of phosphate compounds.

Conclusions: The phosphate compounds inhibit stress-induction phase transformation of zirconia and decrease the biaxial strength.

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1. Introduction

Zirconia has been increasingly used as the coping material for all-ceramic restorations [1,2]. Coping ceramics are commonly veneered with feldspathic porcelain using a condensation method. Recently, various methods of processing all-ceramic

restorations have been developed. One of them, a hot-pressing technique, has become available to simply and rapidly produce a veneering layer on zirconia copings [3–5]. The hot-pressing technique generally uses phosphate-bonded investment to invest zirconia copings waxed-up in the tooth-crown form. However, Ban et al. reported that presintered zirconia reacted with phosphates at high temperatures

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during the final sintering [6,7], resulting in phase transformation from tetragonal to monoclinic zirconia and therefore making the stress-induced phase transformation which provides mechanical stability to the zirconia crystal phase less effective. On the other hand, Sato et al. reported that sandblasting improved the mechanical properties of zirconia, but also decreased the stabilization of the crystal phase [8]. Therefore, there is a possibility that these effects are related – sandblasting may decrease the stabilization of the crystal phase of zirconia by increasing the reactivity of zirconia with phosphate-bonded investment. However, the influence of sandblasting on the reaction between zirconia and phosphate-bonded investment has not yet been revealed. Additionally, the inner surface of zirconia coping should be sandblasted for strong bonding between zirconia coping and abutment tooth [9]. The aim of the present study was to investigate the influence of phosphate-bonded investment on zirconia as it relates to the hot-pressing technique. We have performed extensive experimental measurements: X-ray diffractometry (XRD), scanning electron microscopy (SEM), electron probe microanalysis (EPMA) and biaxial flexural strength and shear bonding tests.

2. Materials and methods

2.1. Materials

The compositions of the two types of zirconia used in this study are listed in Table 1. Disc-shaped specimens, 16 mm diameter and 0.4 mm thick (for the biaxial flexural test) were prepared by cutting and grinding with a 400-grit diamond wheel. These specimens were finally sintered at 1450 °C for 2 h at a heating rate of 3.6 °C/min. Fig. 1 is a flowchart of surface treatment methods used for various specimens. The 16-mm-diameter discs were divided into two subgroups. Both surfaces of the specimens in one subgroup were ground on an abrasive unit (ECOMET 3, Buehler, USA) using diamond abrasive paper (35 μm in grain size) and then sandblasted with 70 μm Al₂O₃ at 0.4 MPa pressure for 10 s at a distance of 10 mm. The other specimens were used in their as-sintered condition, without additional grinding or sandblasting. These large discs were waxed-up, 1.0 mm in thickness, on one surface with paraffin wax (Paraffin Wax, GC, Japan). Clinically, the paraffin wax functions as a spacer to produce a veneering layer. These specimens were invested into three different phosphate-bonded investment materials, as listed in Table 2, and heated after setting according to the manufacturers' directions. These investments were generally used in dental laboratory. Ceravety was universal investment for pressable ceramics and cast alloys. PC-15 was exclusive investment for pressable ceramics. Moreover, Norinvest was investment that used for only zirconia flame. After cooling to room temperature, the specimens were carefully retrieved from the investments and cleaned in distilled water using an ultrasonic cleaner for 10 min. Five specimens were used for each heating condition, and un-heated specimens were used as controls ($n = 10$). The 16-mm-diameter discs were used for biaxial flexural strength tests and for XRD, SEM and EPMA observations. The abbreviations for each treatment are shown in Table 3.

2.2. Elemental analysis

The crystalline phases of phosphate-bonded investments and specimens were determined by XRD (Ultima IV, Rigaku, Japan) at 40 kV and 40 mA. Scans were conducted over a 2θ angular range from 10° to 60° at 2°/min for phosphate-bonded investments and a 2θ angular range from 20° to 76° at 2°/min for specimens used for the biaxial flexural tests.

The surface morphologies of the un-waxed-up sides of the biaxial flexural test specimens were characterized by SEM, using an accelerating voltage of 10 kV. The concentration profiles were measured perpendicular to the surface using EPMA with a Wavelength Dispersive X-ray Spectrometer (WDS, JXA-8530FA, JEOL, Japan). The probe was operated at an accelerating voltage of 10 kV and probe current of 50 nA, focused to produce a 0.1-μm-diameter electron beam at the specimen surface. Scans were performed with a step size of 0.1 μm and measurement time of 10 ms/point.

2.3. Biaxial flexural tests

The piston-on-three-balls method according to ISO-6872 was used for the biaxial flexural tests. Specimen discs were supported on three spheres (2.0 mm in diameter) spaced equally along a 10-mm diameter and centrally loaded at a crosshead speed of 0.5 mm/min using a steel piston (with a 1.1-mm-diameter flat area ground along the surface contact) until fracture occurred. The fracture stress was calculated using the equations listed in ISO-6872 and assuming a Poisson's ratios of 0.25 for both materials. The biaxial flexural test results were statistically analyzed by t-tests. Three-way analysis of variance (three-way ANOVA) was applied to determine the effects on biaxial flexural strength; type of zirconia, with/without sandblasting and type of investment were taken as independent factors. After the three-way ANOVA test the multiple comparison tests were performed using Bonferroni method. The statistical analyses were performed using a statistical package (IBM SPSS Statistics 18.0, IBM, USA).

3. Results

3.1. Elemental analysis

The compositions of phosphate-bonded investments before and after heating, as determined by XRD, are listed in Table 4. Each investment before heating contained SiO₂ (Quartz) (ICCD; No. 00-001-0649), NH₄MgPO₄·6H₂O (ICCD; No. 00-005-0316) and NH₄H₂PO₄ (ICCD; No. 00-001-0245). SiO₂ (Cristobalite) (ICCD; No. 00-001-0424) was determined in Ceravety and PC-15. After heating, Mg₂P₂O₇ (ICCD; No. 00-029-1399) was found in each investment, whereas the SiO₂ (Quartz and Cristobalite) was not changed.

The XRD patterns of non-sandblasted and sandblasted specimens heated with and without phosphate-bonded investment are shown in Fig. 2. ZrP₂O₇ (ICCD; No. 00-024-1491) was produced on the surface of inCoris ZI heated with Ceravety or PC-15 and on P-NANOZR heated with every phosphate-bonded investment (Fig. 2a). Additionally, YPO₄ (ICCD; No. 00-005-0454) and CePO₄ (ICCD; No. 00-004-0632)

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